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Annex

Obtenció d'un copolímer acrílic amb grups epoxi per a l'aplicació en tops per l'acabat de la pell

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1 ANNEXOS

1.1 ANNEX 1. Formulacions de les resines sintetitzades

1.1.1 Resines amb grups carboxílics

A continuació, es troben les taules corresponents a les formulacions de les 4 resines funcionalitzades amb grups carboxílics, identificades com a: R001A, R002A, R003A i R004A.

RESINA – R001A					
Ordre	**Producte**	**Quantitat**	**%**	**% sol.PM**	**% sòlids**
1	Aigua destil·lada	275.60	34.45	0.00	0.00
2	LAURIL SULFAT SÒDIC	20.00	2.50	28.00	0.70
3	Aigua destil·lada	154.31	19.29	0.00	0.00
4	LAURIL SULFAT SÒDIC	8.48	1.06	28.00	0.30
5	**BA - Butil Acrilat**	153.81	19.23	100.00	19.23
6	**MMA - Metacrilat de metil**	107.94	13.49	100.00	13.49
7	**MAA - Àcid metacrílic**	8.10	1.01	100.00	1.01
8	Aigua destil·lada	20.00	2.50	0.00	0.00
9	Persulfat amònic	1.20	0.15	100.00	0.15
10	tert-butil hidroperòxid	0.24	0.03	70.00	0.02
11	Aigua destil·lada	4.00	0.50	0.00	0.00
12	Brugollite	0.24	0.03	100.00	0.03
13	Aigua destil·lada	4.00	0.50	0.00	0.00
14	tert-butil hidroperòxid	0.24	0.03	70.00	0.02
15	Aigua destil·lada	4.00	0.50	0.00	0.00
16	Brugollite	0.24	0.03	100.00	0.03
17	Aigua destil·lada	4.00	0.50	0.00	0.00
18	Hidròxid amònic	4.80	0.60	0.00	0.00
19	Aigua destil·lada	4.00	0.50	0.00	0.00
20	Proxel GXL	0.80	0.10	20.00	0.02
21	Aigua destil·lada	24.00	3.00	0.00	0.00
	SUMA TOTAL	**800.00**	**100.00**		**35.00**

Taula 1. Formulació de la resina carboxílica R001A

RESINA – R002A					
Ordre	**Producte**	**Quantitat**	**%**	**% sol.PM**	**% sòlids**
1	Aigua destil·lada	275.60	34.45	0.00	0.00
2	LAURIL SULFAT SÒDIC	20.00	2.50	28.00	0.70
3	Aigua destil·lada	154.31	19.29	0.00	0.00
4	LAURIL SULFAT SÒDIC	8.48	1.06	28.00	0.30
5	**BA - Butil Acrilat**	134.92	16.87	100.00	16.87
6	**MMA - Metacrilat de metil**	107.94	13.49	100.00	13.49
7	**MAA - Àcid metacrílic**	26.98	3.37	100.00	3.37
8	Aigua destil·lada	20.00	2.50	0.00	0.00
9	Persulfat amònic	1.20	0.15	100.00	0.15
10	tert-butil hidroperòxid	0.24	0.03	70.00	0.02
11	Aigua destil·lada	4.00	0.50	0.00	0.00
12	Brugollite	0.24	0.03	100.00	0.03
13	Aigua destil·lada	4.00	0.50	0.00	0.00
14	tert-butil hidroperòxid	0.2400	0.03	70.00	0.02
15	Aigua destil·lada	4.0000	0.50	0.00	0.00
16	Brugollite	0.2400	0.03	100.00	0.03
17	Aigua destil·lada	4.0000	0.50	0.00	0.00
18	Hidròxid amònic	4.8000	0.60	0.00	0.00
19	Aigua destil·lada	4.0000	0.50	0.00	0.00
20	Proxel GXL	0.8000	0.10	20.00	0.02
21	Aigua destil·lada	24.0000	3.00	0.00	0.00
	SUMA TOTAL	**800.00**	**100.00**		**35.00**

Taula 2. Formulació de la resina carboxílica R002A

RESINA – R003A					
Ordre	**Producte**	**Quantitat**	**%**	**% sol.PM**	**% sòlids**
1	Aigua destil·lada	275.60	34.45	0.00	0.00
2	LAURIL SULFAT SÒDIC	20.00	2.50	28.00	0.70
3	Aigua destil·lada	154.31	19.29	0.00	0.00
4	LAURIL SULFAT SÒDIC	8.48	1.06	28.00	0.30
5	**BA - Butil Acrilat**	161.90	20.24	100.00	20.24
6	**MMA - Metacrilat de metil**	99.84	12.48	100.00	12.48
7	**MAA - Àcid metacrílic**	8.10	1.01	100.00	1.01
8	Aigua destil·lada	20.00	2.50	0.00	0.00
9	Persulfat amònic	1.20	0.15	100.00	0.15
10	tert-butil hidroperòxid	0.24	0.03	70.00	0.02
11	Aigua destil·lada	4.00	0.50	0.00	0.00
12	Brugollite	0.24	0.03	100.00	0.03
13	Aigua destil·lada	4.00	0.50	0.00	0.00
14	tert-butil hidroperòxid	0.24	0.03	70.00	0.02
15	Aigua destil·lada	4.00	0.50	0.00	0.00
16	Brugollite	0.24	0.03	100.00	0.03
17	Aigua destil·lada	4.00	0.50	0.00	0.00
18	Hidròxid amònic	4.80	0.60	0.00	0.00
19	Aigua destil·lada	4.00	0.50	0.00	0.00
20	Proxel GXL	0.80	0.10	20.00	0.02
21	Aigua destil·lada	24.00	3.00	0.00	0.00
	SUMA TOTAL	**800.00**	**100.00**		**35.00**

Taula 3. Formulació de la resina carboxílica R003A

RESINA – R004A					
Ordre	**Producte**	**Quantitat**	**%**	**% sol.PM**	**% sòlids**
1	Aigua destil·lada	275.60	34.45	0.00	0.00
2	LAURIL SULFAT SÒDIC	20.00	2.50	28.00	0.70
3	Aigua destil·lada	154.31	19.29	0.00	0.00
4	LAURIL SULFAT SÒDIC	8.48	1.06	28.00	0.30
5	**BA - Butil Acrilat**	161.90	20.23	100.00	20.24
6	**MMA - Metacrilat de metil**	80.95	10.11	100.00	10.12
7	**MAA - Àcid metacrílic**	26.98	3.37	100.00	3.37
8	Aigua destil·lada	20.00	2.50	0.00	0.00
9	Persulfat amònic	1.20	0.15	100.00	0.15
10	tert-butil hidroperòxid	0.24	0.03	70.00	0.02
11	Aigua destil·lada	4.00	0.50	0.00	0.00
12	Brugollite	0.24	0.03	100.00	0.03
13	Aigua destil·lada	4.00	0.50	0.00	0.00
14	tert-butil hidroperòxid	0.2400	0.03	70.00	0.02
15	Aigua destil·lada	4.0000	0.50	0.00	0.00
16	Brugollite	0.2400	0.03	100.00	0.03
17	Aigua destil·lada	4.0000	0.50	0.00	0.00
18	Hidròxid amònic	4.8000	0.60	0.00	0.00
19	Aigua destil·lada	4.0000	0.50	0.00	0.00
20	Proxel GXL	0.8000	0.10	20.00	0.02
21	Aigua destil·lada	24.0000	3.00	0.00	0.00
	SUMA TOTAL	**800.00**	**100.00**		**35.00**

Taula 4. Formulació de la resina carboxílica R004A

1.1.2 Resines amb grups epoxi

A continuació, es troben les taules corresponents a les formulacions de les 4 resines funcionalitzades amb grups epoxi, identificades com a: R005A, R006A, R007A i R008A.

RESINA – R005A					
Ordre	**Producte**	**Quantitat**	**%**	**% sol.PM**	**% sòlids**
1	Aigua destil·lada	275.60	34.45	0.00	0.00
2	LAURIL SULFAT SÒDIC	20.00	2.50	28.00	0.70
3	Aigua destil·lada	154.31	19.29	0.00	0.00
4	LAURIL SULFAT SÒDIC	8.48	1.06	28.00	0.30
5	**BA - Butil Acrilat**	153.81	19.23	100.00	19.23
6	**MMA - Metacrilat de metil**	107.94	13.49	100.00	13.49
7	**EPOXI**	8.10	1.01	100.00	1.01
8	Aigua destil·lada	20.00	2.50	0.00	0.00
9	Persulfat amònic	1.20	0.15	100.00	0.15
10	tert-butil hidroperòxid	0.24	0.03	70.00	0.02
11	Aigua destil·lada	4.00	0.50	0.00	0.00
12	Brugollite	0.24	0.03	100.00	0.03
13	Aigua destil·lada	4.00	0.50	0.00	0.00
14	tert-butil hidroperòxid	0.24	0.03	70.00	0.02
15	Aigua destil·lada	4.00	0.50	0.00	0.00
16	Brugollite	0.24	0.03	100.00	0.03
17	Aigua destil·lada	0.40	0.05	0.00	0.00
18	Hidròxid amònic	4.80	0.60	0.00	0.00
19	Aigua destil·lada	4.00	0.50	0.00	0.00
20	Proxel GXL	0.80	0.10	20.00	0.02
21	Aigua destil·lada	24.00	3.00	0.00	0.00
	SUMA TOTAL	**800.00**	**100.00**		**35.00**

Taula 5. Formulació de la resina epoxi R005A

RESINA – R006A					
Ordre	**Producte**	**Quantitat**	**%**	**% sol.PM**	**% sòlids**
1	Aigua destil·lada	275.60	34.45	0.00	0.00
2	LAURIL SULFAT SÒDIC	20.00	2.50	28.00	0.70
3	Aigua destil·lada	154.31	19.29	0.00	0.00
4	LAURIL SULFAT SÒDIC	8.48	1.06	28.00	0.30
5	**BA - Butil Acrilat**	134.92	16.87	100.00	16.87
6	**MMA - Metacrilat de metil**	107.94	13.49	100.00	13.49
7	**EPOXI**	26.98	3.37	100.00	3.37
8	Aigua destil·lada	20.00	2.50	0.00	0.00
9	Persulfat amònic	1.20	0.15	100.00	0.15
10	tert-butil hidroperòxid	0.24	0.03	70.00	0.02
11	Aigua destil·lada	4.00	0.50	0.00	0.00
12	Brugollite	0.24	0.03	100.00	0.03
13	Aigua destil·lada	4.00	0.50	0.00	0.00
14	tert-butil hidroperòxid	0.24	0.03	70.00	0.02
15	Aigua destil·lada	4.00	0.50	0.00	0.00
16	Brugollite	0.24	0.03	100.00	0.03
17	Aigua destil·lada	0.40	0.05	0.00	0.00
18	Hidròxid amònic	4.80	0.60	0.00	0.00
19	Aigua destil·lada	4.00	0.50	0.00	0.00
20	Proxel GXL	0.80	0.10	20.00	0.02
21	Aigua destil·lada	24.00	3.00	0.00	0.00
	SUMA TOTAL	**800.00**	**100.00**		**35.00**

Taula 6. Formulació de la resina epoxi R006A

RESINA – R007A					
Ordre	**Producte**	**Quantitat**	**%**	**% sol.PM**	**% sòlids**
1	Aigua destil·lada	275.60	34.45	0.00	0.00
2	LAURIL SULFAT SÒDIC	20.00	2.50	28.00	0.70
3	Aigua destil·lada	154.31	19.29	0.00	0.00
4	LAURIL SULFAT SÒDIC	8.48	1.06	28.00	0.30
5	**BA - Butil Acrilat**	161.90	20.23	100.00	20.24
6	**MMA - Metacrilat de metil**	99.84	12.48	100.00	12.48
7	**EPOXI**	8.10	1.01	100.00	1.01
8	Aigua destil·lada	20.00	2.50	0.00	0.00
9	Persulfat amònic	1.20	0.15	100.00	0.15
10	tert-butil hidroperòxid	0.24	0.03	70.00	0.02
11	Aigua destil·lada	4.00	0.50	0.00	0.00
12	Brugollite	0.24	0.03	100.00	0.03
13	Aigua destil·lada	4.00	0.50	0.00	0.00
14	tert-butil hidroperòxid	0.24	0.03	70.00	0.02
15	Aigua destil·lada	4.00	0.50	0.00	0.00
16	Brugollite	0.24	0.03	100.00	0.03
17	Aigua destil·lada	0.40	0.05	0.00	0.00
18	Hidròxid amònic	4.80	0.60	0.00	0.00
19	Aigua destil·lada	4.00	0.50	0.00	0.00
20	Proxel GXL	0.80	0.10	20.00	0.02
21	Aigua destil·lada	24.00	3.00	0.00	0.00
	SUMA TOTAL	**800.00**	**100.00**		**35.00**

Taula 7. Formulació de la resina epoxi R007A

RESINA – R008A					
Ordre	**Producte**	**Quantitat**	**%**	**% sol.PM**	**% sòlids**
1	Aigua destil·lada	275.60	34.45	0.00	0.00
2	LAURIL SULFAT SÒDIC	20.00	2.50	28.00	0.70
3	Aigua destil·lada	154.31	19.29	0.00	0.00
4	LAURIL SULFAT SÒDIC	8.48	1.06	28.00	0.30
5	**BA - Butil Acrilat**	161.90	20.24	100.00	20.24
6	**MMA - Metacrilat de metil**	80.95	10.12	100.00	10.12
7	**EPOXI**	26.98	3.37	100.00	3.37
8	Aigua destil·lada	20.00	2.50	0.00	0.00
9	Persulfat amònic	1.20	0.15	100.00	0.15
10	tert-butil hidroperòxid	0.24	0.03	70.00	0.02
11	Aigua destil·lada	4.00	0.50	0.00	0.00
12	Brugollite	0.24	0.03	100.00	0.03
13	Aigua destil·lada	4.00	0.50	0.00	0.00
14	tert-butil hidroperòxid	0.24	0.03	70.00	0.02
15	Aigua destil·lada	4.00	0.50	0.00	0.00
16	Brugollite	0.24	0.03	100.00	0.03
17	Aigua destil·lada	0.40	0.05	0.00	0.00
18	Hidròxid amònic	4.80	0.60	0.00	0.00
19	Aigua destil·lada	4.00	0.50	0.00	0.00
20	Proxel GXL	0.80	0.10	20.00	0.02
21	Aigua destil·lada	24.00	3.00	0.00	0.00
	SUMA TOTAL	**800.00**	**100.00**		**35.00**

Taula 8. Formulació de la resina epoxi R008A

1.1.3 Resines amb grups amínics

A continuació, es troben les taules corresponents a les formulacions de les 4 resines funcionalitzades amb grups amínics, identificades com a: R015A, R016A, R017A i R018A

RESINA – R015A					
Ordre	**Producte**	**Quantitat**	**%**	**% sol.PM**	**% sòlids**
1	Aigua destil·lada	275,6008	34,4501	0,00	0,00
2	LAURIL SULFAT SÒDIC	20,0000	2,5000	28,00	0,70
3	Aigua destil·lada	154,3192	19,2899	0,00	0,00
4	LAURIL SULFAT SÒDIC	8,4800	1,0600	28,00	0,30
5	**BA - Butil Acrilat**	153,8088	19,2261	100,00	19,23
6	**MMA - Metacrilat de metil**	107,9360	13,4920	100,00	13,49
7	**TBAEMA**	8,0952	1,0119	100,00	1,01
8	Aigua destil·lada	20,0000	2,5000	0,00	0,00
9	Persulfat amònic	1,2000	0,1500	100,00	0,15
10	tert-butil hidroperòxid	0,2400	0,0300	70,00	0,02
11	Aigua destil·lada	4,0000	0,5000	0,00	0,00
12	Brugollite	0,2400	0,0300	100,00	0,03
13	Aigua destil·lada	4,0000	0,5000	0,00	0,00
14	tert-butil hidroperòxid	0,2400	0,0300	70,00	0,02
15	Aigua destil·lada	4,0000	0,5000	0,00	0,00
16	Brugollite	0,2400	0,0300	100,00	0,03
17	Aigua destil·lada	4,0000	0,5000	0,00	0,00
18	Hidròxid amònic	4,8000	0,6000	0,00	0,00
19	Aigua destil·lada	4,0000	0,5000	0,00	0,00
20	Proxel GXL	0,8000	0,1000	20,00	0,02
21	Aigua destil·lada	24,0000	3,0000	0,00	0,00
	SUMA TOTAL	**800.00**	**100.00**		**35.00**

Taula 9. Formulació de la resina amínica R015A

RESINA – R016A					
Ordre	**Producte**	**Quantitat**	**%**	**% sol.PM**	**% sòlids**
1	Aigua destil·lada	275,6008	34,4501	0,00	0,00
2	LAURIL SULFAT SÒDIC	20,0000	2,5000	28,00	0,70
3	Aigua destil·lada	154,3192	19,2899	0,00	0,00
4	LAURIL SULFAT SÒDIC	8,4800	1,0600	28,00	0,30
5	**BA - Butil Acrilat**	134,9200	16,8650	100,00	16,87
6	**MMA - Metacrilat de metil**	107,9360	13,4920	100,00	13,49
7	**TBAEMA**	26,9840	3,3730	100,00	3,37
8	Aigua destil·lada	20,0000	2,5000	0,00	0,00
9	Persulfat amònic	1,2000	0,1500	100,00	0,15
10	tert-butil hidroperòxid	0,2400	0,0300	70,00	0,02
11	Aigua destil·lada	4,0000	0,5000	0,00	0,00
12	Brugollite	0,2400	0,0300	100,00	0,03
13	Aigua destil·lada	4,0000	0,5000	0,00	0,00
14	tert-butil hidroperòxid	0,2400	0,0300	70,00	0,02
15	Aigua destil·lada	4,0000	0,5000	0,00	0,00
16	Brugollite	0,2400	0,0300	100,00	0,03
17	Aigua destil·lada	4,0000	0,5000	0,00	0,00
18	Hidròxid amònic	4,8000	0,6000	0,00	0,00
19	Aigua destil·lada	4,0000	0,5000	0,00	0,00
20	Proxel GXL	0,8000	0,1000	20,00	0,02
21	Aigua destil·lada	24,0000	3,0000	0,00	0,00
	SUMA TOTAL	**800.00**	**100.00**		**35.00**

Taula 10. Formulació de la resina amínica R016A

RESINA – R017A					
Ordre	**Producte**	**Quantitat**	**%**	**% sol.PM**	**% sòlids**
1	Aigua destil·lada	275,6008	34,4501	0,00	0,00
2	LAURIL SULFAT SÒDIC	20,0000	2,5000	28,00	0,70
3	Aigua destil·lada	154,3192	19,2899	0,00	0,00
4	LAURIL SULFAT SÒDIC	8,4800	1,0600	28,00	0,30
5	**BA - Butil Acrilat**	161,9040	20,2380	100,00	20,24
6	**MMA - Metacrilat de metil**	99,8408	12,4801	100,00	12,48
7	**TBAEMA**	8,0952	1,0119	100,00	1,01
8	Aigua destil·lada	20,0000	2,5000	0,00	0,00
9	Persulfat amònic	1,2000	0,1500	100,00	0,15
10	tert-butil hidroperòxid	0,2400	0,0300	70,00	0,02
11	Aigua destil·lada	4,0000	0,5000	0,00	0,00
12	Brugollite	0,2400	0,0300	100,00	0,03
13	Aigua destil·lada	4,0000	0,5000	0,00	0,00
14	tert-butil hidroperòxid	0,2400	0,0300	70,00	0,02
15	Aigua destil·lada	4,0000	0,5000	0,00	0,00
16	Brugollite	0,2400	0,0300	100,00	0,03
17	Aigua destil·lada	4,0000	0,5000	0,00	0,00
18	Hidròxid amònic	4,8000	0,6000	0,00	0,00
19	Aigua destil·lada	4,0000	0,5000	0,00	0,00
20	Proxel GXL	0,8000	0,1000	20,00	0,02
21	Aigua destil·lada	24,0000	3,0000	0,00	0,00
	SUMA TOTAL	**800.00**	**100.00**		**35.00**

Taula 11. Formulació de la resina amínica R017A

RESINA – R018A					
Ordre	**Producte**	**Quantitat**	**%**	**% sol.PM**	**% sòlids**
1	Aigua destil·lada	275,6008	34,4501	0,00	0,00
2	LAURIL SULFAT SÒDIC	20,0000	2,5000	28,00	0,70
3	Aigua destil·lada	154,3192	19,2899	0,00	0,00
4	LAURIL SULFAT SÒDIC	8,4800	1,0600	28,00	0,30
5	**BA - Butil Acrilat**	161,9040	20,2380	100,00	20,24
6	**MMA - Metacrilat de metil**	80,9520	10,1190	100,00	10,12
7	**TBAEMA**	26,9840	3,3730	100,00	3,37
8	Aigua destil·lada	20,0000	2,5000	0,00	0,00
9	Persulfat amònic	1,2000	0,1500	100,00	0,15
10	tert-butil hidroperòxid	0,2400	0,0300	70,00	0,02
11	Aigua destil·lada	4,0000	0,5000	0,00	0,00
12	Brugollite	0,2400	0,0300	100,00	0,03
13	Aigua destil·lada	4,0000	0,5000	0,00	0,00
14	tert-butil hidroperòxid	0,2400	0,0300	70,00	0,02
15	Aigua destil·lada	4,0000	0,5000	0,00	0,00
16	Brugollite	0,2400	0,0300	100,00	0,03
17	Aigua destil·lada	4,0000	0,5000	0,00	0,00
18	Hidròxid amònic	4,8000	0,6000	0,00	0,00
19	Aigua destil·lada	4,0000	0,5000	0,00	0,00
20	Proxel GXL	0,8000	0,1000	20,00	0,02
21	Aigua destil·lada	24,0000	3,0000	0,00	0,00
	SUMA TOTAL	**800.00**	**100.00**		**35.00**

Taula 12. Formulació de la resina amínica R018A

1.1.4 Resines Seleccionades: carboxílica, epoxi, amínica

A continuació, es troben les taules corresponents a les formulacions de les 3 resines seleccionades, una amb cada grup funcional.

RESINA SELECCIONADA CARBOXÍLICA					
Ordre	**Producte**	**Quantitat**	**%**	**% sol.PM**	**% sòlids**
1	Aigua destil·lada	275,6008	34,4501	0,00	0,00
2	LAURIL SULFAT SÒDIC	20,0000	2,5000	28,00	0,70
3	Aigua destil·lada	154,3192	19,2899	0,00	0,00
4	LAURIL SULFAT SÒDIC	8,4800	1,0600	28,00	0,30
5	**BA - Butil Acrilat**	**134,8800**	**16,8600**	100,00	16,86
6	**MMA - Metacrilat de metil**	**80,9600**	**10,1200**	100,00	10,12
7	**MAA - Àcid metacrílic**	**54,0000**	**6,7500**	100,00	6,75
8	Aigua destil·lada	20,0000	2,5000	0,00	0,00
9	Persulfat amònic	1,2000	0,1500	100,00	0,15
10	tert-butil hidroperòxid	0,2400	0,0300	70,00	0,02
11	Aigua destil·lada	4,0000	0,5000	0,00	0,00
12	Brugollite	0,2400	0,0300	100,00	0,03
13	Aigua destil·lada	4,0000	0,5000	0,00	0,00
14	tert-butil hidroperòxid	0,2400	0,0300	70,00	0,02
15	Aigua destil·lada	4,0000	0,5000	0,00	0,00
16	Brugollite	0,2400	0,0300	100,00	0,03
17	Aigua destil·lada	4,0000	0,5000	0,00	0,00
18	Hidròxid amònic	4,8000	0,6000	0,00	0,00
19	Aigua destil·lada	4,0000	0,5000	0,00	0,00
20	Proxel GXL	0,8000	0,1000	20,00	0,02
21	Aigua destil·lada	24,0000	3,0000	0,00	0,00
	SUMA TOTAL	**800.00**	**100.00**		**35.00**

Taula 13. Formulació de la resina carboxílica seleccionada

RESINA SELECCIONADA EPOXI					
Ordre	**Producte**	**Quantitat**	**%**	**% sol.PM**	**% sòlids**
1	Aigua destil·lada	275,6008	34,4501	0,00	0,00
2	LAURIL SULFAT SÒDIC	20,0000	2,5000	28,00	0,70
3	Aigua destil·lada	154,3192	19,2899	0,00	0,00
4	LAURIL SULFAT SÒDIC	8,4800	1,0600	28,00	0,30
5	**BA - Butil Acrilat**	**180,8000**	**22,6000**	100,00	22,60
6	**MMA - Metacrilat de metil**	**80,9600**	**10,1200**	100,00	10,12
7	**GMA**	**8,0800**	**1,0100**	100,00	1,01
8	Aigua destil·lada	20,0000	2,5000	0,00	0,00
9	Persulfat amònic	1,2000	0,1500	100,00	0,15
10	tert-butil hidroperòxid	0,2400	0,0300	70,00	0,02
11	Aigua destil·lada	4,0000	0,5000	0,00	0,00
12	Brugollite	0,2400	0,0300	100,00	0,03
13	Aigua destil·lada	4,0000	0,5000	0,00	0,00
14	tert-butil hidroperòxid	0,2400	0,0300	70,00	0,02
15	Aigua destil·lada	4,0000	0,5000	0,00	0,00
16	Brugollite	0,2400	0,0300	100,00	0,03
17	Aigua destil·lada	4,0000	0,5000	0,00	0,00
18	Hidròxid amònic	4,8000	0,6000	0,00	0,00
19	Aigua destil·lada	4,0000	0,5000	0,00	0,00
20	Proxel GXL	0,8000	0,1000	20,00	0,02
21	Aigua destil·lada	24,0000	3,0000	0,00	0,00
	SUMA TOTAL	**800.00**	**100.00**		**35.00**

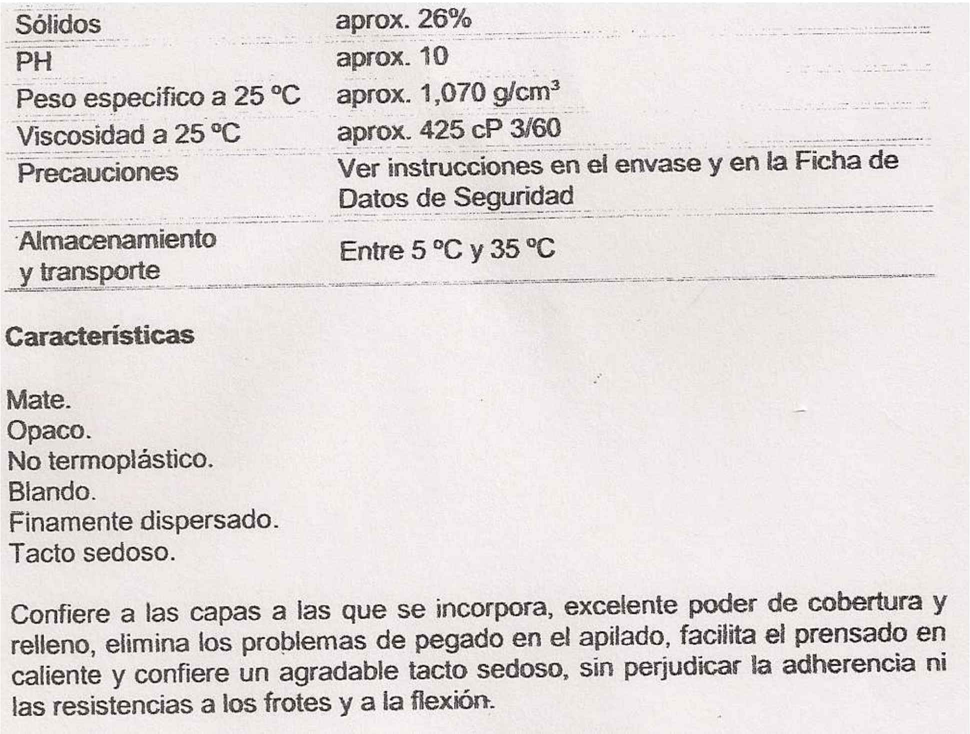
Taula 14. Formulació de la resina epoxi seleccionada

RESINA SELECCIONADA AMÍNICA					
Ordre	**Producte**	**Quantitat**	**%**	**% sol.PM**	**% sòlids**
1	Aigua destil·lada	275,6008	34,4501	0,00	0,00
2	LAURIL SULFAT SÒDIC	20,0000	2,5000	28,00	0,70
3	Aigua destil·lada	154,3192	19,2899	0,00	0,00
4	LAURIL SULFAT SÒDIC	8,4800	1,0600	28,00	0,30
5	**BA - Butil Acrilat**	**134,9600**	**16,8700**	100,00	16,87
6	**MMA - Metacrilat de metil**	**126,8000**	**15,8500**	100,00	15,85
7	**TBAEMA**	**8,0800**	**1,0100**	100,00	1,01
8	Aigua destil·lada	20,0000	2,5000	0,00	0,00
9	Persulfat amònic	1,2000	0,1500	100,00	0,15
10	tert-butil hidroperòxid	0,2400	0,0300	70,00	0,02
11	Aigua destil·lada	4,0000	0,5000	0,00	0,00
12	Brugollite	0,2400	0,0300	100,00	0,03
13	Aigua destil·lada	4,0000	0,5000	0,00	0,00
14	tert-butil hidroperòxid	0,2400	0,0300	70,00	0,02
15	Aigua destil·lada	4,0000	0,5000	0,00	0,00
16	Brugollite	0,2400	0,0300	100,00	0,03
17	Aigua destil·lada	4,0000	0,5000	0,00	0,00
18	Hidròxid amònic	4,8000	0,6000	0,00	0,00
19	Aigua destil·lada	4,0000	0,5000	0,00	0,00
20	Proxel GXL	0,8000	0,1000	20,00	0,02
21	Aigua destil·lada	24,0000	3,0000	0,00	0,00
	SUMA TOTAL	**800.00**	**100.00**		**35.00**

Taula 15. Formulació de la resina amínica seleccionada

1.2 ANNEX 2. Fitxes tècniques dels productes utilitzats en l'acabat de la pell

1.2.1 Complex de Silici (Caolí)



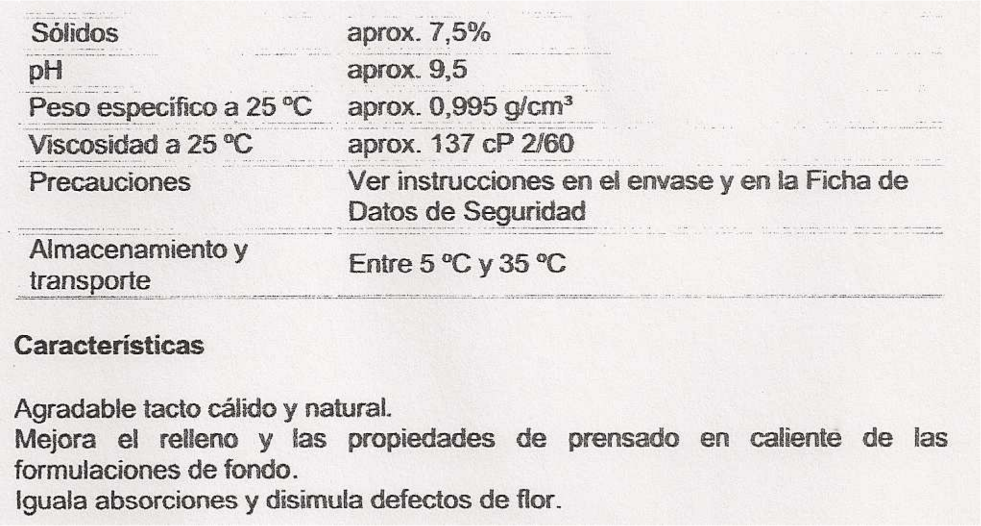
Sólidos	aprox. 26%
PH	aprox. 10
Peso especifico a 25 °C	aprox. 1,070 g/cm³
Viscosidad a 25 °C	aprox. 425 cP 3/60
Precauciones	Ver instrucciones en el envase y en la Ficha de Datos de Seguridad
Almacenamiento y transporte	Entre 5 °C y 35 °C

Características

Mate.
Opaco.
No termoplástico.
Blando.
Finamente dispersado.
Tacto sedoso.

Confiere a las capas a las que se incorpora, excelente poder de cobertura y relleno, elimina los problemas de pegado en el apilado, facilita el prensado en caliente y confiere un agradable tacto sedoso, sin perjudicar la adherencia ni las resistencias a los frotes y a la flexión.

1.2.2 Emulsió de Ceres

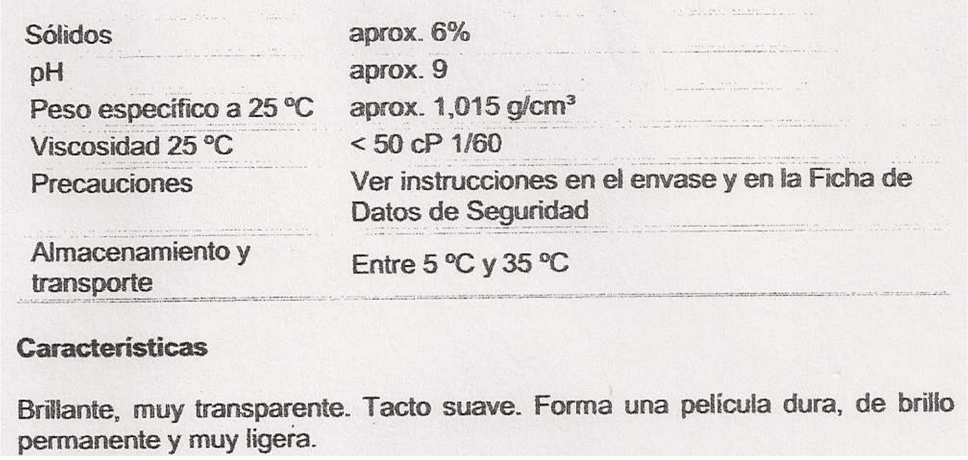


Sólidos	aprox. 7,5%
pH	aprox. 9,5
Peso especifico a 25 °C	aprox. 0,995 g/cm³
Viscosidad a 25 °C	aprox. 137 cP 2/60
Precauciones	Ver instrucciones en el envase y en la Ficha de Datos de Seguridad
Almacenamiento y transporte	Entre 5 °C y 35 °C

Características

Agradable tacto cálido y natural.
Mejora el relleno y las propiedades de prensado en caliente de las formulaciones de fondo.
Iguala absorciones y disimula defectos de flor.

1.2.3 Lligant Proteínic

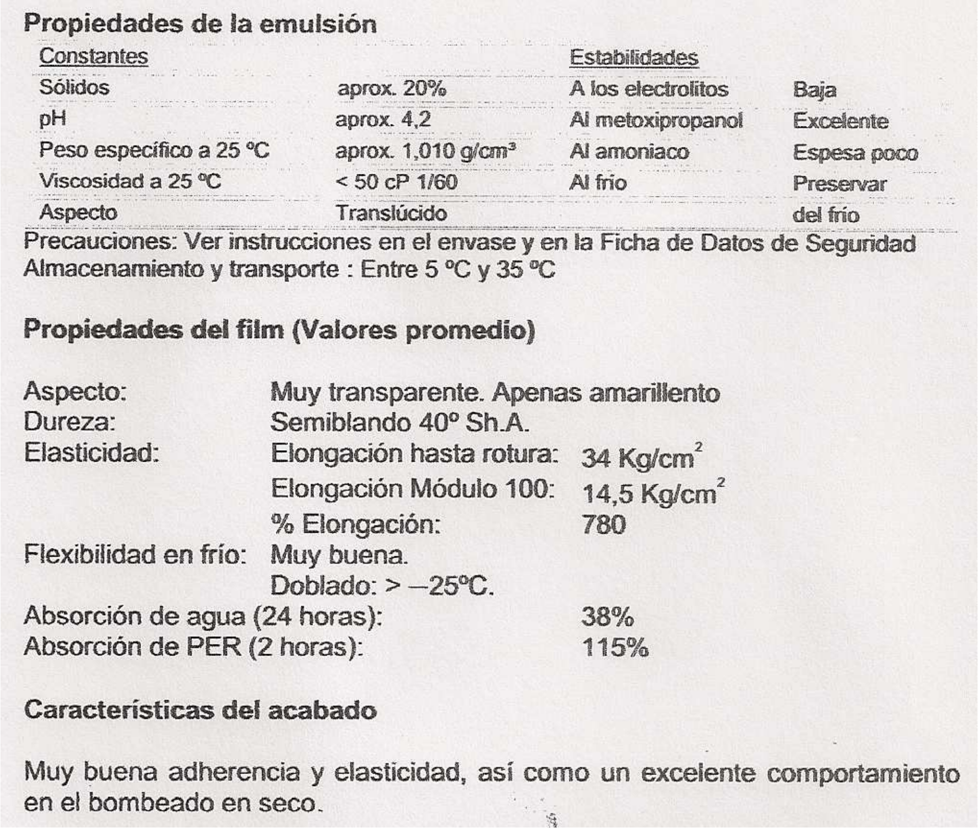


Sólidos	aprox. 6%
pH	aprox. 9
Peso especifico a 25 ºC	aprox. 1,015 g/cm^3
Viscosidad 25 ºC	< 50 cP 1/60
Precauciones	Ver instrucciones en el envase y en la Ficha de Datos de Seguridad
Almacenamiento y transporte	Entre 5 ºC y 35 ºC

Características

Brillante, muy transparente. Tacto suave. Forma una película dura, de brillo permanente y muy ligera.

1.2.4 Resina Acrílica



Propiedades de la emulsión

Constantes		Estabilidades	
Sólidos	aprox. 20%	A los electrolitos	Baja
pH	aprox. 4,2	Al metoxipropanol	Excelente
Peso específico a 25 ºC	aprox. 1,010 g/cm^3	Al amoniaco	Espesa poco
Viscosidad a 25 ºC	< 50 cP 1/60	Al frio	Preservar
Aspecto	Translúcido		del frío

Precauciones: Ver instrucciones en el envase y en la Ficha de Datos de Seguridad
Almacenamiento y transporte : Entre 5 ºC y 35 ºC

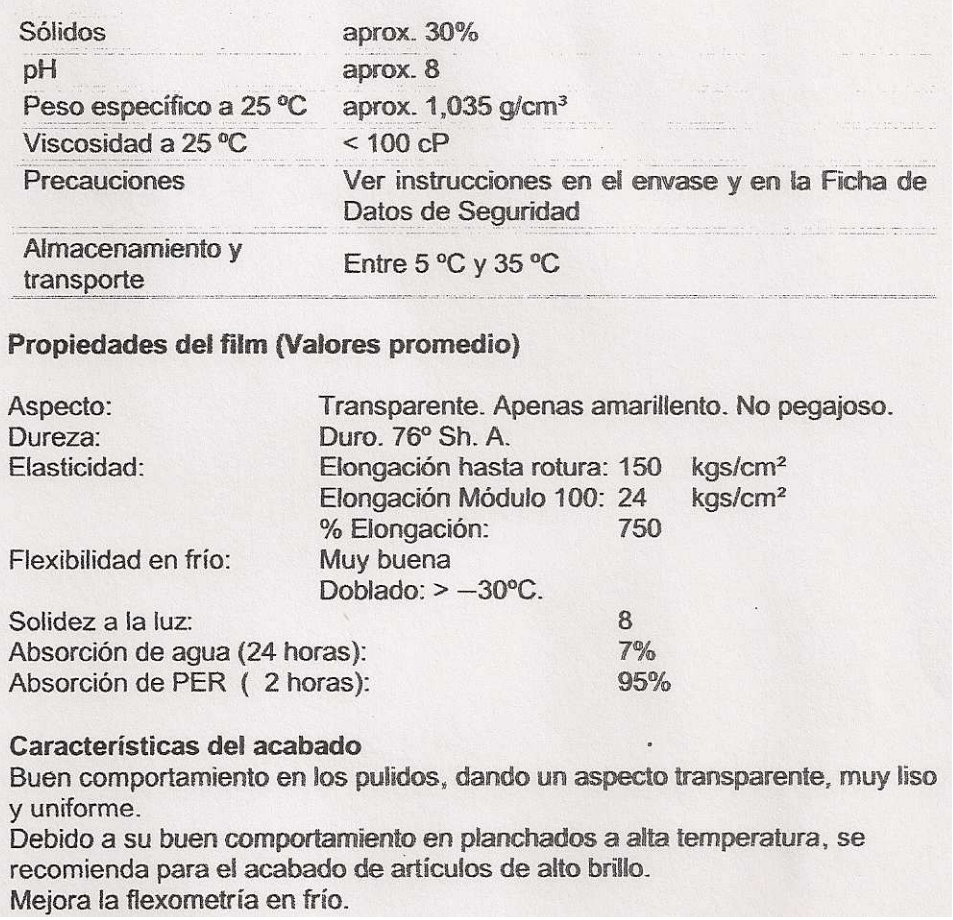
Propiedades del film (Valores promedio)

Aspecto: Muy transparente. Apenas amarillento
Dureza: Semiblando 40º Sh.A.
Elasticidad: Elongación hasta rotura: 34 Kg/cm^2
Elongación Módulo 100: 14,5 Kg/cm^2
% Elongación: 780
Flexibilidad en frío: Muy buena.
Doblado: > –25ºC.
Absorción de agua (24 horas): 38%
Absorción de PER (2 horas): 115%

Características del acabado

Muy buena adherencia y elasticidad, así como un excelente comportamiento en el bombeado en seco.

1.2.5 Resina d'Uretà



Sólidos	aprox. 30%
pH	aprox. 8
Peso específico a 25 ºC	aprox. 1,035 g/cm^3
Viscosidad a 25 ºC	< 100 cP
Precauciones	Ver instrucciones en el envase y en la Ficha de Datos de Seguridad
Almacenamiento y transporte	Entre 5 ºC y 35 ºC

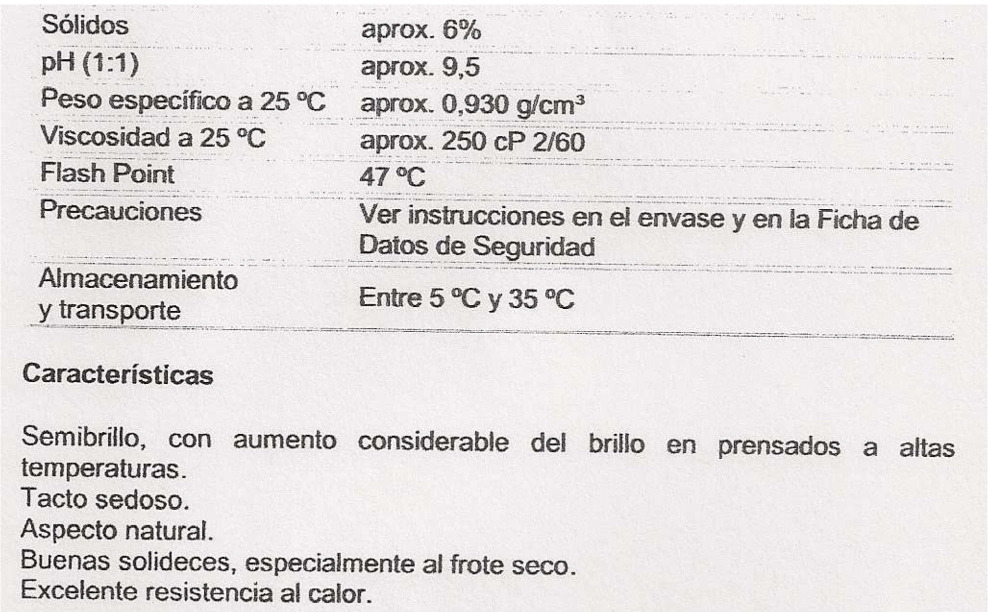
Propiedades del film (Valores promedio)

Aspecto:	Transparente. Apenas amarillento. No pegajoso.
Dureza:	Duro. 76º Sh. A.
Elasticidad:	Elongación hasta rotura: 150 kgs/cm^2 Elongación Módulo 100: 24 kgs/cm^2 % Elongación: 750
Flexibilidad en frío:	Muy buena Doblado: > –30ºC.
Solidez a la luz:	8
Absorción de agua (24 horas):	7%
Absorción de PER (2 horas):	95%

Características del acabado

Buen comportamiento en los pulidos, dando un aspecto transparente, muy liso y uniforme.
Debido a su buen comportamiento en planchados a alta temperatura, se recomienda para el acabado de artículos de alto brillo.
Mejora la flexometría en frío.

1.2.6 Laca

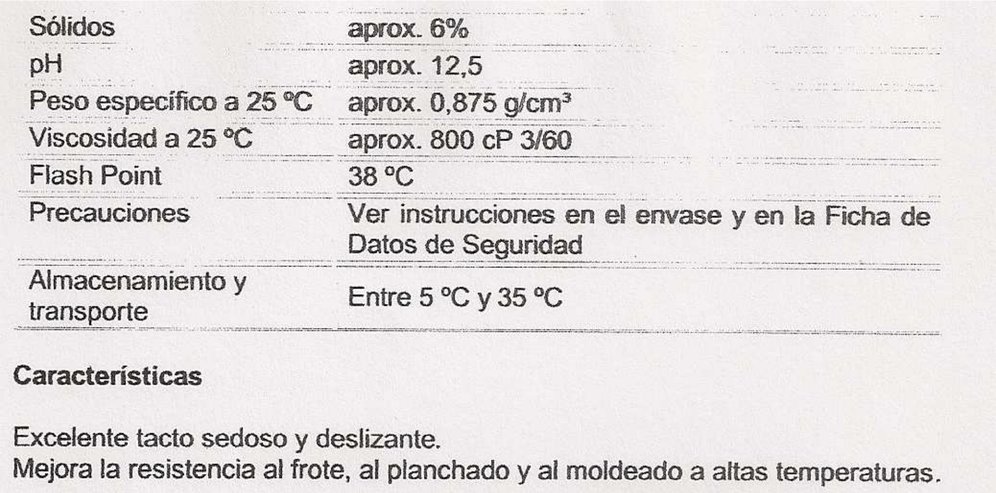


Sólidos	aprox. 6%
pH (1:1)	aprox. 9,5
Peso específico a 25 ºC	aprox. 0,930 g/cm^3
Viscosidad a 25 ºC	aprox. 250 cP 2/60
Flash Point	47 ºC
Precauciones	Ver instrucciones en el envase y en la Ficha de Datos de Seguridad
Almacenamiento y transporte	Entre 5 ºC y 35 ºC

Características

Semibrillo, con aumento considerable del brillo en prensados a altas temperaturas.
Tacto sedoso.
Aspecto natural.
Buenas solideces, especialmente al frote seco.
Excelente resistencia al calor.

1.2.7 Silicona

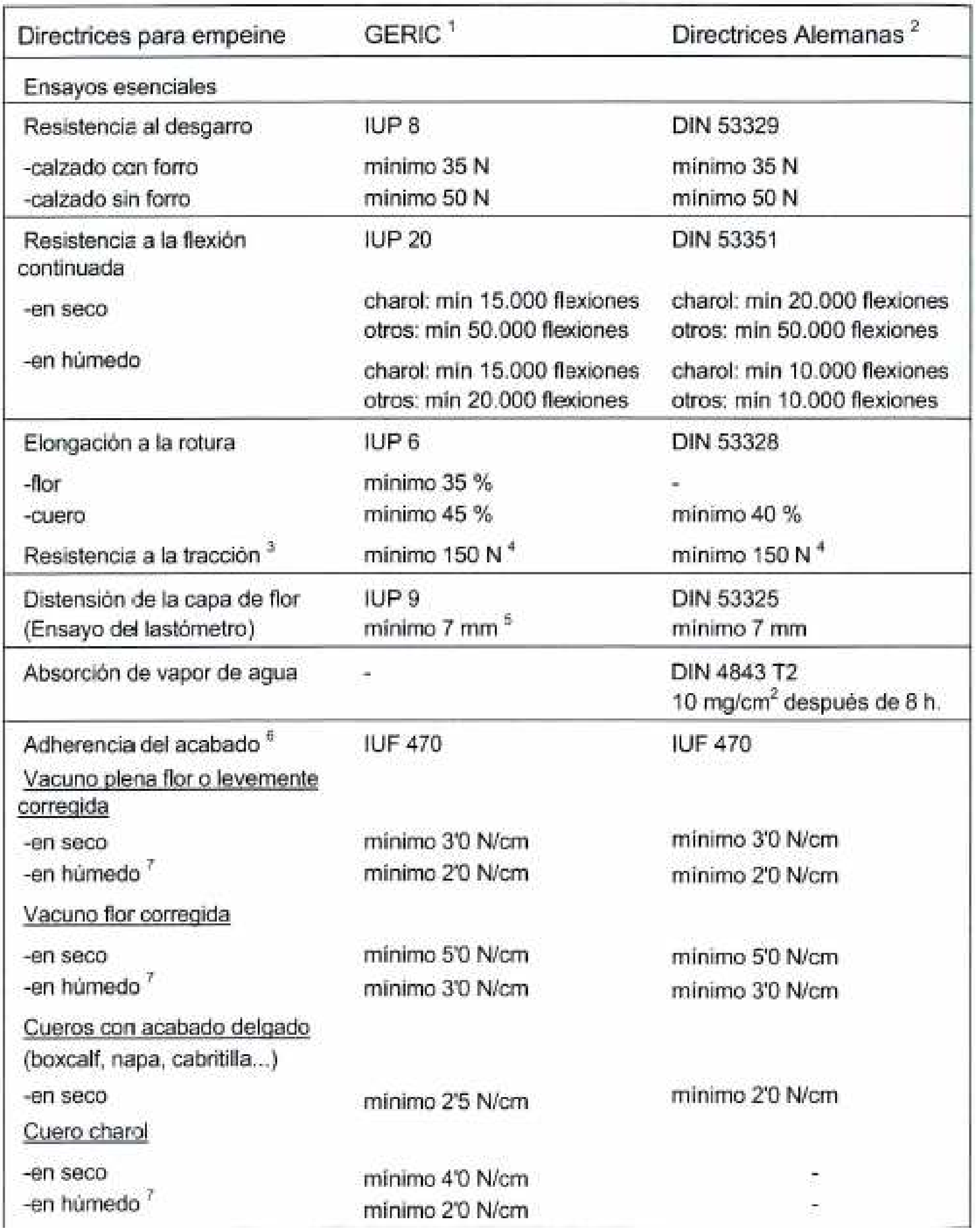


Sólidos	aprox. 6%
pH	aprox. 12,5
Peso específico a 25 ºC	aprox. 0,875 g/cm³
Viscosidad a 25 ºC	aprox. 800 cP 3/60
Flash Point	38 ºC
Precauciones	Ver instrucciones en el envase y en la Ficha de Datos de Seguridad
Almacenamiento y transporte	Entre 5 ºC y 35 ºC

Características

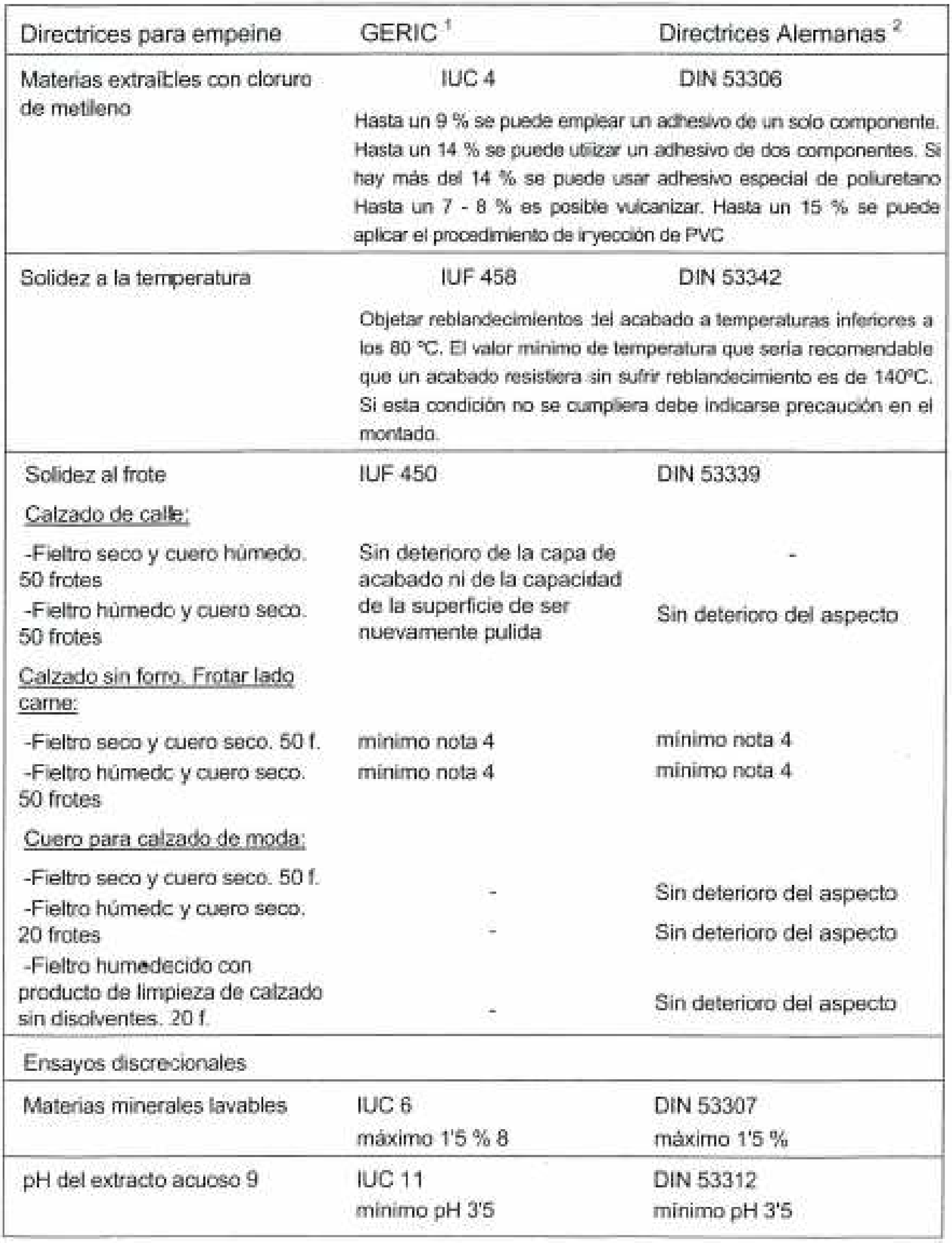
Excelente tacto sedoso y deslizante.
Mejora la resistencia al frote, al planchado y al moldeado a altas temperaturas.

1.3 ANNEX 3. Recomanacions GERIC: qualitat de la pell per a empenya de calçat



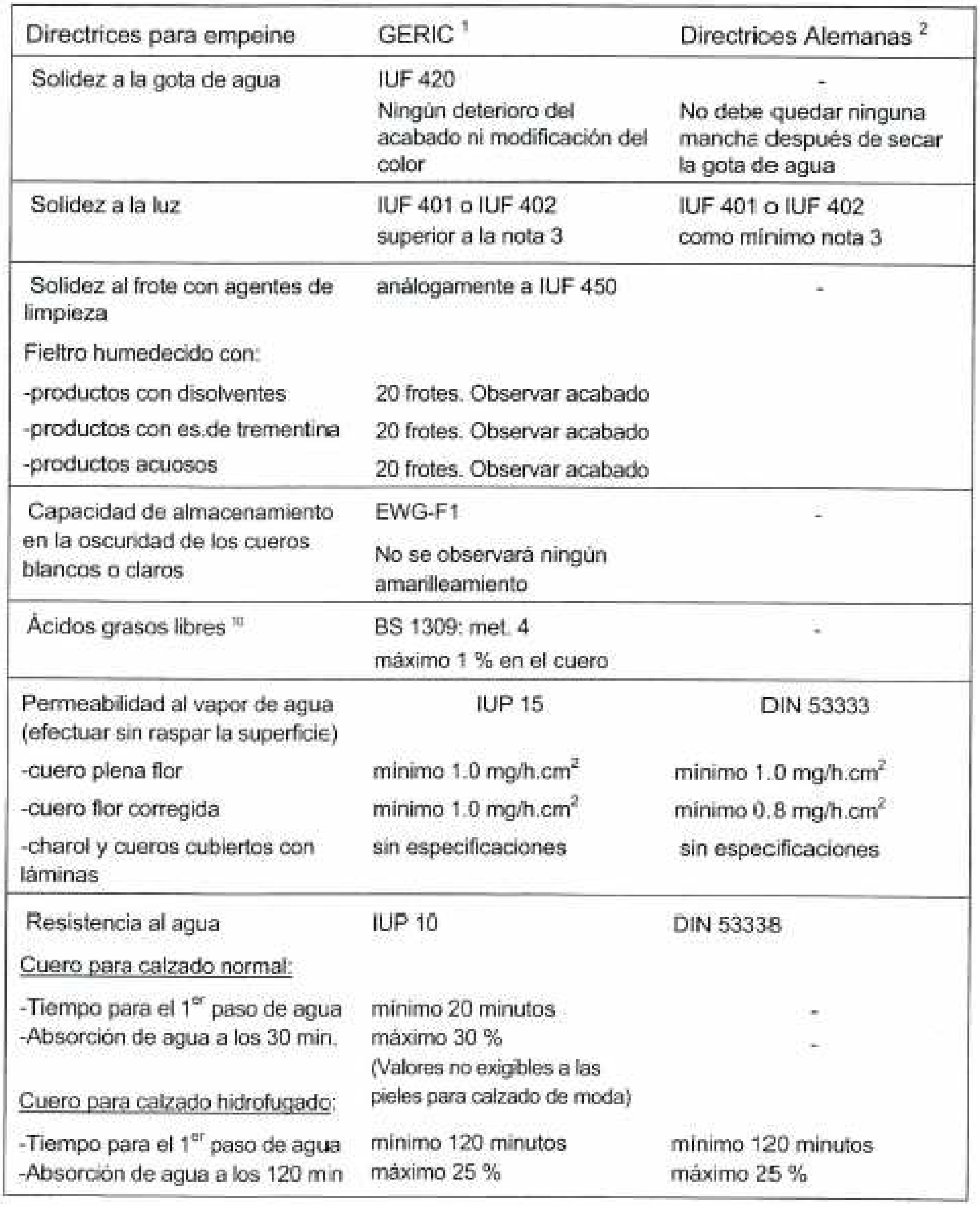
Directrices para empeine	GERIC [1]	Directrices Alemanas [2]
Ensayos esenciales		
Resistencia al desgarro	IUP 8	DIN 53329
-calzado con forro	minimo 35 N	mínimo 35 N
-calzado sin forro	minimo 50 N	mínimo 50 N
Resistencia a la flexión continuada	IUP 20	DIN 53351
-en seco	charol: min 15.000 flexiones otros: min 50.000 flexiones	charol: min 20.000 flexiones otros: min 50.000 flexiones
-en húmedo	charol: min 15.000 flexiones otros: min 20.000 flexiones	charol: min 10.000 flexiones otros: min 10.000 flexiones
Elongación a la rotura	IUP 6	DIN 53328
-flor	minimo 35 %	-
-cuero	minimo 45 %	mínimo 40 %
Resistencia a la tracción [3]	minimo 150 N [4]	mínimo 150 N [4]
Distensión de la capa de flor (Ensayo del lastómetro)	IUP 9 mínimo 7 mm [5]	DIN 53325 mínimo 7 mm
Absorción de vapor de agua	-	DIN 4843 T2 10 mg/cm² después de 8 h.
Adherencia del acabado [6]	IUF 470	IUF 470
Vacuno plena flor o levemente corregida		
-en seco	mínimo 3'0 N/cm	mínimo 3'0 N/cm
-en húmedo [7]	mínimo 2'0 N/cm	mínimo 2'0 N/cm
Vacuno flor corregida		
-en seco	mínimo 5'0 N/cm	mínimo 5'0 N/cm
-en húmedo [7]	mínimo 3'0 N/cm	mínimo 3'0 N/cm
Cueros con acabado delgado (boxcalf, napa, cabritilla...)		
-en seco	minimo 2'5 N/cm	minimo 2'0 N/cm
Cuero charol		
-en seco	mínimo 4'0 N/cm	-
-en húmedo [7]	mínimo 2'0 N/cm	-

Taula 16. Recomanacions GERIC per a la qualitat de la pell per a empenya de calçat



Directrices para empeine	GERIC [1]	Directrices Alemanas [2]
Materias extraíbles con cloruro de metileno	IUC 4 Hasta un 9 % se puede emplear un adhesivo de un solo componente. Hasta un 14 % se puede utilizar un adhesivo de dos componentes. Si hay más del 14 % se puede usar adhesivo especial de poliuretano Hasta un 7 - 8 % es posible vulcanizar. Hasta un 15 % se puede aplicar el procedimiento de inyección de PVC	DIN 53306
Solidez a la temperatura	IUF 458 Objetar reblandecimientos del acabado a temperaturas inferiores a los 80 °C. El valor mínimo de temperatura que sería recomendable que un acabado resistiera sin sufrir reblandecimiento es de 140ºC. Si esta condición no se cumpliera debe indicarse precaución en el montado.	DIN 53342
Solidez al frote	IUF 450	DIN 53339
Calzado de calle:		
-Fieltro seco y cuero húmedo. 50 frotes -Fieltro húmedo y cuero seco. 50 frotes	Sin deterioro de la capa de acabado ni de la capacidad de la superficie de ser nuevamente pulida	- Sin deterioro del aspecto
Calzado sin forro. Frotar lado carne:		
-Fieltro seco y cuero seco. 50 f.	mínimo nota 4	mínimo nota 4
-Fieltro húmedo y cuero seco. 50 frotes	mínimo nota 4	mínimo nota 4
Cuero para calzado de moda:		
-Fieltro seco y cuero seco. 50 f.	-	Sin deterioro del aspecto
-Fieltro húmedo y cuero seco. 20 frotes	-	Sin deterioro del aspecto
-Fieltro humedecido con producto de limpieza de calzado sin disolventes. 20 f.	-	Sin deterioro del aspecto
Ensayos discrecionales		
Materias minerales lavables	IUC 6 máximo 1'5 % 8	DIN 53307 máximo 1'5 %
pH del extracto acuoso 9	IUC 11 mínimo pH 3'5	DIN 53312 mínimo pH 3'5

Taula 17. Recomanacions GERIC per a la qualitat de la pell per a empenya de calçat



Directrices para empeine	GERIC [1]	Directrices Alemanas [2]
Solidez a la gota de agua	IUF 420 Ningún deterioro del acabado ni modificación del color	- No debe quedar ninguna mancha después de secar la gota de agua
Solidez a la luz	IUF 401 o IUF 402 superior a la nota 3	IUF 401 o IUF 402 como mínimo nota 3
Solidez al frote con agentes de limpieza	análogamente a IUF 450	-
Fieltro humedecido con:		
-productos con disolventes	20 frotes. Observar acabado	
-productos con es.de trementina	20 frotes. Observar acabado	
-productos acuosos	20 frotes. Observar acabado	
Capacidad de almacenamiento en la oscuridad de los cueros blancos o claros	EWG-F1 No se observará ningún amarilleamiento	-
Ácidos grasos libres [10]	BS 1309: met. 4 máximo 1 % en el cuero	-
Permeabilidad al vapor de agua (efectuar sin raspar la superficie)	IUP 15	DIN 53333
-cuero plena flor	mínimo 1.0 mg/h.cm^2	mínimo 1.0 mg/h.cm^2
-cuero flor corregida	mínimo 1.0 mg/h.cm^2	mínimo 0.8 mg/h.cm^2
-charol y cueros cubiertos con láminas	sin especificaciones	sin especificaciones
Resistencia al agua	IUP 10	DIN 53338
Cuero para calzado normal:		
-Tiempo para el 1er paso de agua	mínimo 20 minutos	-
-Absorción de agua a los 30 min.	máximo 30 % (Valores no exigibles a las pieles para calzado de moda)	-
Cuero para calzado hidrofugado:		
-Tiempo para el 1er paso de agua	mínimo 120 minutos	mínimo 120 minutos
-Absorción de agua a los 120 min	máximo 25 %	máximo 25 %

Taula 18. Recomanacions GERIC per a la qualitat de la pell per a empenya de calçat

1.4 ANNEX 4. Procediment i aparells dels anàlisis realitzats a la resina i al film

1.4.1 pH

El pH s'ha mesurat amb un pH-metre.

1.4.2 Viscositat

La viscositat s'ha determinat amb la copa Ford nº 4. Es tracta d'un recipient cilíndric foradat per un dels extrems per poder-lo omplir del líquid a analitzar. L'altre extrem del cilindre té un petit orifici.

El sistema de mesura consisteix en omplir el cilindre amb el petit orifici obturat. Al mateix moment en que l'orifici es destapa, s'encén el cronòmetre, que caldrà parar en el moment en que el recipient quedi buit. El temps total en segons, correspondrà a la viscositat del líquid en qüestió.

Figura 1. Copa Ford nº 4

Taula 19. Resultat de l'anàlisi de variància

Taula 20. Resultat de l'anàlisi d'optimització

Gràfic 1. Diagrama de superfície de resposta

1.4.3 Densitat

Per tal de determinar la densitat de les resines sintetitzades, s'ha optat per utilitzar un matràs aforat de 25mL. La mesura consisteix en omplir el matràs de resina fins a l'enràs, i anotar el pes corresponent a aquest volum. Així doncs, la densitat (g/mL) correspon al quocient del pes entre els 25mL.

1.4.4 Percentatge de Sòlids

El procés experimental per a mesurar el percentatge de sòlids, consisteix en calcular el tant per cent de la diferència de pes d'una quantitat concreta de resina (1g), abans i després d'haver estat 1 hora a l'estufa a 100ºC.

1.4.5 Brillantor

Per tal de mesurar la brillantor del film i del top aplicat a la pell, així com les solideses al fregament (en sec, humit i suor), s'ha utilitzat un espectrofotòmetre DATACOLOR Spectrum 400 TM.

Figura 2. Espectrofotòmetre utilitzat en la mesura de la brillantor i de les solideses al fregament

1.4.6 Duresa

La duresa s'ha determinat mitjançant un DURÒMETRE. L'aparell dona el resultat de la duresa en graus Shore A (ºSh A).

Figura 3. Duròmetre

1.4.7 Absorció d'aigua

Per tal de determinar l'absorció d'aigua del film, cal agafar-ne un bocí (d'1g aproximadament), pesar-lo, introduir-lo en aigua desionitzada durant 24 hores, i tornar-lo a pesar. El resultat s'ha expressat en percentatge d'absorció d'aigua:

$$\% \quad \text{Absorció} \;\; d'\text{aigua} = \frac{\text{Pes fi} - \text{Pes inici}}{\text{Pes inici}} \cdot 100$$

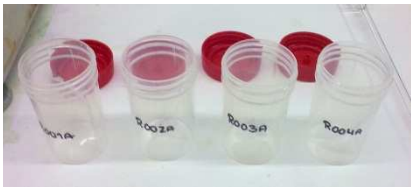


Figura 4. Preparació de les mostres

1.5 ANNEX 5. Normativa dels assajos físics per avaluar la resistència de la pell

En aquest annex es recullen els procediments per a assajos físics que s'han dut a terme en aquest treball. Se'ls anomena IUP.

Al 1947 es va fundar l'Unió Internacional d'Associacions de Químics i Tècnics de la Indústria del Cuir (**IULTCS**). Actualment, hi ha moltes altres associacions que en formen part, com per exemple, l'Associació Química Espanyola de la Indústria del Cuir (**AQEIC**). La IULTCS disposa de tres comissions d'assajos. Una d'elles és la IUP ja comentada, les altres dues són IUC, per assajos químics, i IUF per a assajos de solideses.

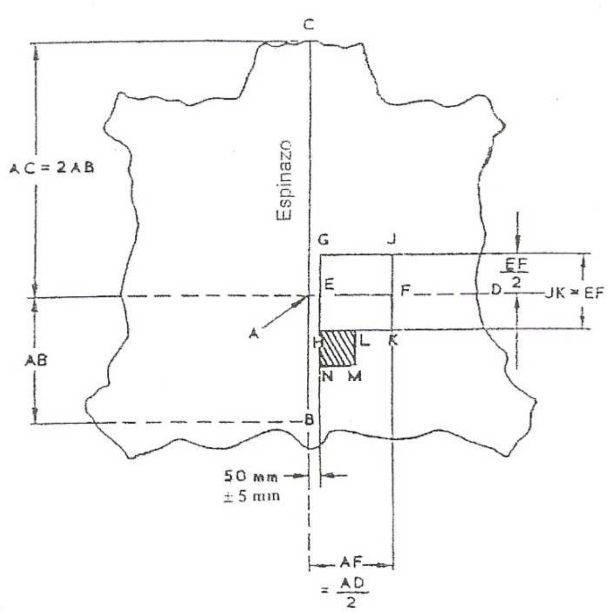
1.5.1 *Presa de Mostres (IUP-2 / IUC-2)*

Aquest mètode de la IULTCS s'ha obtingut per combinació dels mètodes originals IULTCS per a mostreig per a l'anàlisi químic (IUC-2, publicat a JSLTC, 49, 6, 1995) i l'assaig físic (IUP-2, publicat originalment a JSLTC, 42, 382, 1958).

Es tracta d'un mètode que s'aplica a tot tipus de pells i cuirs procedents de mamífers, independentment del tipus de curtició; no és aplicable a pells d'aus, peixos o rèptils.

Es tracta d'una norma que defineix la localització d'una mostra d'assaig dins d'una peça de cuir. Les zones seleccionades per prendre mostres han d'estar exemptes de qualsevol defecte òbviament visible, ja siguin rascades o talls d'escorxat.

- *Cuirs sencers de boví, pells petites (xai, cabra...) i fulles*:



B: inici de la cua.

AD: línia perpendicular a **BC**.

Les línies **GH** i **JK** són paral·leles.

AC = 2AB

AF = FD

JK = EF

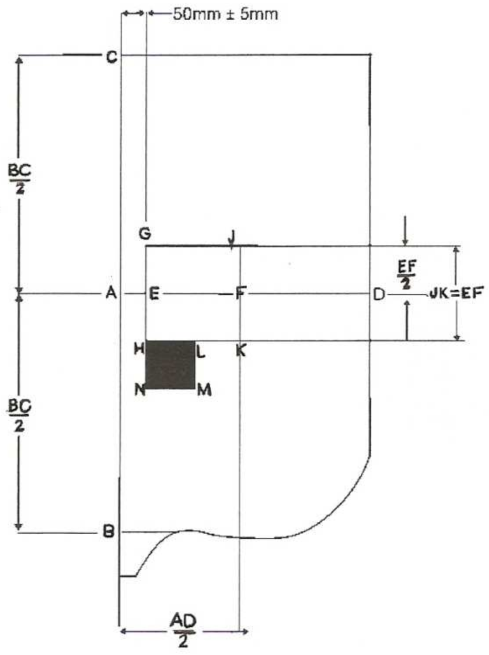
GE = EH

HL = LK = HN

AE = 50mm ± 5mm

Figura 5. Esquema de presa de mostres per cuirs sencers de boví i pells petites

- *Crupons i mitjos crupons*:



B: inici de la cua.

AD: línia perpendicular a **BC**.

Les línies **GH** i **JK** són paral·leles.

CA = BC

AF = FD

JK = EF

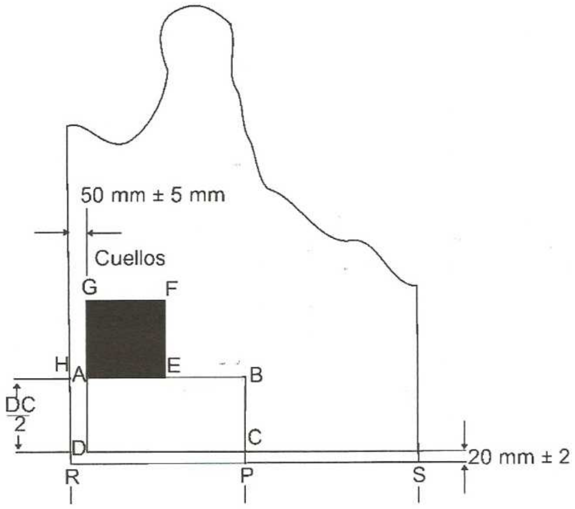
GE = EH

HL = LK = HN

AE = 50mm ± 5mm

Figura 6. Esquema de presa de mostres per crupons i mitjos crupons

- *Colls*:



DCS és una línia paral·lela a **RS**.

BCP és una línia paral·lela a l'espinada.

AB és paral·lela a **DC**.

RP = PS

DC = 2AD

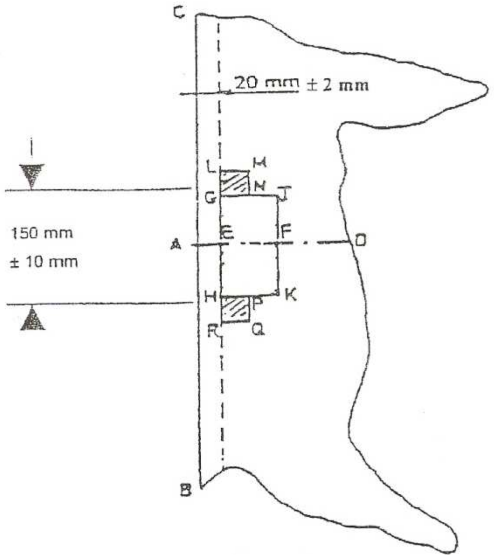
AE = EB = AG

CP = 20mm ± 2mm

AM = 50mm ± 5mm

Figura 7. Esquema de presa de mostres per colls

- *Faldes*:



AD: línia perpendicular a **BC**.

CA = AB

LG= HR = GH/4

GE = EH = EF

AE = 20mm ± 2mm

GH = 150mm ± 15mm

Figura 8. Esquema de presa de mostres per faldes

Cal emmagatzemar les provetes de manera que s'eviti la contaminació i l'escalfament localitzat. A més, a l'hora de marcar les provetes, cal diferenciar la direcció espinada mitjançant una fletxa que assenyali el cap, sobre la vora més propera a l'espinada.

1.5.2 Condicionament de les mostres (IUP-3)

Aquesta norma és una actualització de la versió declarada mètode oficial de la IULTCS (març, 2001) publicat a la revista JSLTC 82, pàgina 199, 1998. La IUP-3 especifica la preparació de les provetes de pell destinades a assajos físics i mecànics, i és aplicable a tot tipus de pells seques.

Denominació	Temperatura (ºC)	Humitat Relativa (%)
23/50	23±2	50±5

Taula 21. Condicions de temperatura i humitat determinades per la norma IUP-3

Aquest taula mostra les condicions que venen determinades per aquesta norma, tenint en compte que:

- *Atmosfera*: són les condicions ambientals definides pels paràmetres de temperatura i humitat relativa.
- *Atmosfera estàndard*: és l'atmosfera en la que es manté una proveta, dins de les toleràncies prescrites, durant un període de temps determinat, abans de ser sotmesa a un assaig.
- *Condicionat*: aquesta és la operació destinada a portar una proveta a unes condicions determinades de temperatura i humitat relativa, mitjançant el seu manteniment, durant un període de temps determinat, en una atmosfera estàndard amb lliure circulació d'aire per totes les seves superfícies.

1.5.3 Mesura de la distensió i resistència de la flor a l'assaig de l'esclat (IUP-9)

La resistència al trencament de la capa flor és el paràmetre més significatiu per a jutjar l'aptitud de la pell pel muntatge de calçat. Les directrius de qualitat per calçat especifiquen el compliment d'un mínim de 7mm, encara que per major seguretat s'hauria de superar una distensió de 8mm.

El mètode més utilitzat és el descrit a la IUP[1]-9. Aquest procediment es duu a terme amb una dispositiu anomenat ***lastòmetre,*** desenvolupat per SATRA. La prova consisteix en sotmetre una proveta circular de pell a una tensió exercida per un èmbol, tal i com es mostra en la figura següent:

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[1] **IUP,** procediments per assajos físics; **IUF**, procediments per assajos de solideses; **IUC**, procediments per assajos químics.

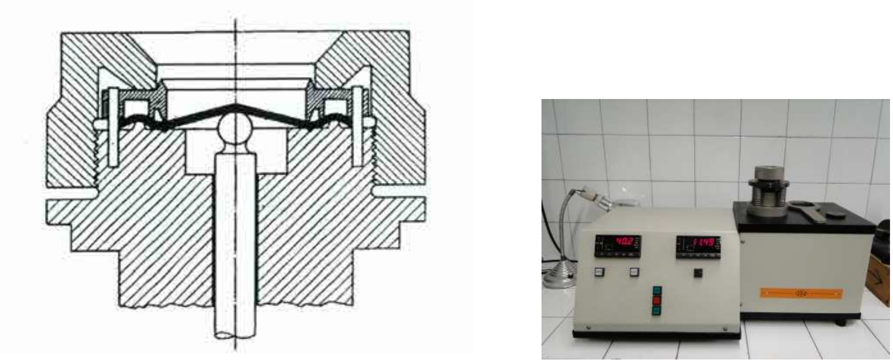


Figura 9. Esquema gràfic i imatge de l'aparell que mesura la distensió de flor d'una pell

Aquest instrument conté una abraçadora, per tal de subjectar la proveta de pell amb el costat flor encarat cap a l'exterior. A més, disposa d'un mecanisme que impulsa un èmbol a velocitat constant. L'acció ascendent de l'èmbol, situat al centre de la proveta, deforma progressivament el cuir. La proveta va adquirint una forma semblant a un con, de manera que la flor pateix una tensió continua fins a arribar al punt en que es produeix la primera fissura. En aquest precís instant, cal anotar la força exercida per l'èmbol, així com la distància en mil·límetres que ha recorregut (*distensió*).

La distensió en el moment de la primera fissura és el paràmetre més significatiu a l'hora de jutjar l'aptitud del cuir per al muntat del calçat. A **l'ANNEX 4** es troben les directrius de qualitat per a empenya de calçat.

1.5.4 *Resistència a la flexió continuada (IUP-20)*

La pell acabada utilitzada per calçat és un dels exemples més clars quan es parla de pells que a la pràctica s'exposen a una flexió continuada. L'exemple més característic és la zona de flexió de l'empenya de calçat. El defecte més comú és l'aparició d'esquerdes en l'acabat, i a vegades, també es pot observar un canvi de color degut a la pèrdua d'adhesió entre les capes de l'acabat o de la pell.

El procediment més utilitzat per analitzar el comportament de a la flexió d'un acabat és l'assaig IUP 20, "*mesura de la resistència a la flexió continuada de cuirs lleugers i el seu acabat*", i l'aparell que s'utilitza per determinar aquesta propietat és el flexòmetre.

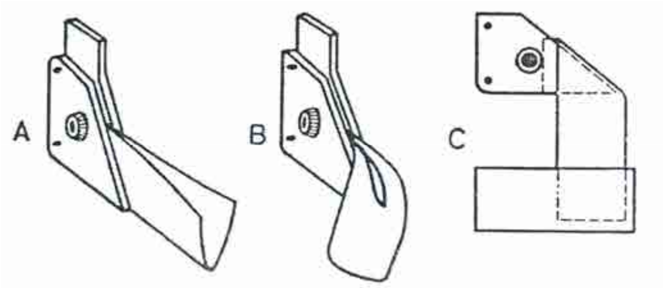


Figura 10. Esquema gràfic que mostra la col·locació de les mostres al flexòmetre

Com es pot observar a la figura, la proveta de pell se subjecta entre dues pinces. La pinça inferior és fixa, i la superior es mou endavant i endarrere en un angle de 22'5º, que ve a ser l'angle mig de flexió del peu en marxa.

Un comptador va anotant el nombre de flexions realitzades.

Per a pells destinades a climes freds és interessant realitzar la flexometria a baixes temperatures. Hi ha acabats capaços de resistir 100000 flexions a 20ºC, però que a -10ºC s'esquerden abans de les 10000. En aquests casos es recomana utilitzar resines amb una temperatura de transició (Tg) el més baixa possible.

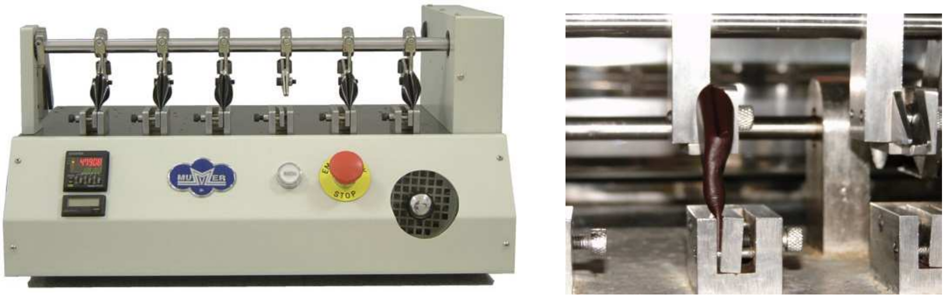


Figura 11. Flexòmetre exterior i mostra de cuir a assajar, ja col·locada en les pinces

1.5.5 *Permeabilitat al vapor d'aigua (IUP-15)*

Una de les propietats més importants de la pell destinada a calçat és la permeabilitat al vapor d'aigua. La capacitat del cuir per transmetre vapor d'aigua és, doncs, una qualitat molt important que l'acredita com a material idoni per a confecció de calçat, peces per vestir i guants.

El procediment més utilitat per la mesura de la permeabilitat del vapor d'aigua és el mètode IUP 15. En aquest mètode la proveta de pell se subjecta mitjançant una tapa roscada que té una obertura circular al voltant de la boca d'una ampolla de vidre que conté gel de sílice sec. L'única entrada possible d'aire a l'ampolla és a través del cuir.

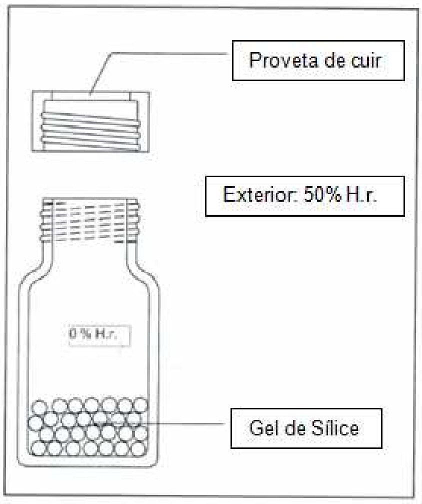


Figura 12. Esquema gràfic de la preparació de la mostra per a realitzar l'assaig de permeabilitat

L'ampolla es manté en moviment continu, i l'acció d'un ventilador assegura la renovació de l'aire. L'aire exterior de l'ampolla la temperatura i humitat relatives pròpies d'un laboratori condicionat segons IUP 3 (23 ± 2 ºC i 50 ± 5 % humitat relativa).

La proveta de pell es col·loca amb la cara que en ús pràctic serà exposada a major humitat cap a l'exterior de l'ampolla.

Després d'un període de condicionament de 20 ± 4 hores la proveta i la tapa roscada es traspassen a una segona ampolla que conté gel de sílice acabada de regenerar; es pesa el conjunt, es posa en marxa l'equip, i al cap de 11'5 ± 4 hores es torna a pesar per tal de determinar la massa de vapor que ha passat a través de la pell i ha estat absorbida pel gel de sílice.

La permeabilitat al vapor d'aigua es defineix, doncs, com la massa de vapor d'aigua transmesa pel cuir, per unitat de superfície i temps; expressada en $mg/cm^2 \cdot h$:

$$P = \frac{1{,}273 \cdot m}{d^2 \cdot t}$$

$m = increment\ de\ pes\ entre\ les\ dues\ pesades\ (mg)$
$d = diàmetre\ efectiu\ de\ la\ proveta\ (cm)$
$t = \mathrm{int}erval\ de\ temps\ entre\ les\ dues\ pesades\ (hores)$

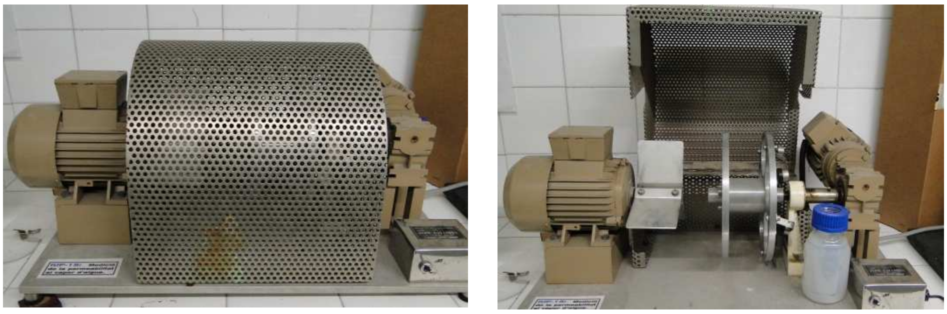


Figura 13. Aparell utilitzat per dur a terme l'assaig de permeabilitat

1.6 ANNEX 6. Normatives i procediments aplicats per avaluar les solideses de la pell

1.6.1 *Solidesa al fregament (IUF-450)*

La resistència al fregament també és una de les propietats més importants del cuir i una de les més difícils de satisfer en humit. Al cuir destinat a tapisseria i en concret per a tapisseria d'automòbil, se li exigeixen unes solideses molt elevades, que només poden complir acabats molt reticulats. Gairebé tots els tipus de curtits estan obligats a complir un grau determinat de resistència al fregament.

Hi ha dos tipus d'assaig per a mesurar la solidesa al fregament: el Satra i el Veslic. En aquest estudi s'ha utilitzat el Veslic, ja que és el que va ser adoptat com a mètode IUF 450, i és el més utilitat.

Al mètode IUF 450, la mostra de pell es fixa amb la cara a assajar cap amunt, sobre la plataforma horitzontal capaç de desenvolupar un moviment de vaivé amb un recorregut de 3,5cm i una freqüència de 40 cicles per minut. La mostra s'estira un 10% de la seva longitud, en la mateixa direcció en la que s'efectua el moviment. El feltre, de llana i de forma quadrada, s'aplica sobre la superfície del cuir amb una càrrega ajustable. La càrrega mínima és de 500g, encara que aquesta només s'aplica en cuirs afelpats. La càrrega normal és d'1kg.El nombre de cicles dependrà de les exigències de l'article concret (oscil·la entre 20 i 2000).
Normalment, es realitzen dos assajos, un amb el feltre sec, i un altre amb el feltre humit. També es pot dur a terme amb el feltre humidificat amb suor artificial, dissolvents, productes de neteja, o altres substàncies representatives de situacions en particular.

Una vegada realitzat l'assaig, el feltre pot quedar més o menys colorejat a causa de la transferència de qualsevol tipus de matèria colorejada, com pot ser colorant o pols d'esmeril.

A més del color, la superfície del cuir pot quedar alterada. Les variacions de color es valoren amb l'ajuda de l'escala de grisos (tant del cuir com del feltre). Com sempre, la nota 5 correspon a màxima solidesa i la nota 1 a la més baixa. És important que els feltres humits es valorin una vegada secs.

En la valoració del cuir és oportú anotar: pèrdua de brillantor, l'efecte de pulit, l'aixafat de la felpa, o el deteriorament de l'acabat.

Figura 14. Aparell utilitzat en la determinació de la solidesa al fregament

1.6.2 *Solidesa a la gota d'aigua (IUF-420)*

Es tracta d'un dels assajos més importants per a confecció i marroquineria. El procediment consisteix en determinar el temps necessari per a la penetració d'una gota d'aigua de 0,15mL dipositada sobre la superfície de la pell. Una vegada la pell s'ha assecat, es valora l'aspecte de la pell, examinant la possible formació de l'aureola, taca, variació del color, inflament o pèrdua de brillo. La norma que descriu aquest mètode i la manera de valorar els resultats és la IUF 420.

Per a mesurar els resultats es fa servir l'escala de grisos.

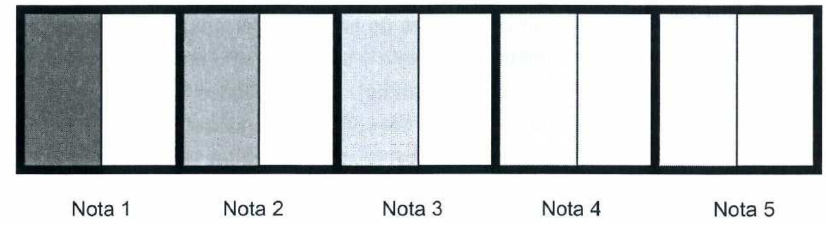
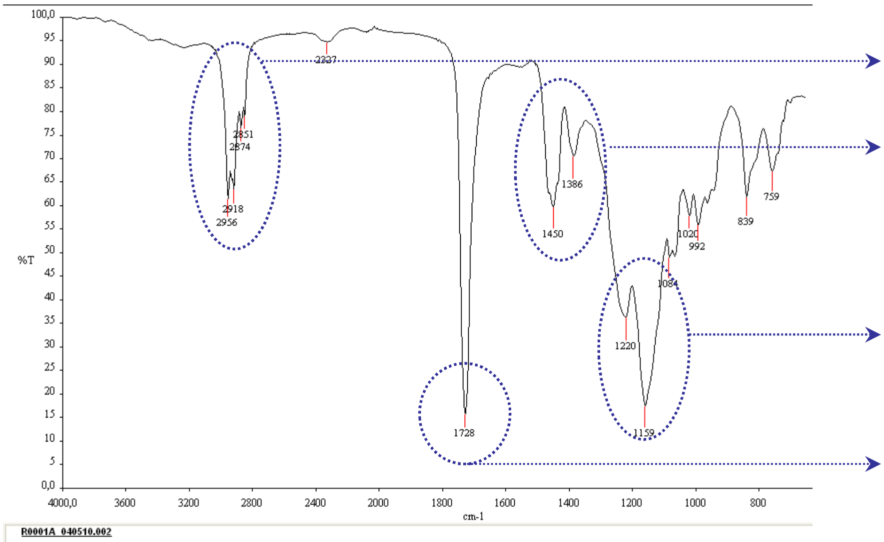


Figura 15. Escala de grisos utilitzada en l'acotació dels resultats

1.7 ANNEX 7. IR de les resines sintetitzades

1.7.1 Resines amb grups carboxílics

A continuació, es troben els IR corresponents a les 4 resines funcionalitzades amb grups carboxílics: R001A.



2800-3000cm^{-1} – CH –

Butil Acrilat

$CH2=CHCOO(CH_2)_3CH_3$

Grup Acrilat

$CH_2=CHCOOR_1$

Grup carboxílic de l'éster

– COO^-

Figura 16. Gràfic IR de la resina R001A

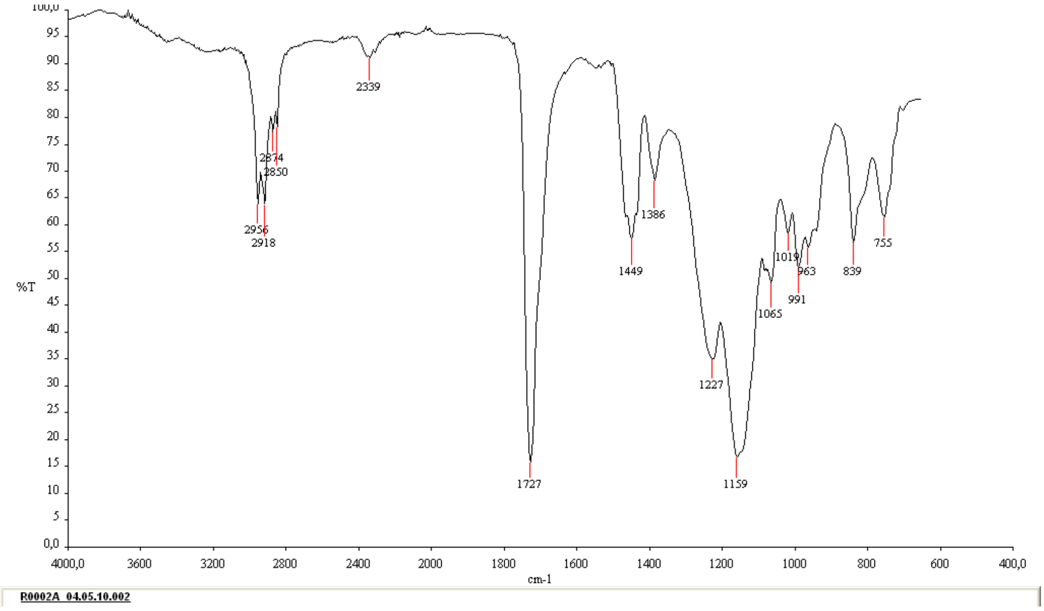


Figura 17. Gràfic IR de la resina R002A

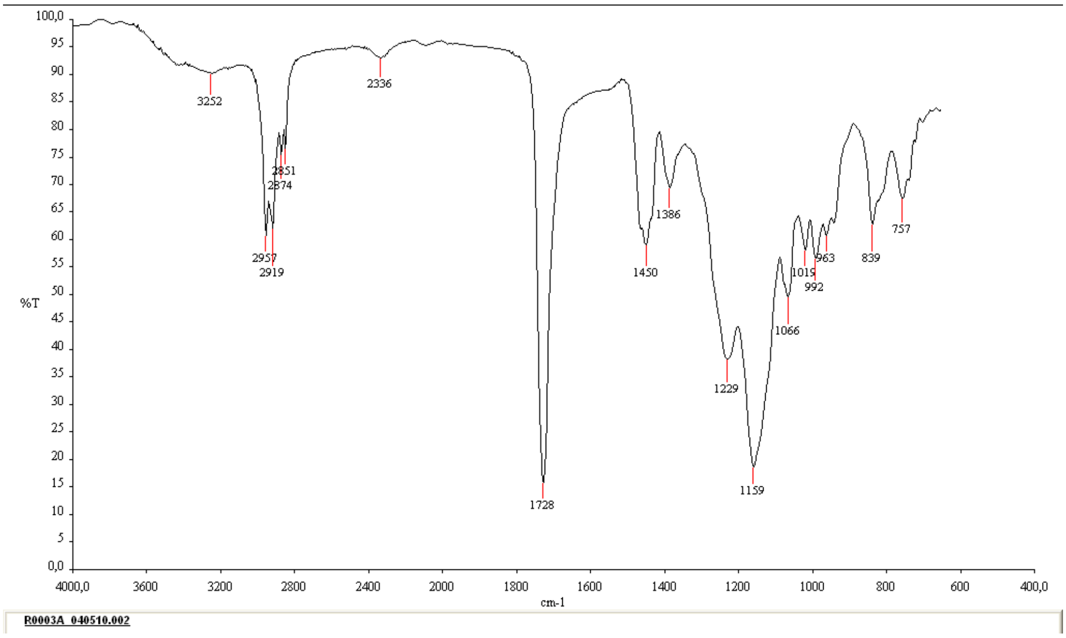


Figura 18. Gràfic IR de la resina R003A

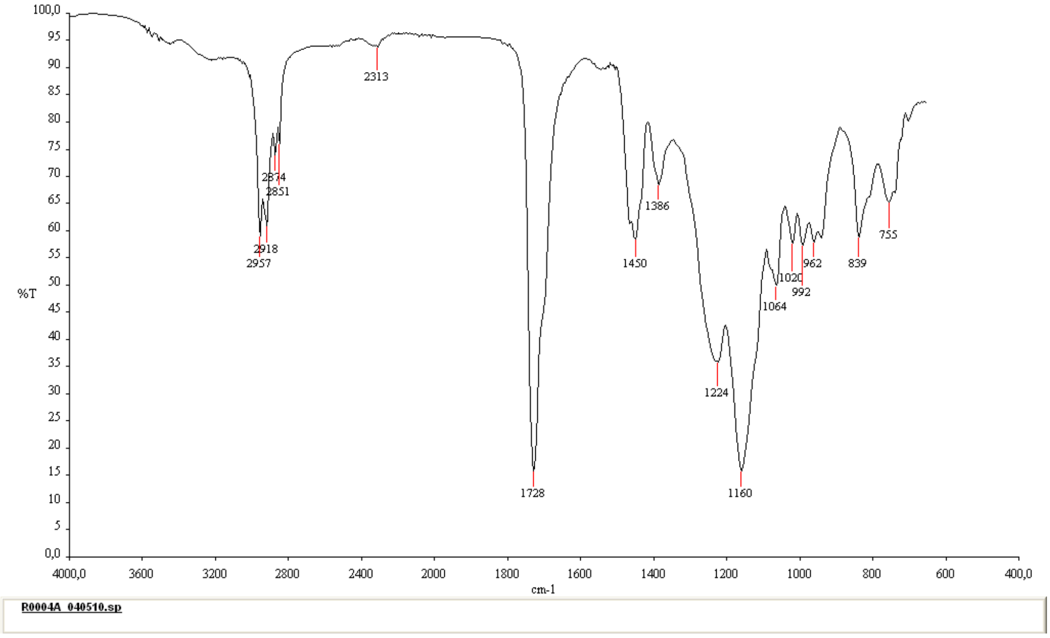


Figura 19. Gràfic IR de la resina R004A

1.7.2 Resines amb grups epoxi

A continuació, es troben els IR corresponents a les 4 resines funcionalitzades amb grups epoxi: R005A, R006A, R007A i R008A.

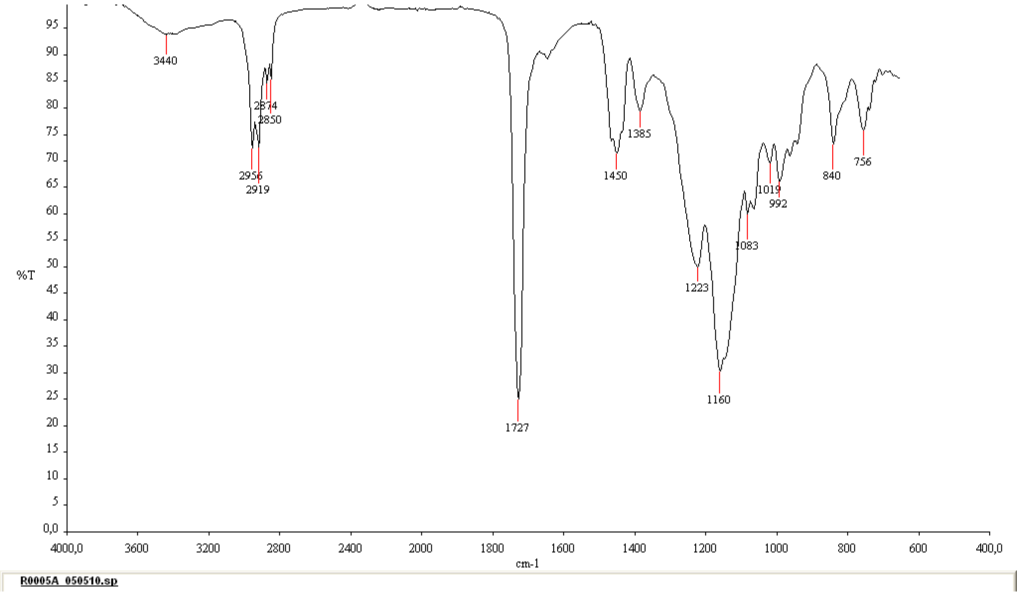


Figura 20. Gràfic IR de la resina R005A

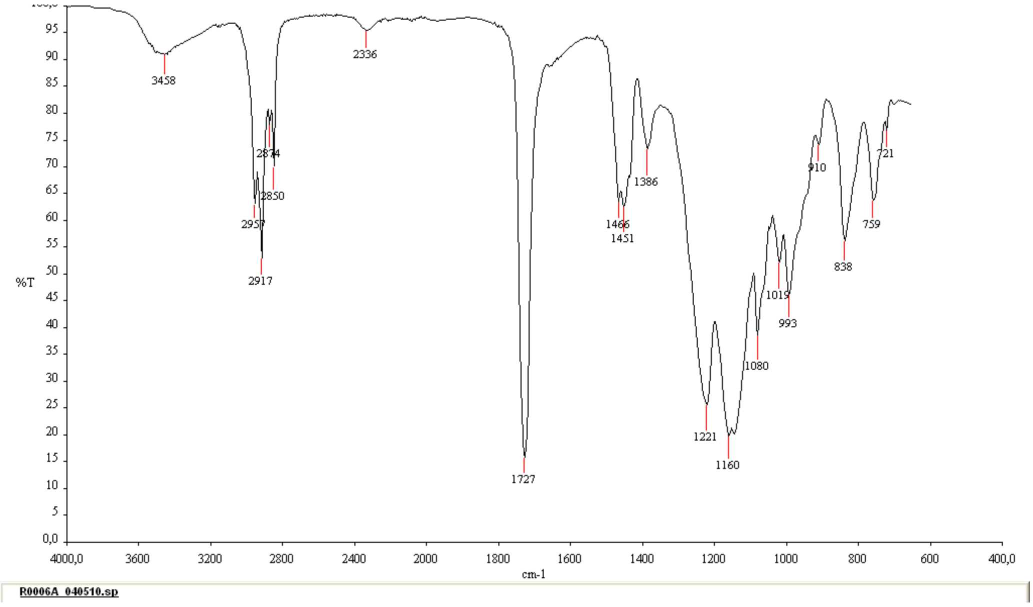


Figura 21. Gràfic IR de la resina R006A

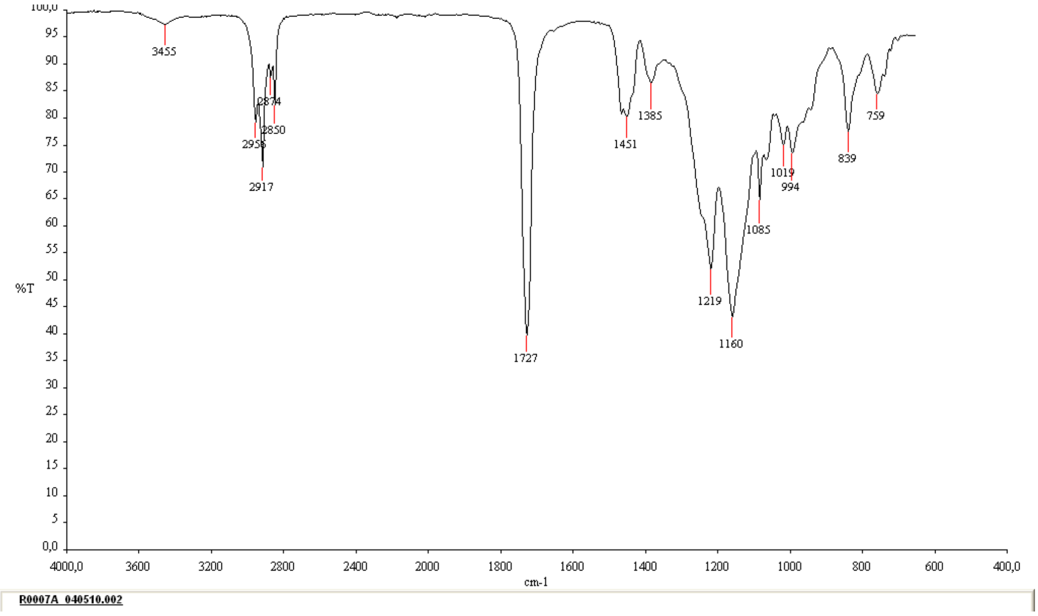


Figura 22. Gràfic IR de la resina R007A

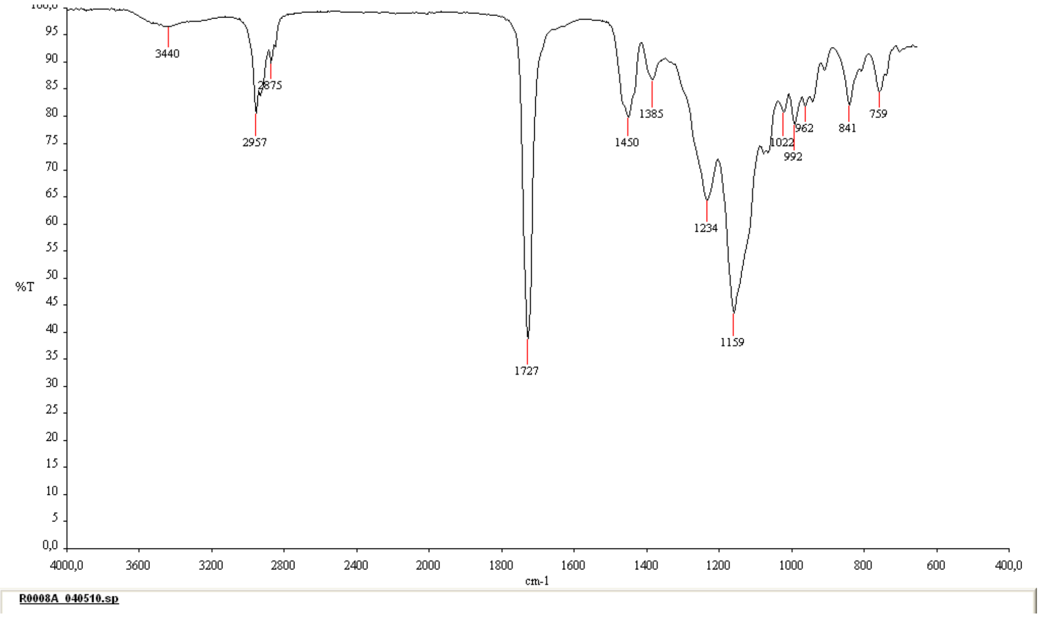


Figura 23. Gràfic IR de la resina R008A

1.7.3 Resines amb grups amínics

A continuació, es troben els IR corresponents a les 4 resines funcionalitzades amb grups amínics: R015A, R016A, R017A i R018A.

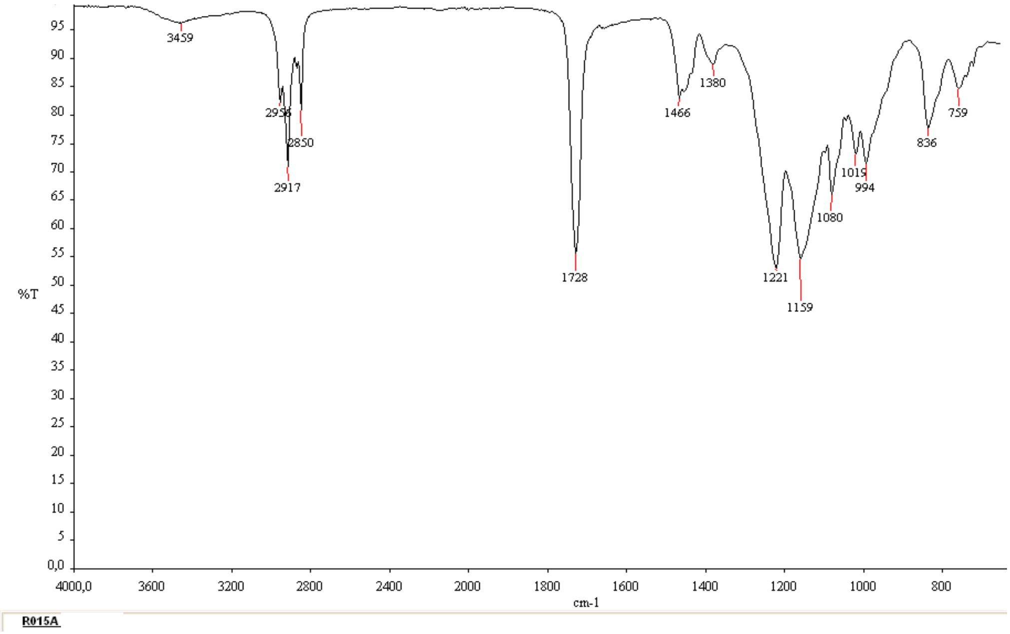


Figura 24. Gràfic IR de la resina R015A

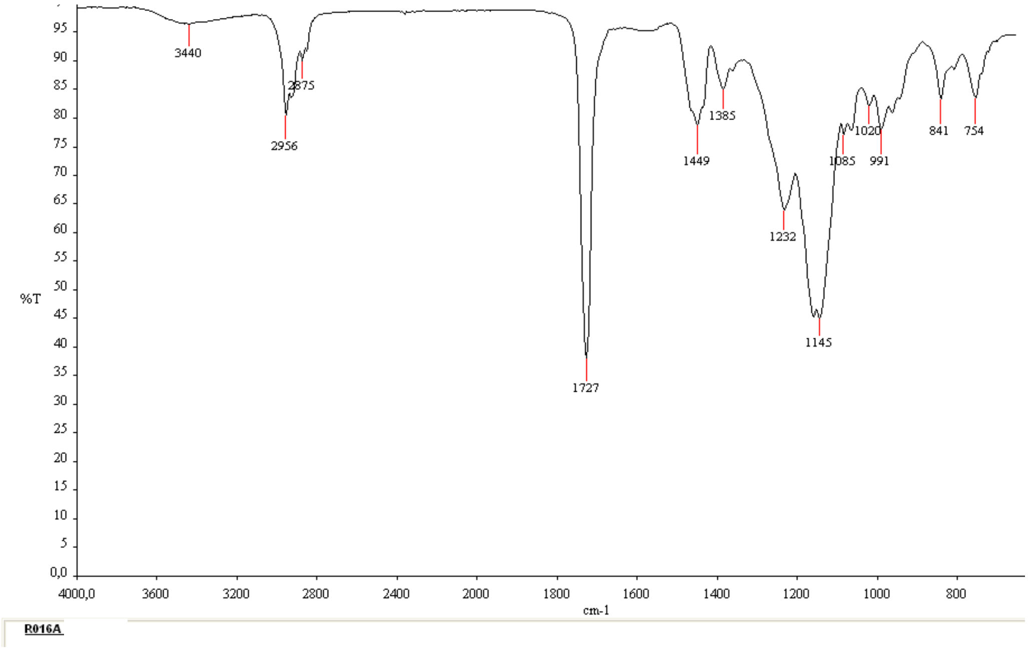


Figura 25. Gràfic IR de la resina R016A

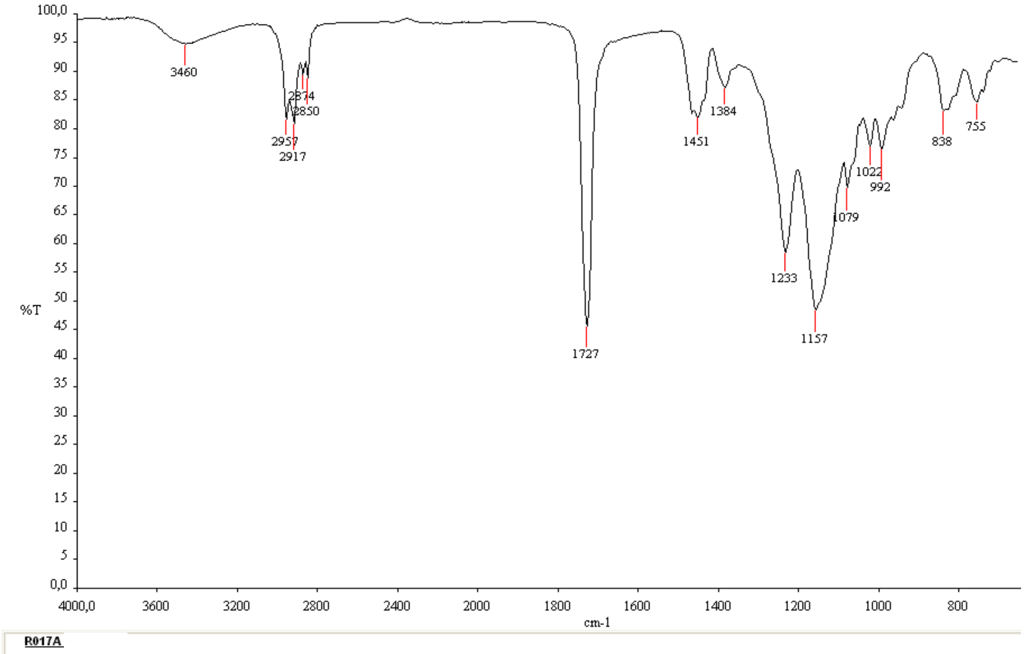


Figura 26. Gràfic IR de la resina R017A

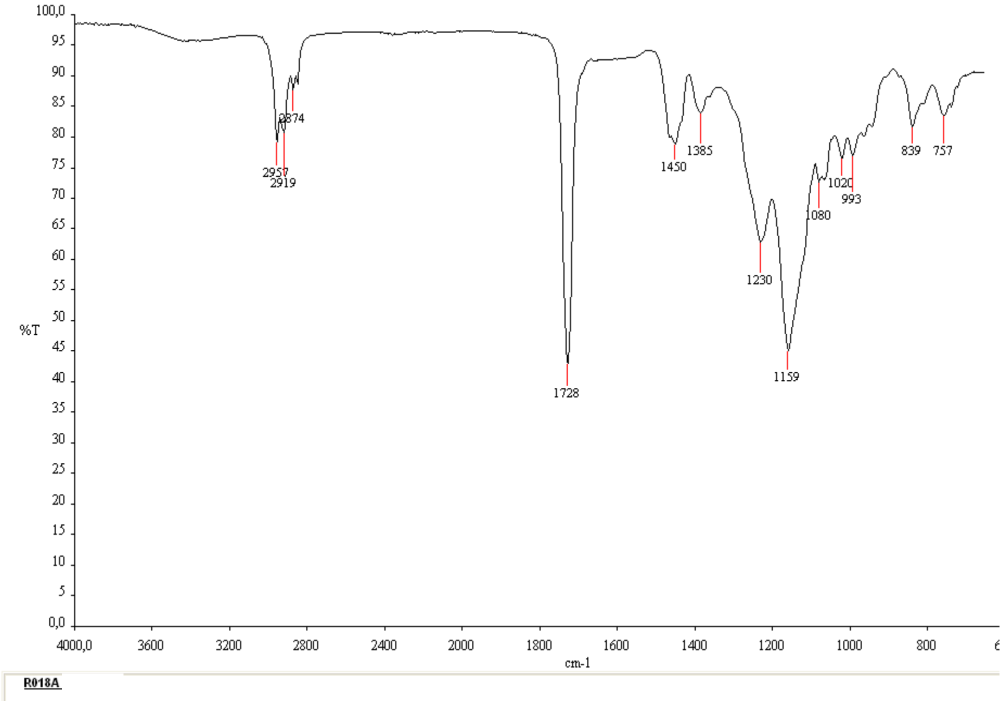


Figura 27. Gràfic IR de la resina R018A

1.7.4 Barreges de resines: epoxi-carboxílic (EC), epoxi-amínic (EA)

A continuació, es troben 2 IR que corresponen a la primera barreja EC i a la primera barreja EA, respectivament.

	EC1	EC2	EC3	EC4	EC5	EC6	EC7	EC8	EC9	EC10	EC11	EC12
R seleccionada - COOH (grams)	3	3	15	15	9	9	0	18	9	9	9	9
R seleccionada - EPOXI (grams)	29	171	29	171	0	200	100	100	100	100	100	100

Taula 22. Proporcions de resina corresponents a les barreges de resina Carboxílica i resina Epoxi

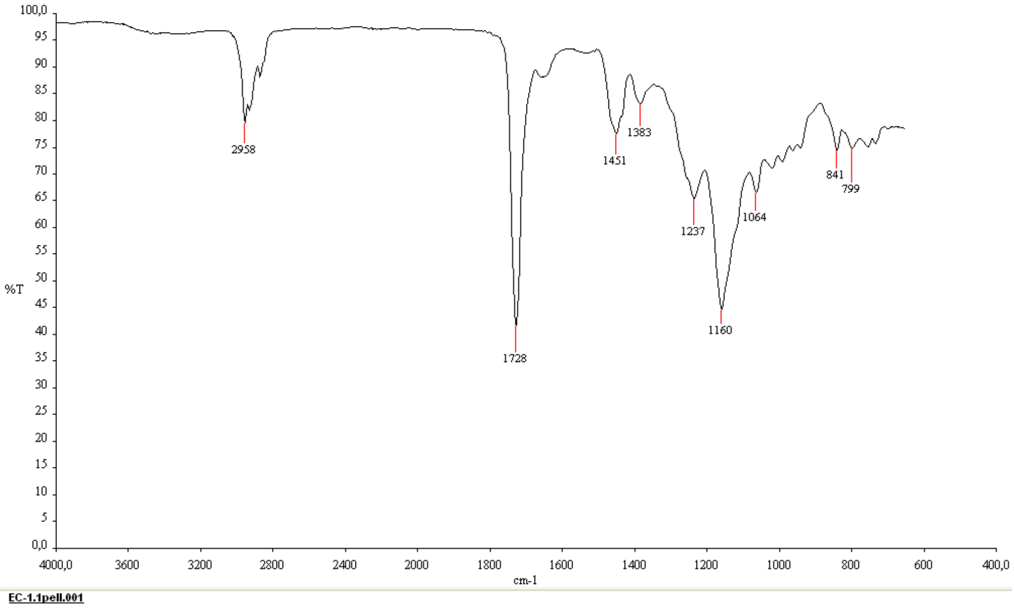


Figura 28. Gràfic IR de la barreja Epoxi-Carboxílica 1 (EC1)

	EA1	EA 2	EA 3	EA 4	EA5	EA6	EA7	EA8	EA9	EA10	EA11	EA12
R seleccionada - AMIN (grams)	29	29	171	171	100	100	0	200	100	100	100	100
R seleccionada - EPOXI (grams)	22	128	22	128	0	150	75	75	75	75	75	75

Taula 23. Proporcions de resina corresponents a les barreges de resina Amínica i resina Epoxi

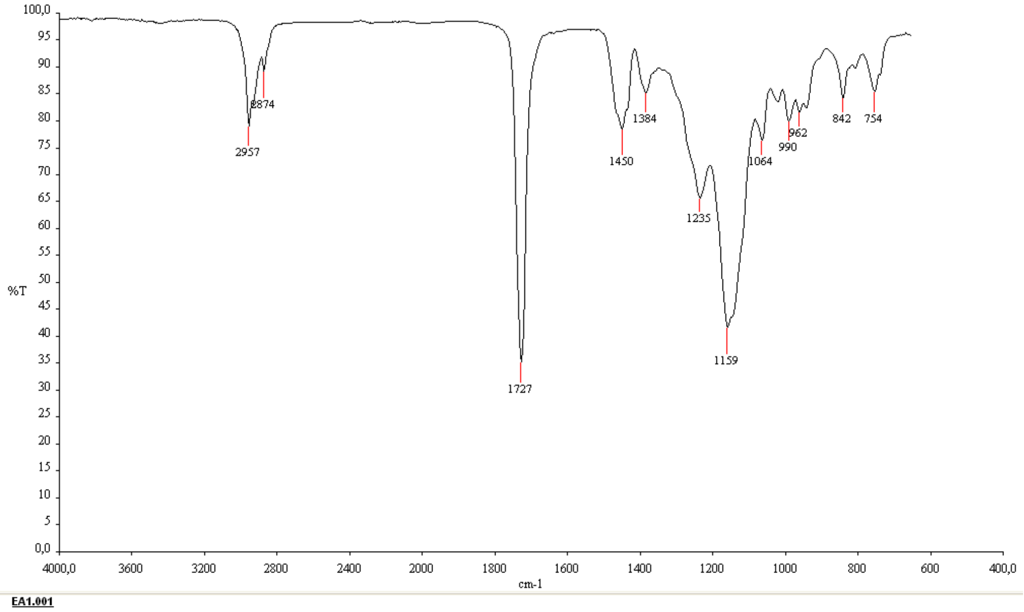


Figura 29. Gràfic IR de la barreja Epoxi-Amínica 1 (EA1)

1.8 ANNEX 8. Anàlisi matemàtic dels resultats

(Programa informàtic d'anàlisi estadístic: Statgraphics)

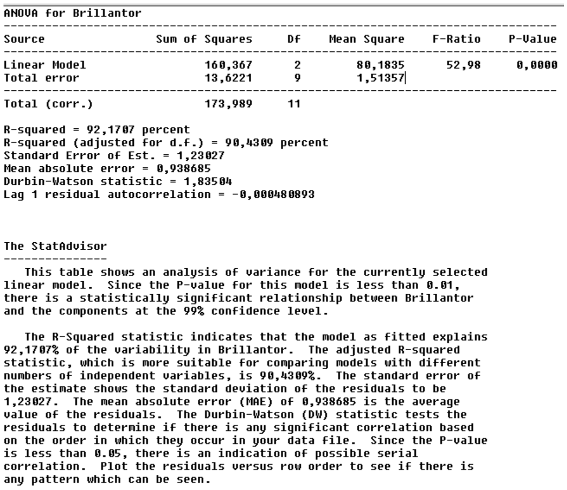
S'ha dut a terme l'anàlisi estadístic de totes les variables estudiades. En total s'han analitzat 10 variables, una a una: duresa, distensió flor, frec sec (100 frecs), frec sec (500 frecs), frec humit (25 frecs), frec humit (50 frecs), gota d'aigua, permeabilitat al vapor d'aigua, absorció d'aigua, brillo del top. Degut a l'elevat nombre de variables a analitzar, s'ha considerat excessiu incloure l'anàlisi estadístic de totes les variables. Per aquest motiu, només s'inclouran els anàlisis estadístics de dues variables de cada bloc. No és considera necessari incloure tots els resultats ja que l'objectiu és donar a conèixer la manera d'expressar els resultats de l'*Statgraphics*.

1.8.1 Anàlisis dels resultats de les resines funcionalitzades amb grups carboxílics

		R001A	**R002A**	**R003A**	**R004A**
Brillantor (%)		5.3 ± 0.2	15.2 ± 0.6	6.7 ± 0.4	9.8 ± 0.4
Flexió sec (nº)		100000	100000	100000	100000
Flexió humit (nº)		50000	50000	50000	50000
Fregament Sec (nota)	**500**	4.5	4.5	4.5	5.0
Fregament Humit (nota)	**50**	2.0	4.0	2.5	4.0
Distensió Flor (mm)		14.7 ± 0.4	17.3 ± 0.2	14.3 ± 1.2	15.8 ± 1.2
Gota d'aigua (temps)		5min ± 1	12min ± 1	>30min ± 0	>30min ± 0
Permeabilitat vapor aigua (%)		1.02 ± 0.04	1.38 ± 0.04	1.09 ± 0.07	0.72 ± 0.06
Tacking		De + a - : R001A, R003A, i R002A = R004A			
Tacte pell		De + suau a -: 3,2,4,1			

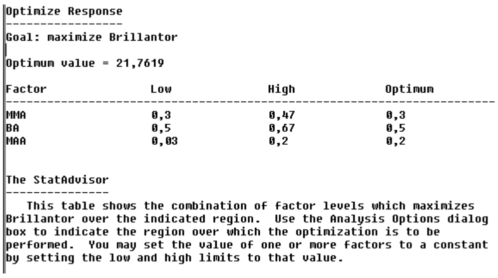
Taula 24. Resum de resultats de les resines funcionalitzades amb grups carboxílics

- **BRILLANTOR**



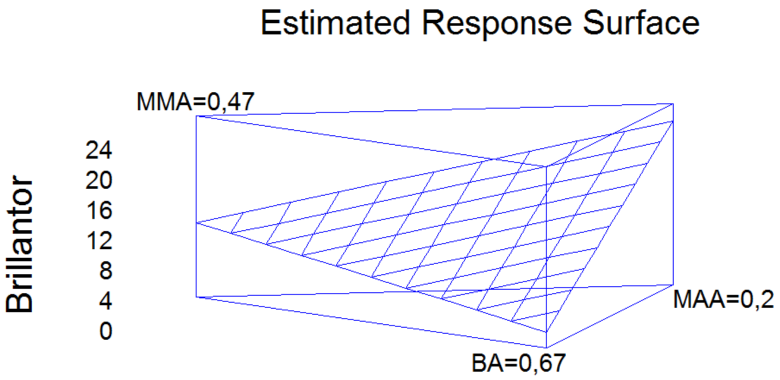
```
ANOVA for Brillantor
--------------------------------------------------------------------------------
Source                  Sum of Squares     Df    Mean Square    F-Ratio    P-Value
--------------------------------------------------------------------------------
Linear Model                   160,367      2        80,1835      52,98     0,0000
Total error                    13,6221      9        1,51357
--------------------------------------------------------------------------------
Total (corr.)                  173,989     11

R-squared = 92,1707 percent
R-squared (adjusted for d.f.) = 90,4309 percent
Standard Error of Est. = 1,23027
Mean absolute error = 0,938685
Durbin-Watson statistic = 1,83504
Lag 1 residual autocorrelation = -0,000480893


The StatAdvisor
---------------
   This table shows an analysis of variance for the currently selected
linear model.  Since the P-value for this model is less than 0.01,
there is a statistically significant relationship between Brillantor
and the components at the 99% confidence level.

   The R-Squared statistic indicates that the model as fitted explains
92,1707% of the variability in Brillantor.  The adjusted R-squared
statistic, which is more suitable for comparing models with different
numbers of independent variables, is 90,4309%.  The standard error of
the estimate shows the standard deviation of the residuals to be
1,23027.  The mean absolute error (MAE) of 0,938685 is the average
value of the residuals.  The Durbin-Watson (DW) statistic tests the
residuals to determine if there is any significant correlation based
on the order in which they occur in your data file.  Since the P-value
is less than 0.05, there is an indication of possible serial
correlation.  Plot the residuals versus row order to see if there is
any pattern which can be seen.
```

Taula 25. Resultat de l'anàlisi de variància



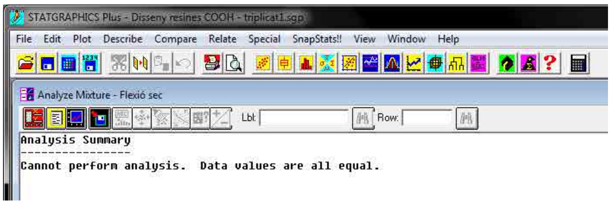
```
Optimize Response
-----------------
Goal: maximize Brillantor

Optimum value = 21,7619

Factor               Low               High              Optimum
-----------------------------------------------------------------------
MMA                  0,3               0,47              0,3
BA                   0,5               0,67              0,5
MAA                  0,03              0,2               0,2


The StatAdvisor
---------------
   This table shows the combination of factor levels which maximizes
Brillantor over the indicated region.  Use the Analysis Options dialog
box to indicate the region over which the optimization is to be
performed.  You may set the value of one or more factors to a constant
by setting the low and high limits to that value.
```

Taula 26. Resultat de l'anàlisi d'optimització



Gràfic 2. Diagrama de superfície de resposta

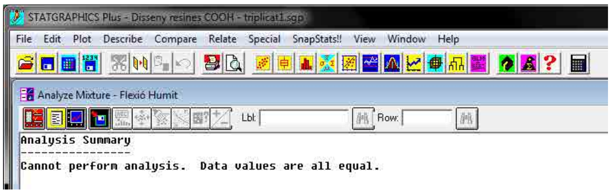
- ## FLEXIÓ SEC



```
Analysis Summary
----------------
Cannot perform analysis.  Data values are all equal.
```

Figura 30. No hi ha anàlisi, ja que tots els resultats tenen el mateix valor

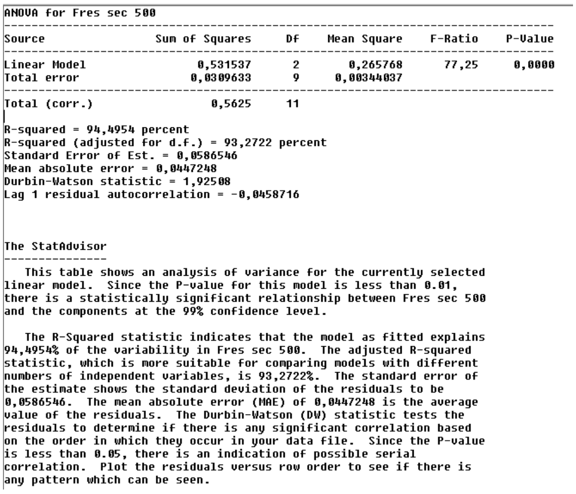
- ## FLEXIÓ HUMIT



```
Analysis Summary
----------------
Cannot perform analysis.  Data values are all equal.
```

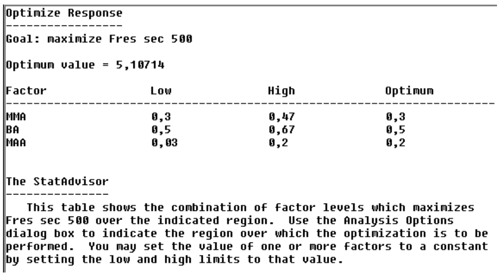
Figura 31. No hi ha anàlisi, ja que tots els resultats tenen el mateix valor

- ## FREC SEC 500



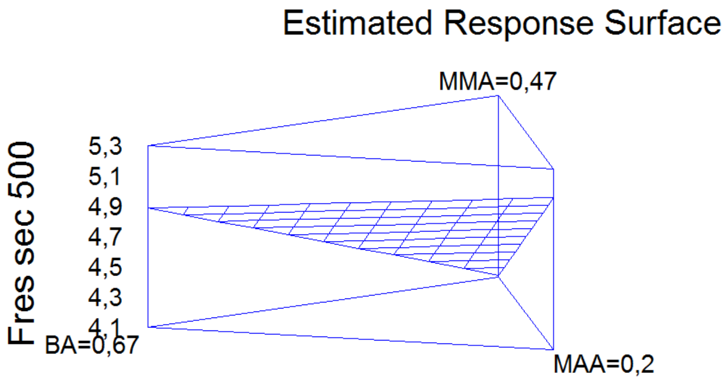
```
ANOVA for Fres sec 500
--------------------------------------------------------------------------------
Source                 Sum of Squares     Df    Mean Square    F-Ratio    P-Value
--------------------------------------------------------------------------------
Linear Model                 0,531537      2       0,265768      77,25     0,0000
Total error                 0,0309633      9     0,00344037
--------------------------------------------------------------------------------
Total (corr.)                  0,5625     11

R-squared = 94,4954 percent
R-squared (adjusted for d.f.) = 93,2722 percent
Standard Error of Est. = 0,0586546
Mean absolute error = 0,0447248
Durbin-Watson statistic = 1,92508
Lag 1 residual autocorrelation = -0,0458716



The StatAdvisor
---------------
   This table shows an analysis of variance for the currently selected
linear model.  Since the P-value for this model is less than 0.01,
there is a statistically significant relationship between Fres sec 500
and the components at the 99% confidence level.

   The R-Squared statistic indicates that the model as fitted explains
94,4954% of the variability in Fres sec 500.  The adjusted R-squared
statistic, which is more suitable for comparing models with different
numbers of independent variables, is 93,2722%.  The standard error of
the estimate shows the standard deviation of the residuals to be
0,0586546.  The mean absolute error (MAE) of 0,0447248 is the average
value of the residuals.  The Durbin-Watson (DW) statistic tests the
residuals to determine if there is any significant correlation based
on the order in which they occur in your data file.  Since the P-value
is less than 0.05, there is an indication of possible serial
correlation.  Plot the residuals versus row order to see if there is
any pattern which can be seen.
```

Taula 27. Resultat de l'anàlisi de variància



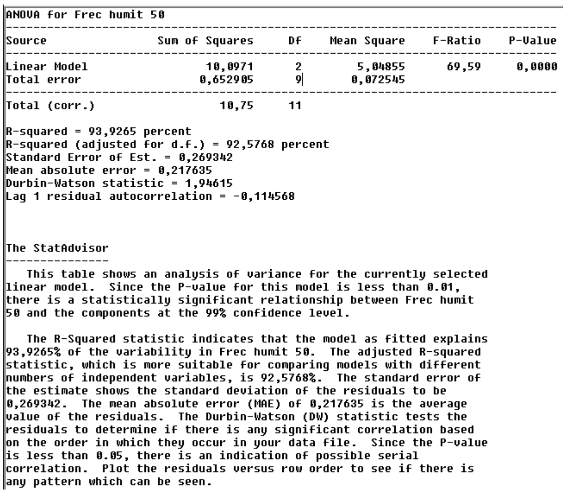
```
Optimize Response
-----------------
Goal: maximize Fres sec 500

Optimum value = 5,10714

Factor                Low              High              Optimum
-----------------------------------------------------------------------
MMA                   0,3              0,47              0,3
BA                    0,5              0,67              0,5
MAA                   0,03             0,2               0,2


The StatAdvisor
---------------
   This table shows the combination of factor levels which maximizes
Fres sec 500 over the indicated region.  Use the Analysis Options
dialog box to indicate the region over which the optimization is to be
performed.  You may set the value of one or more factors to a constant
by setting the low and high limits to that value.
```

Taula 28. Resultat de l'anàlisi d'optimització



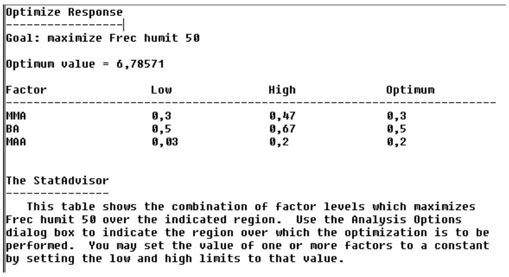
Gràfic 3. Diagrama de superfície de resposta

- ### FREC HUMIT 50



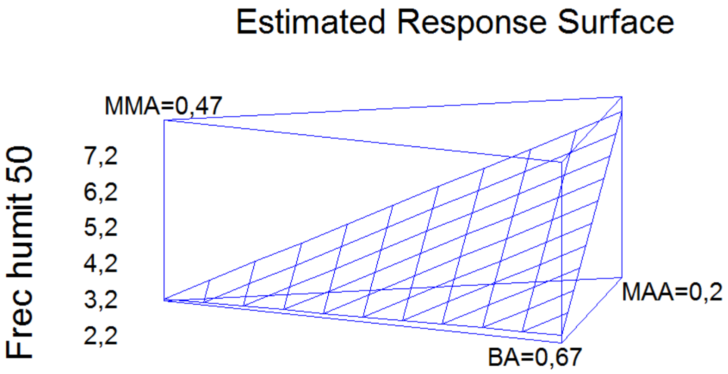
```
ANOVA for Frec humit 50
-----------------------------------------------------------------------------
Source                    Sum of Squares     Df     Mean Square     F-Ratio     P-Value
-----------------------------------------------------------------------------
Linear Model                     10,0971      2         5,04855       69,59      0,0000
Total error                     0,652905      9|       0,072545
-----------------------------------------------------------------------------
Total (corr.)                      10,75     11

R-squared = 93,9265 percent
R-squared (adjusted for d.f.) = 92,5768 percent
Standard Error of Est. = 0,269342
Mean absolute error = 0,217635
Durbin-Watson statistic = 1,94615
Lag 1 residual autocorrelation = -0,114568



The StatAdvisor
---------------
   This table shows an analysis of variance for the currently selected
linear model.  Since the P-value for this model is less than 0.01,
there is a statistically significant relationship between Frec humit
50 and the components at the 99% confidence level.

   The R-Squared statistic indicates that the model as fitted explains
93,9265% of the variability in Frec humit 50.  The adjusted R-squared
statistic, which is more suitable for comparing models with different
numbers of independent variables, is 92,5768%.  The standard error of
the estimate shows the standard deviation of the residuals to be
0,269342.  The mean absolute error (MAE) of 0,217635 is the average
value of the residuals.  The Durbin-Watson (DW) statistic tests the
residuals to determine if there is any significant correlation based
on the order in which they occur in your data file.  Since the P-value
is less than 0.05, there is an indication of possible serial
correlation.  Plot the residuals versus row order to see if there is
any pattern which can be seen.
```

Taula 29. Resultat de l'anàlisi de variància



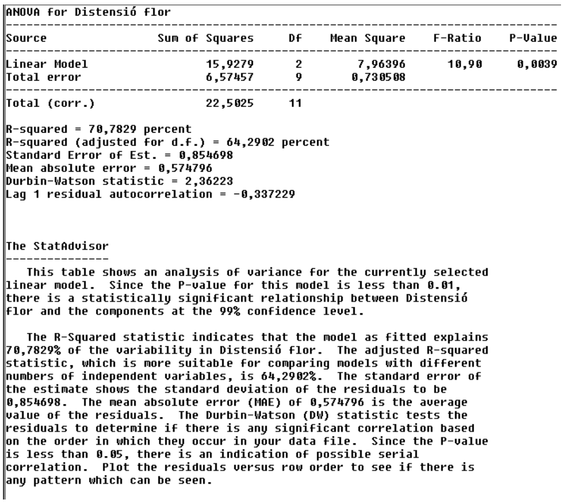
```
Optimize Response
-----------------
Goal: maximize Frec humit 50

Optimum value = 6,78571

Factor                Low              High             Optimum
-----------------------------------------------------------------------
MMA                   0,3              0,47             0,3
BA                    0,5              0,67             0,5
MAA                   0,03             0,2              0,2


The StatAdvisor
---------------
   This table shows the combination of factor levels which maximizes
Frec humit 50 over the indicated region.  Use the Analysis Options
dialog box to indicate the region over which the optimization is to be
performed.  You may set the value of one or more factors to a constant
by setting the low and high limits to that value.
```

Taula 30. Resultat de l'anàlisi d'optimització



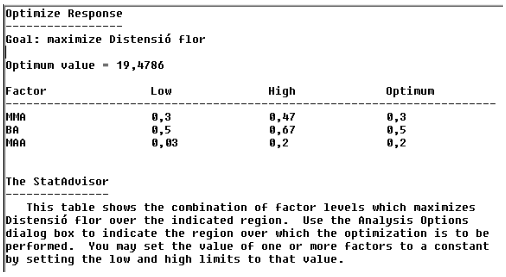
Gràfic 4. Diagrama de superfície de resposta

- ## DISTENSIÓ FLOR



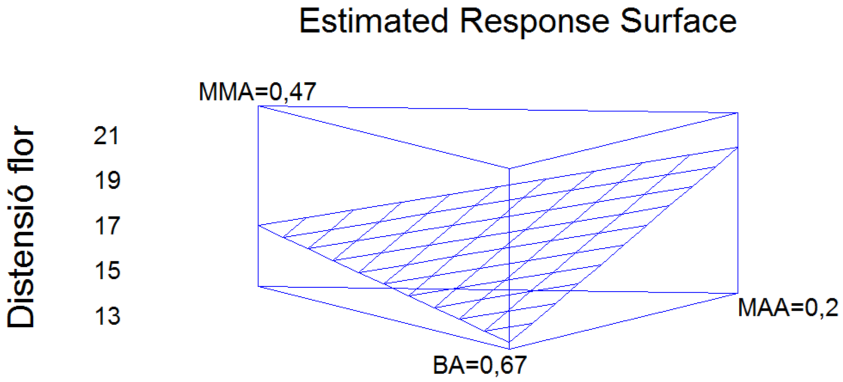
```
ANOVA for Distensió flor
-----------------------------------------------------------------------------
Source                 Sum of Squares     Df    Mean Square    F-Ratio    P-Value
-----------------------------------------------------------------------------
Linear Model                  15,9279      2        7,96396      10,90     0,0039
Total error                   6,57457      9       0,730508
-----------------------------------------------------------------------------
Total (corr.)                 22,5025     11

R-squared = 70,7829 percent
R-squared (adjusted for d.f.) = 64,2902 percent
Standard Error of Est. = 0,854698
Mean absolute error = 0,574796
Durbin-Watson statistic = 2,36223
Lag 1 residual autocorrelation = -0,337229



The StatAdvisor
---------------
   This table shows an analysis of variance for the currently selected
linear model.  Since the P-value for this model is less than 0.01,
there is a statistically significant relationship between Distensió
flor and the components at the 99% confidence level.

   The R-Squared statistic indicates that the model as fitted explains
70,7829% of the variability in Distensió flor.  The adjusted R-squared
statistic, which is more suitable for comparing models with different
numbers of independent variables, is 64,2902%.  The standard error of
the estimate shows the standard deviation of the residuals to be
0,854698.  The mean absolute error (MAE) of 0,574796 is the average
value of the residuals.  The Durbin-Watson (DW) statistic tests the
residuals to determine if there is any significant correlation based
on the order in which they occur in your data file.  Since the P-value
is less than 0.05, there is an indication of possible serial
correlation.  Plot the residuals versus row order to see if there is
any pattern which can be seen.
```

Taula 31. Resultat de l'anàlisi de variància



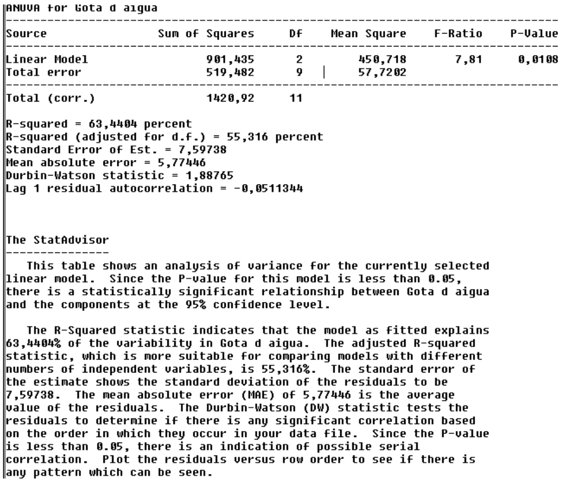
```
Optimize Response
-----------------
Goal: maximize Distensió flor

Optimum value = 19,4786

Factor                Low               High              Optimum
-----------------------------------------------------------------------
MMA                   0,3               0,47              0,3
BA                    0,5               0,67              0,5
MAA                   0,03              0,2               0,2


The StatAdvisor
---------------
   This table shows the combination of factor levels which maximizes
Distensió flor over the indicated region.  Use the Analysis Options
dialog box to indicate the region over which the optimization is to be
performed.  You may set the value of one or more factors to a constant
by setting the low and high limits to that value.
```

Taula 32. Resultat de l'anàlisi d'optimització



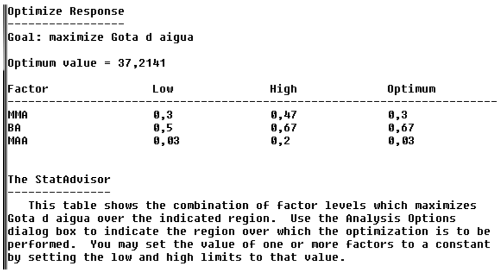
Gràfic 5. Diagrama de superfície de resposta

- ### GOTA D'AIGUA



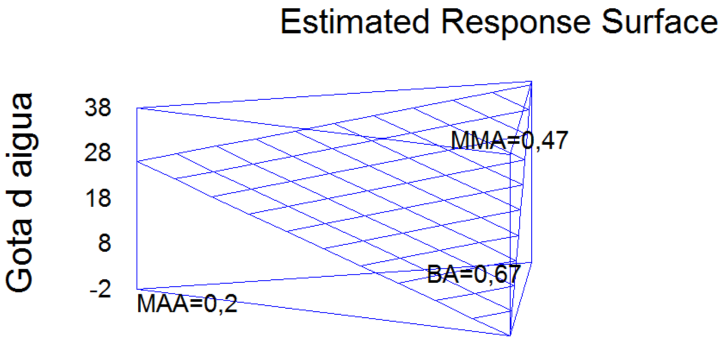
```
ANOVA for Gota d aigua
-------------------------------------------------------------------------------
Source                   Sum of Squares     Df     Mean Square    F-Ratio    P-Value
-------------------------------------------------------------------------------
Linear Model                    901,435      2         450,718       7,81     0,0108
Total error                     519,482      9    |    57,7202
-------------------------------------------------------------------------------
Total (corr.)                   1420,92     11

R-squared = 63,4404 percent
R-squared (adjusted for d.f.) = 55,316 percent
Standard Error of Est. = 7,59738
Mean absolute error = 5,77446
Durbin-Watson statistic = 1,88765
Lag 1 residual autocorrelation = -0,0511344



The StatAdvisor
---------------
   This table shows an analysis of variance for the currently selected
linear model.  Since the P-value for this model is less than 0.05,
there is a statistically significant relationship between Gota d aigua
and the components at the 95% confidence level.

   The R-Squared statistic indicates that the model as fitted explains
63,4404% of the variability in Gota d aigua.  The adjusted R-squared
statistic, which is more suitable for comparing models with different
numbers of independent variables, is 55,316%.  The standard error of
the estimate shows the standard deviation of the residuals to be
7,59738.  The mean absolute error (MAE) of 5,77446 is the average
value of the residuals.  The Durbin-Watson (DW) statistic tests the
residuals to determine if there is any significant correlation based
on the order in which they occur in your data file.  Since the P-value
is less than 0.05, there is an indication of possible serial
correlation.  Plot the residuals versus row order to see if there is
any pattern which can be seen.
```

Taula 33. Resultat de l'anàlisi de variància



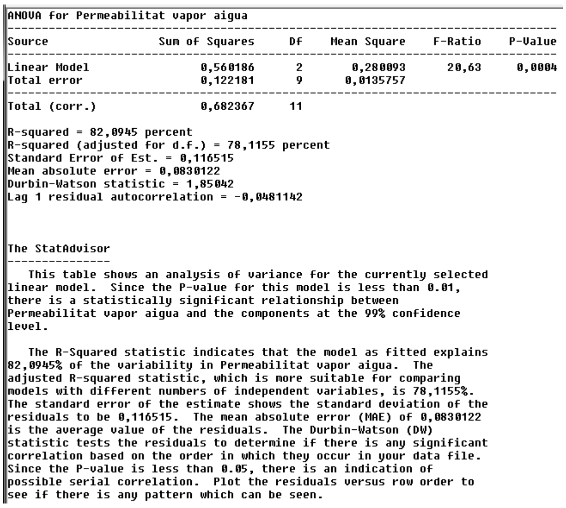
```
Optimize Response
-----------------
Goal: maximize Gota d aigua

Optimum value = 37,2141

Factor               Low                 High                Optimum
-----------------------------------------------------------------------
MMA                  0,3                 0,47                0,3
BA                   0,5                 0,67                0,67
MAA                  0,03                0,2                 0,03


The StatAdvisor
---------------
   This table shows the combination of factor levels which maximizes
Gota d aigua over the indicated region.  Use the Analysis Options
dialog box to indicate the region over which the optimization is to be
performed.  You may set the value of one or more factors to a constant
by setting the low and high limits to that value.
```

Taula 34. Resultat de l'anàlisi d'optimització



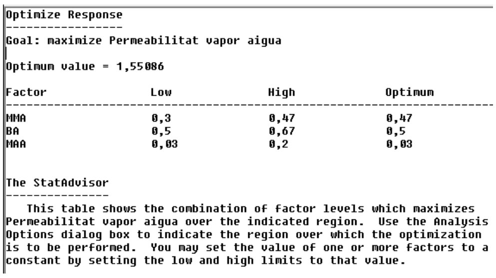
Gràfic 6. Diagrama de superfície de resposta

PERMEABILITAT AL VAPOR D'AIGUA



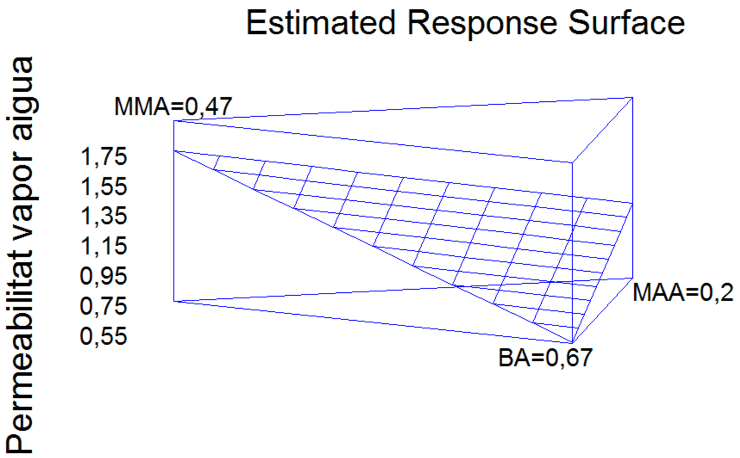
```
ANOVA for Permeabilitat vapor aigua
-----------------------------------------------------------------------------
Source                  Sum of Squares     Df     Mean Square    F-Ratio    P-Value
-----------------------------------------------------------------------------
Linear Model                  0,560186      2        0,280093      20,63     0,0004
Total error                   0,122181      9       0,0135757
-----------------------------------------------------------------------------
Total (corr.)                 0,682367     11

R-squared = 82,0945 percent
R-squared (adjusted for d.f.) = 78,1155 percent
Standard Error of Est. = 0,116515
Mean absolute error = 0,0830122
Durbin-Watson statistic = 1,85042
Lag 1 residual autocorrelation = -0,0481142



The StatAdvisor
---------------
   This table shows an analysis of variance for the currently selected
linear model.  Since the P-value for this model is less than 0.01,
there is a statistically significant relationship between
Permeabilitat vapor aigua and the components at the 99% confidence
level.

   The R-Squared statistic indicates that the model as fitted explains
82,0945% of the variability in Permeabilitat vapor aigua.  The
adjusted R-squared statistic, which is more suitable for comparing
models with different numbers of independent variables, is 78,1155%.
The standard error of the estimate shows the standard deviation of the
residuals to be 0,116515.  The mean absolute error (MAE) of 0,0830122
is the average value of the residuals.  The Durbin-Watson (DW)
statistic tests the residuals to determine if there is any significant
correlation based on the order in which they occur in your data file.
Since the P-value is less than 0.05, there is an indication of
possible serial correlation.  Plot the residuals versus row order to
see if there is any pattern which can be seen.
```

Taula 35. Resultat de l'anàlisi de variància



```
Optimize Response
-----------------
Goal: maximize Permeabilitat vapor aigua

Optimum value = 1,55086

Factor               Low               High              Optimum
--------------------------------------------------------------------
MMA                  0,3               0,47              0,47
BA                   0,5               0,67              0,5
MAA                  0,03              0,2               0,03


The StatAdvisor
---------------
   This table shows the combination of factor levels which maximizes
Permeabilitat vapor aigua over the indicated region.  Use the Analysis
Options dialog box to indicate the region over which the optimization
is to be performed.  You may set the value of one or more factors to a
constant by setting the low and high limits to that value.
```

Taula 36. Resultat de l'anàlisi d'optimització



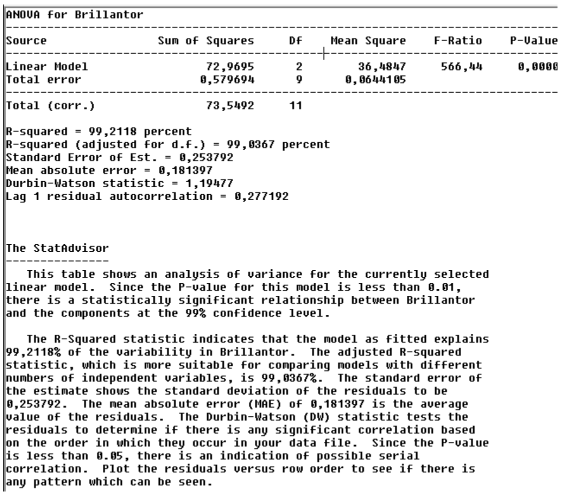
Gràfic 7. Diagrama de superfície de resposta

1.8.2 Anàlisis dels resultats de les resines funcionalitzades amb grups epoxi

		R005A	R006A	R007A	R008A
Brillantor (%)		5.2 ± 0.3	10.5 ± 0.2	3.9 ± 0.2	7.0 ± 0.3
Flexió sec (nº)		100000	100000	100000	100000
Flexió humit (nº)		50000	50000	50000	50000
Fregament Sec (nota)	**500**	5.0	4.0	5.0	5.0
Fregament Humit (nota)	**50**	5.0	3.5	4.5	3.5
Distensió Flor (mm)		11.8 ± 0.4	12.2 ± 0.5	19.5 ± 0.3	15.4 ± 0.4
Gota d'aigua (temps)		>30min ± 0	>30min ± 0	>30min ± 0	>30min ± 0
Permeabilitat vapor aigua (%)		0.74 ± 0.04	0.63 ± 0.02	1.83 ± 0.03	1.36 ± 0.02
Tacking		De + a - : R008A = R005A, R007A = R006A			
Tacte pell		Totes tenen un tacte molt frenant degut al tacking. Fet que es pot solucionar afegint silicona a la formulació del Top.			

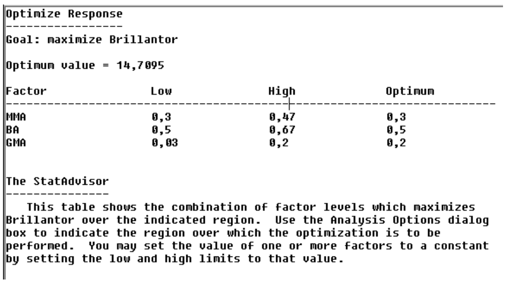
Taula 37. Resum de resultats de les resines funcionalitzades amb grups epoxi

- **BRILLANTOR**



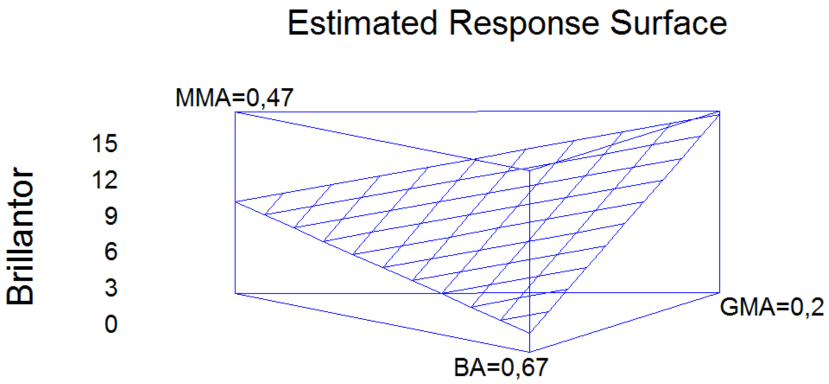
```
ANOVA for Brillantor
-----------------------------------------------------------------------------
Source                    Sum of Squares     Df    Mean Square    F-Ratio    P-Value
-----------------------------------------------------------------------------
Linear Model                     72,9695      2        36,4847     566,44     0,0000
Total error                     0,579694      9      0,0644105
-----------------------------------------------------------------------------
Total (corr.)                    73,5492     11

R-squared = 99,2118 percent
R-squared (adjusted for d.f.) = 99,0367 percent
Standard Error of Est. = 0,253792
Mean absolute error = 0,181397
Durbin-Watson statistic = 1,19477
Lag 1 residual autocorrelation = 0,277192



The StatAdvisor
---------------
   This table shows an analysis of variance for the currently selected
linear model.  Since the P-value for this model is less than 0.01,
there is a statistically significant relationship between Brillantor
and the components at the 99% confidence level.

   The R-Squared statistic indicates that the model as fitted explains
99,2118% of the variability in Brillantor.  The adjusted R-squared
statistic, which is more suitable for comparing models with different
numbers of independent variables, is 99,0367%.  The standard error of
the estimate shows the standard deviation of the residuals to be
0,253792.  The mean absolute error (MAE) of 0,181397 is the average
value of the residuals.  The Durbin-Watson (DW) statistic tests the
residuals to determine if there is any significant correlation based
on the order in which they occur in your data file.  Since the P-value
is less than 0.05, there is an indication of possible serial
correlation.  Plot the residuals versus row order to see if there is
any pattern which can be seen.
```

Taula 38. Resultat de l'anàlisi de variància



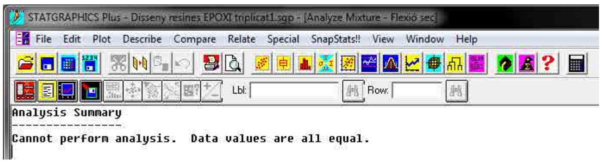
```
Optimize Response
-----------------
Goal: maximize Brillantor

Optimum value = 14,7095

Factor                Low                High               Optimum
-----------------------------------------------------------------------
MMA                   0,3                0,47               0,3
BA                    0,5                0,67               0,5
GMA                   0,03               0,2                0,2


The StatAdvisor
---------------
   This table shows the combination of factor levels which maximizes
Brillantor over the indicated region.  Use the Analysis Options dialog
box to indicate the region over which the optimization is to be
performed.  You may set the value of one or more factors to a constant
by setting the low and high limits to that value.
```

Taula 39. Resultat de l'anàlisi d'optimització



Gràfic 8. Diagrama de superfície de resposta

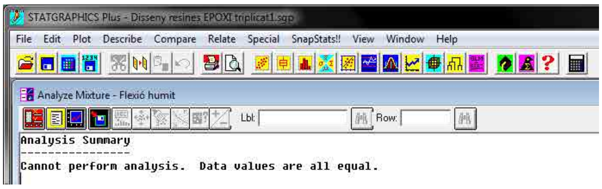
- ## FLEXIÓ SEC



```
Analysis Summary
----------------
Cannot perform analysis.  Data values are all equal.
```

Figura 32. No hi ha anàlisi, ja que tots els resultats tenen el mateix valor

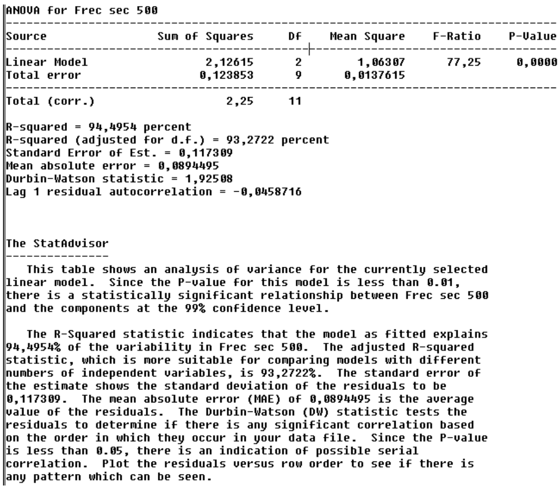
- ## FLEXIÓ HUMIT



```
Analysis Summary
----------------
Cannot perform analysis.  Data values are all equal.
```

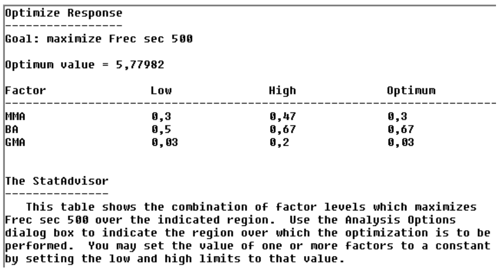
Figura 33. No hi ha anàlisi, ja que tots els resultats tenen el mateix valor

- ## FREC SEC 500



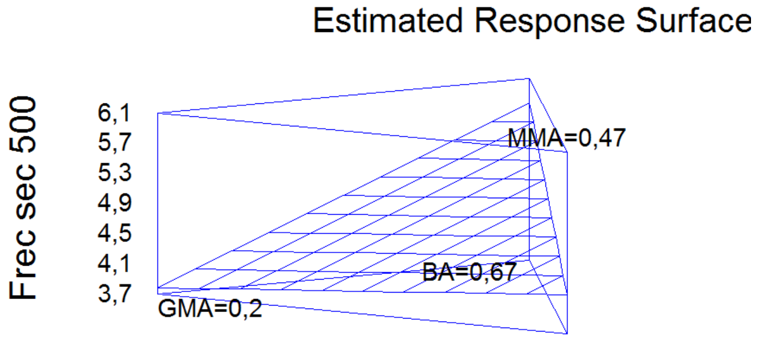
```
ANOVA for Frec sec 500
-----------------------------------------------------------------------------
Source                  Sum of Squares     Df    Mean Square    F-Ratio     P-Value
-----------------------------------------------------------------------------
Linear Model                  2,12615       2        1,06307      77,25      0,0000
Total error                  0,123853       9      0,0137615
-----------------------------------------------------------------------------
Total (corr.)                    2,25      11

R-squared = 94,4954 percent
R-squared (adjusted for d.f.) = 93,2722 percent
Standard Error of Est. = 0,117309
Mean absolute error = 0,0894495
Durbin-Watson statistic = 1,92508
Lag 1 residual autocorrelation = -0,0458716



The StatAdvisor
---------------
   This table shows an analysis of variance for the currently selected
linear model.  Since the P-value for this model is less than 0.01,
there is a statistically significant relationship between Frec sec 500
and the components at the 99% confidence level.

   The R-Squared statistic indicates that the model as fitted explains
94,4954% of the variability in Frec sec 500.  The adjusted R-squared
statistic, which is more suitable for comparing models with different
numbers of independent variables, is 93,2722%.  The standard error of
the estimate shows the standard deviation of the residuals to be
0,117309.  The mean absolute error (MAE) of 0,0894495 is the average
value of the residuals.  The Durbin-Watson (DW) statistic tests the
residuals to determine if there is any significant correlation based
on the order in which they occur in your data file.  Since the P-value
is less than 0.05, there is an indication of possible serial
correlation.  Plot the residuals versus row order to see if there is
any pattern which can be seen.
```

Taula 40. Resultat de l’anàlisi de variància



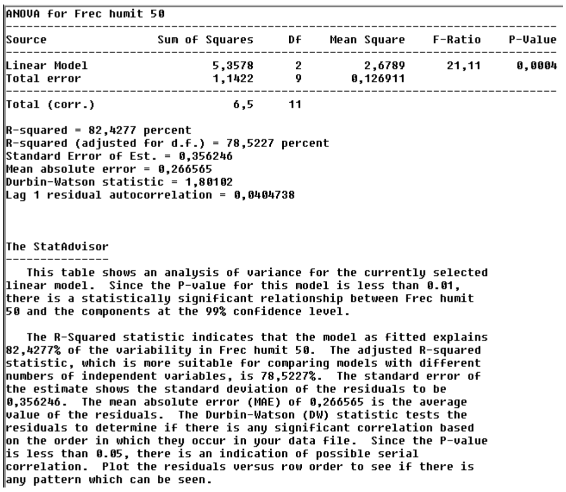
```
Optimize Response
-----------------
Goal: maximize Frec sec 500

Optimum value = 5,77982

Factor                  Low                High                Optimum
-----------------------------------------------------------------------------
MMA                     0,3                0,47                0,3
BA                      0,5                0,67                0,67
GMA                     0,03               0,2                 0,03


The StatAdvisor
---------------
   This table shows the combination of factor levels which maximizes
Frec sec 500 over the indicated region.  Use the Analysis Options
dialog box to indicate the region over which the optimization is to be
performed.  You may set the value of one or more factors to a constant
by setting the low and high limits to that value.
```

Taula 41. Resultat de l’anàlisi d’optimització



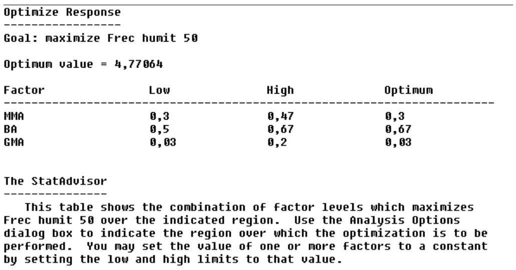
Gràfic 9. Diagrama de superfície de resposta

- **FREC HUMIT 50**



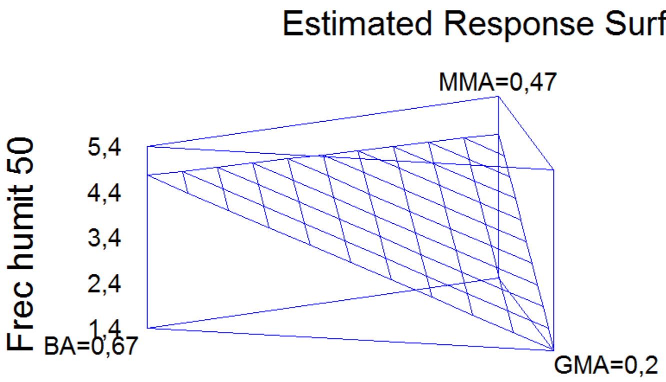
```
ANOVA for Frec humit 50
--------------------------------------------------------------------------------
Source                    Sum of Squares     Df    Mean Square    F-Ratio    P-Value
--------------------------------------------------------------------------------
Linear Model                      5,3578      2         2,6789      21,11     0,0004
Total error                       1,1422      9       0,126911
--------------------------------------------------------------------------------
Total (corr.)                        6,5     11

R-squared = 82,4277 percent
R-squared (adjusted for d.f.) = 78,5227 percent
Standard Error of Est. = 0,356246
Mean absolute error = 0,266565
Durbin-Watson statistic = 1,80102
Lag 1 residual autocorrelation = 0,0404738



The StatAdvisor
---------------
   This table shows an analysis of variance for the currently selected
linear model.  Since the P-value for this model is less than 0.01,
there is a statistically significant relationship between Frec humit
50 and the components at the 99% confidence level.

   The R-Squared statistic indicates that the model as fitted explains
82,4277% of the variability in Frec humit 50.  The adjusted R-squared
statistic, which is more suitable for comparing models with different
numbers of independent variables, is 78,5227%.  The standard error of
the estimate shows the standard deviation of the residuals to be
0,356246.  The mean absolute error (MAE) of 0,266565 is the average
value of the residuals.  The Durbin-Watson (DW) statistic tests the
residuals to determine if there is any significant correlation based
on the order in which they occur in your data file.  Since the P-value
is less than 0.05, there is an indication of possible serial
correlation.  Plot the residuals versus row order to see if there is
any pattern which can be seen.
```

Taula 42. Resultat de l'anàlisi de variància



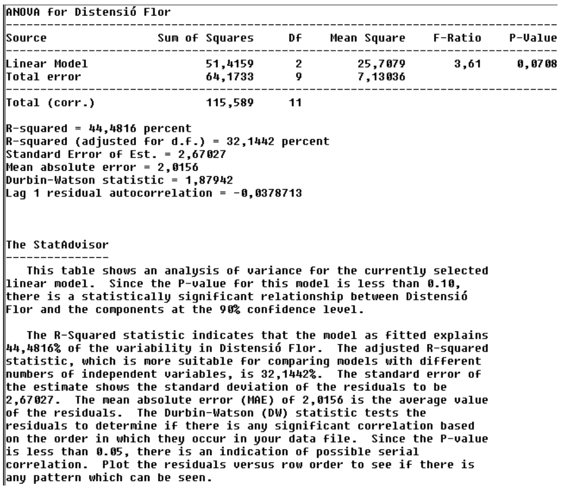
```
Optimize Response
-----------------
Goal: maximize Frec humit 50

Optimum value = 4,77064

Factor                   Low                 High                Optimum
-----------------------------------------------------------------------------
MMA                      0,3                 0,47                0,3
BA                       0,5                 0,67                0,67
GMA                      0,03                0,2                 0,03


The StatAdvisor
---------------
   This table shows the combination of factor levels which maximizes
Frec humit 50 over the indicated region.  Use the Analysis Options
dialog box to indicate the region over which the optimization is to be
performed.  You may set the value of one or more factors to a constant
by setting the low and high limits to that value.
```

Taula 43. Resultat de l'anàlisi d'optimització



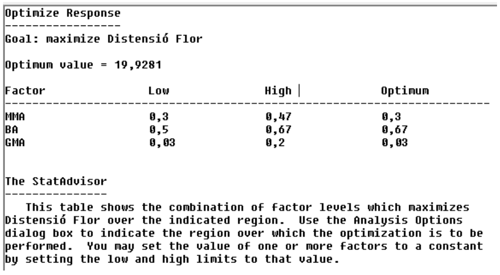
Gràfic 10. Diagrama de superfície de resposta

- ## DISTENSIÓ FLOR



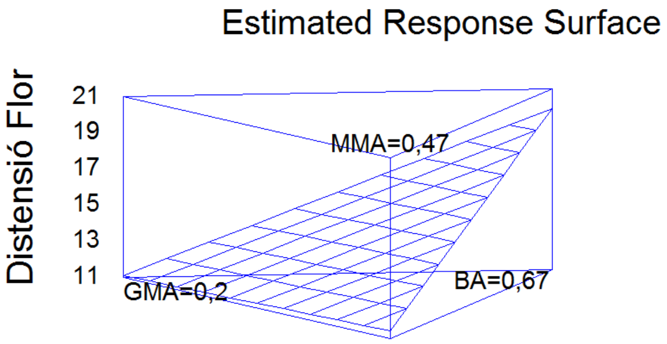
```
ANOVA for Distensió Flor
--------------------------------------------------------------------------------
Source                   Sum of Squares     Df    Mean Square    F-Ratio     P-Value
--------------------------------------------------------------------------------
Linear Model                    51,4159      2        25,7079       3,61      0,0708
Total error                     64,1733      9        7,13036
--------------------------------------------------------------------------------
Total (corr.)                   115,589     11

R-squared = 44,4816 percent
R-squared (adjusted for d.f.) = 32,1442 percent
Standard Error of Est. = 2,67027
Mean absolute error = 2,0156
Durbin-Watson statistic = 1,87942
Lag 1 residual autocorrelation = -0,0378713



The StatAdvisor
---------------
   This table shows an analysis of variance for the currently selected
linear model.  Since the P-value for this model is less than 0.10,
there is a statistically significant relationship between Distensió
Flor and the components at the 90% confidence level.

   The R-Squared statistic indicates that the model as fitted explains
44,4816% of the variability in Distensió Flor.  The adjusted R-squared
statistic, which is more suitable for comparing models with different
numbers of independent variables, is 32,1442%.  The standard error of
the estimate shows the standard deviation of the residuals to be
2,67027.  The mean absolute error (MAE) of 2,0156 is the average value
of the residuals.  The Durbin-Watson (DW) statistic tests the
residuals to determine if there is any significant correlation based
on the order in which they occur in your data file.  Since the P-value
is less than 0.05, there is an indication of possible serial
correlation.  Plot the residuals versus row order to see if there is
any pattern which can be seen.
```

Taula 44. Resultat de l'anàlisi de variància



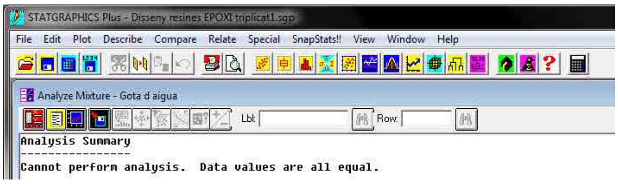
```
Optimize Response
-----------------
Goal: maximize Distensió Flor

Optimum value = 19,9281

Factor               Low                High                Optimum
-----------------------------------------------------------------------
MMA                  0,3                0,47                0,3
BA                   0,5                0,67                0,67
GMA                  0,03               0,2                 0,03


The StatAdvisor
---------------
   This table shows the combination of factor levels which maximizes
Distensió Flor over the indicated region.  Use the Analysis Options
dialog box to indicate the region over which the optimization is to be
performed.  You may set the value of one or more factors to a constant
by setting the low and high limits to that value.
```

Taula 45. Resultat de l'anàlisi d'optimització



Gràfic 11. Diagrama de superfície de resposta

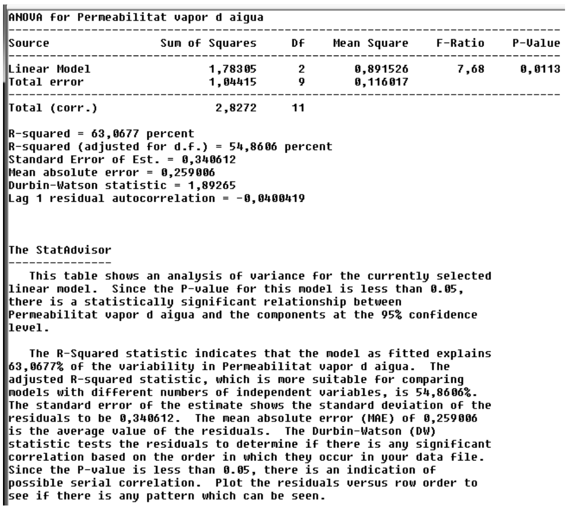
GOTA D'AIGUA



```
Analysis Summary
----------------
Cannot perform analysis.  Data values are all equal.
```

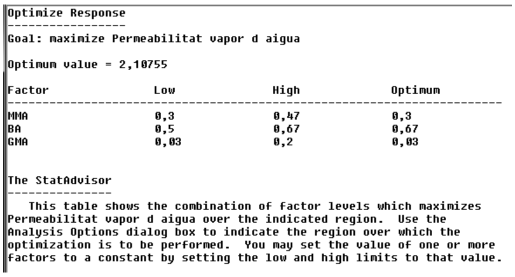
Figura 34. No hi ha anàlisi, ja que tots els resultats tenen el mateix valor

PERMEABILITAT AL VAPOR D'AIGUA



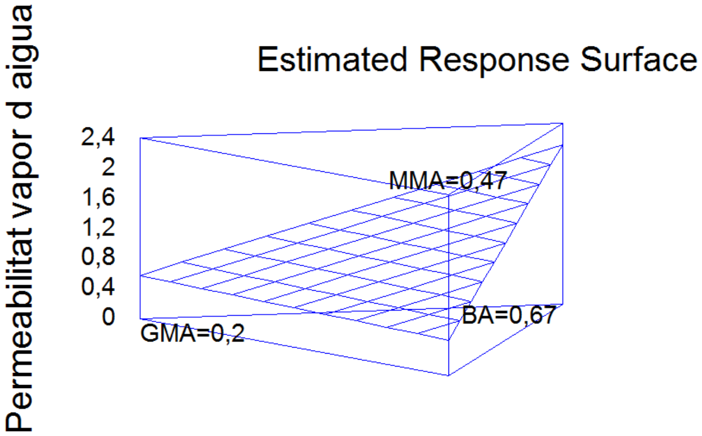
```
ANOVA for Permeabilitat vapor d aigua
-----------------------------------------------------------------------------
Source                   Sum of Squares     Df    Mean Square    F-Ratio      P-Value
-----------------------------------------------------------------------------
Linear Model                    1,78305      2       0,891526       7,68       0,0113
Total error                     1,04415      9       0,116017
-----------------------------------------------------------------------------
Total (corr.)                    2,8272     11

R-squared = 63,0677 percent
R-squared (adjusted for d.f.) = 54,8606 percent
Standard Error of Est. = 0,340612
Mean absolute error = 0,259006
Durbin-Watson statistic = 1,89265
Lag 1 residual autocorrelation = -0,0400419



The StatAdvisor
---------------
   This table shows an analysis of variance for the currently selected
linear model.  Since the P-value for this model is less than 0.05,
there is a statistically significant relationship between
Permeabilitat vapor d aigua and the components at the 95% confidence
level.

   The R-Squared statistic indicates that the model as fitted explains
63,0677% of the variability in Permeabilitat vapor d aigua.  The
adjusted R-squared statistic, which is more suitable for comparing
models with different numbers of independent variables, is 54,8606%.
The standard error of the estimate shows the standard deviation of the
residuals to be 0,340612.  The mean absolute error (MAE) of 0,259006
is the average value of the residuals.  The Durbin-Watson (DW)
statistic tests the residuals to determine if there is any significant
correlation based on the order in which they occur in your data file.
Since the P-value is less than 0.05, there is an indication of
possible serial correlation.  Plot the residuals versus row order to
see if there is any pattern which can be seen.
```

Taula 46. Resultat de l'anàlisi de variància



```
Optimize Response
-----------------
Goal: maximize Permeabilitat vapor d aigua

Optimum value = 2,10755

Factor                Low                High               Optimum
-----------------------------------------------------------------------
MMA                   0,3                0,47               0,3
BA                    0,5                0,67               0,67
GMA                   0,03               0,2                0,03


The StatAdvisor
---------------
   This table shows the combination of factor levels which maximizes
Permeabilitat vapor d aigua over the indicated region.  Use the
Analysis Options dialog box to indicate the region over which the
optimization is to be performed.  You may set the value of one or more
factors to a constant by setting the low and high limits to that value.
```

Taula 47. Resultat de l’anàlisi d’optimització



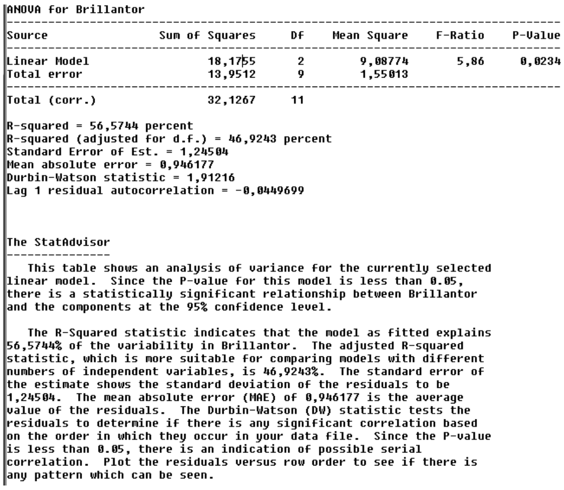
Gràfic 12. Diagrama de superfície de resposta

1.8.3 Anàlisis dels resultats de les resines funcionalitzades amb grups amínics

		R015A	R016A	R017A	R018A
Brillantor (%)		9.6 ± 0.1	5.6 ± 0.2	6.5 ± 0	5.7 ± 0.1
Flexió sec (nº)		75000	100000	85000	60000
Flexió humit (nº)		40000	50000	30000	35000
Fregament Sec (nota)	**500**	4.5	5.0	4.5	4.5
Fregament Humit (nota)	**50**	4.0	4.0	4.5	4.5
Distensió Flor (mm)		11.7 ± 0.2	12.1 ± 0.2	10.4 ± 0.1	14.4 ± 0.3
Gota d'aigua (temps)		>30min ± 0	20min ± 1	>30min ± 0	>30min ± 0
Permeabilitat vapor aigua (%)		0.07 ± 0.01	0.09 ± 0.01	0.05 ± 0.00	0.05 ± 0.01
Tacking		De + a - : R0018, R0017, R0015 = R0016			
Tacte pell		De + suau a -: 18, 17=15, 16			

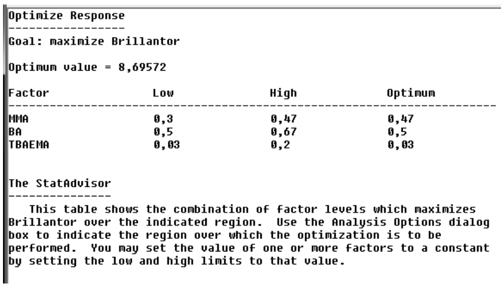
Taula 48. Resum de resultats de les resines funcionalitzades amb grups amínics

BRILLANTOR



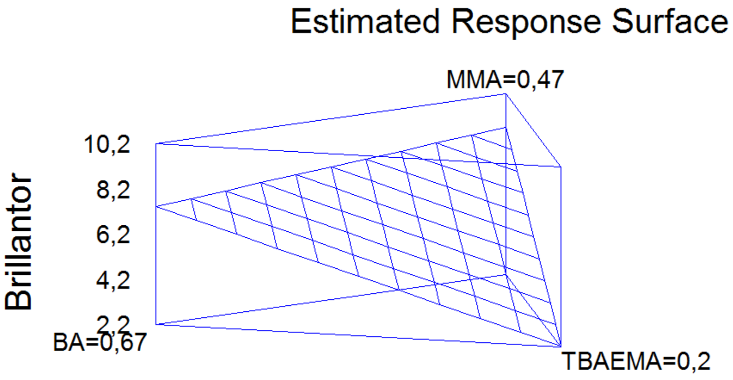
```
ANOVA for Brillantor
--------------------------------------------------------------------------------
Source                   Sum of Squares     Df     Mean Square     F-Ratio     P-Value
--------------------------------------------------------------------------------
Linear Model                    18,1755      2         9,08774        5,86      0,0234
Total error                     13,9512      9         1,55013
--------------------------------------------------------------------------------
Total (corr.)                   32,1267     11

R-squared = 56,5744 percent
R-squared (adjusted for d.f.) = 46,9243 percent
Standard Error of Est. = 1,24504
Mean absolute error = 0,946177
Durbin-Watson statistic = 1,91216
Lag 1 residual autocorrelation = -0,0449699


The StatAdvisor
---------------
   This table shows an analysis of variance for the currently selected
linear model.  Since the P-value for this model is less than 0.05,
there is a statistically significant relationship between Brillantor
and the components at the 95% confidence level.

   The R-Squared statistic indicates that the model as fitted explains
56,5744% of the variability in Brillantor.  The adjusted R-squared
statistic, which is more suitable for comparing models with different
numbers of independent variables, is 46,9243%.  The standard error of
the estimate shows the standard deviation of the residuals to be
1,24504.  The mean absolute error (MAE) of 0,946177 is the average
value of the residuals.  The Durbin-Watson (DW) statistic tests the
residuals to determine if there is any significant correlation based
on the order in which they occur in your data file.  Since the P-value
is less than 0.05, there is an indication of possible serial
correlation.  Plot the residuals versus row order to see if there is
any pattern which can be seen.
```

Taula 49. Resultat de l'anàlisi de variància



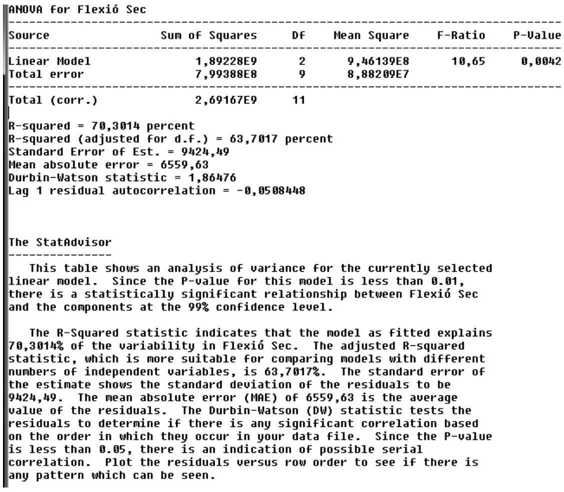
```
Optimize Response
-----------------
Goal: maximize Brillantor

Optimum value = 8,69572

Factor               Low               High               Optimum
-----------------------------------------------------------------------
MMA                  0,3               0,47               0,47
BA                   0,5               0,67               0,5
TBAEMA               0,03              0,2                0,03


The StatAdvisor
---------------
   This table shows the combination of factor levels which maximizes
Brillantor over the indicated region.  Use the Analysis Options dialog
box to indicate the region over which the optimization is to be
performed.  You may set the value of one or more factors to a constant
by setting the low and high limits to that value.
```

Taula 50. Resultat de l'anàlisi d'optimització



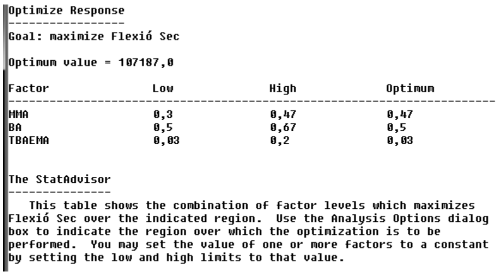
Gràfic 13. Diagrama de superfície de resposta

- **FLEXIÓ SEC**



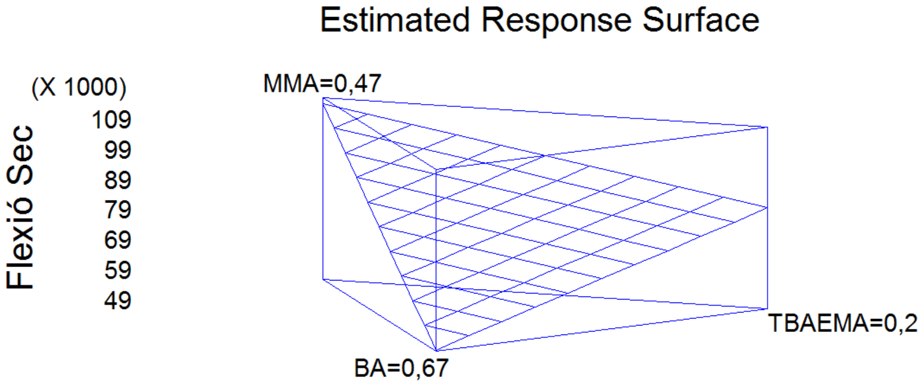
```
ANOVA for Flexió Sec
--------------------------------------------------------------------------------
Source                   Sum of Squares     Df     Mean Square    F-Ratio     P-Value
--------------------------------------------------------------------------------
Linear Model                  1,89228E9      2       9,46139E8      10,65      0,0042
Total error                   7,99388E8      9       8,88209E7
--------------------------------------------------------------------------------
Total (corr.)                 2,69167E9     11

R-squared = 70,3014 percent
R-squared (adjusted for d.f.) = 63,7017 percent
Standard Error of Est. = 9424,49
Mean absolute error = 6559,63
Durbin-Watson statistic = 1,86476
Lag 1 residual autocorrelation = -0,0508448



The StatAdvisor
---------------
   This table shows an analysis of variance for the currently selected
linear model.  Since the P-value for this model is less than 0.01,
there is a statistically significant relationship between Flexió Sec
and the components at the 99% confidence level.

   The R-Squared statistic indicates that the model as fitted explains
70,3014% of the variability in Flexió Sec.  The adjusted R-squared
statistic, which is more suitable for comparing models with different
numbers of independent variables, is 63,7017%.  The standard error of
the estimate shows the standard deviation of the residuals to be
9424,49.  The mean absolute error (MAE) of 6559,63 is the average
value of the residuals.  The Durbin-Watson (DW) statistic tests the
residuals to determine if there is any significant correlation based
on the order in which they occur in your data file.  Since the P-value
is less than 0.05, there is an indication of possible serial
correlation.  Plot the residuals versus row order to see if there is
any pattern which can be seen.
```

Taula 51. Resultat de l’anàlisi de variància



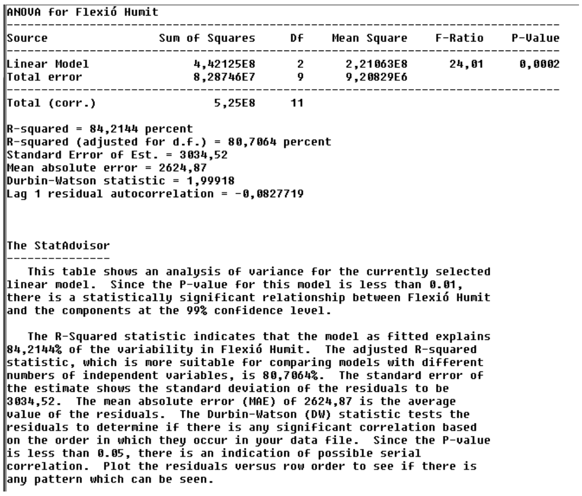
```
Optimize Response
-----------------
Goal: maximize Flexió Sec

Optimum value = 107187,0

Factor              Low               High              Optimum
-----------------------------------------------------------------------------
MMA                 0,3               0,47              0,47
BA                  0,5               0,67              0,5
TBAEMA              0,03              0,2               0,03


The StatAdvisor
---------------
   This table shows the combination of factor levels which maximizes
Flexió Sec over the indicated region.  Use the Analysis Options dialog
box to indicate the region over which the optimization is to be
performed.  You may set the value of one or more factors to a constant
by setting the low and high limits to that value.
```

Taula 52. Resultat de l’anàlisi d’optimització



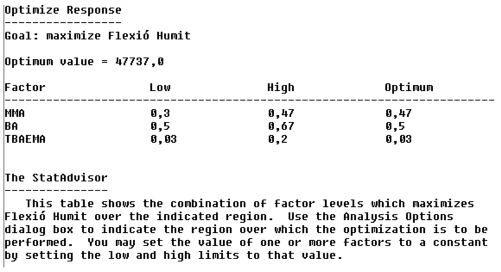
Gràfic 14. Diagrama de superfície de resposta

- **FLEXIÓ HUMIT**



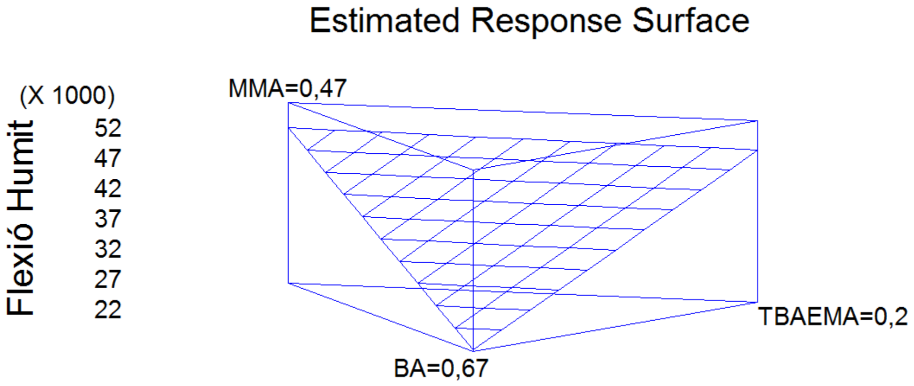
```
ANOVA for Flexió Humit
--------------------------------------------------------------------------------
Source                   Sum of Squares     Df     Mean Square    F-Ratio    P-Value
--------------------------------------------------------------------------------
Linear Model                  4,42125E8      2       2,21063E8      24,01     0,0002
Total error                   8,28746E7      9       9,20829E6
--------------------------------------------------------------------------------
Total (corr.)                    5,25E8     11

R-squared = 84,2144 percent
R-squared (adjusted for d.f.) = 80,7064 percent
Standard Error of Est. = 3034,52
Mean absolute error = 2624,87
Durbin-Watson statistic = 1,99918
Lag 1 residual autocorrelation = -0,0827719



The StatAdvisor
---------------
   This table shows an analysis of variance for the currently selected
linear model.  Since the P-value for this model is less than 0.01,
there is a statistically significant relationship between Flexió Humit
and the components at the 99% confidence level.

   The R-Squared statistic indicates that the model as fitted explains
84,2144% of the variability in Flexió Humit.  The adjusted R-squared
statistic, which is more suitable for comparing models with different
numbers of independent variables, is 80,7064%.  The standard error of
the estimate shows the standard deviation of the residuals to be
3034,52.  The mean absolute error (MAE) of 2624,87 is the average
value of the residuals.  The Durbin-Watson (DW) statistic tests the
residuals to determine if there is any significant correlation based
on the order in which they occur in your data file.  Since the P-value
is less than 0.05, there is an indication of possible serial
correlation.  Plot the residuals versus row order to see if there is
any pattern which can be seen.
```

Taula 53. Resultat de l'anàlisi de variància



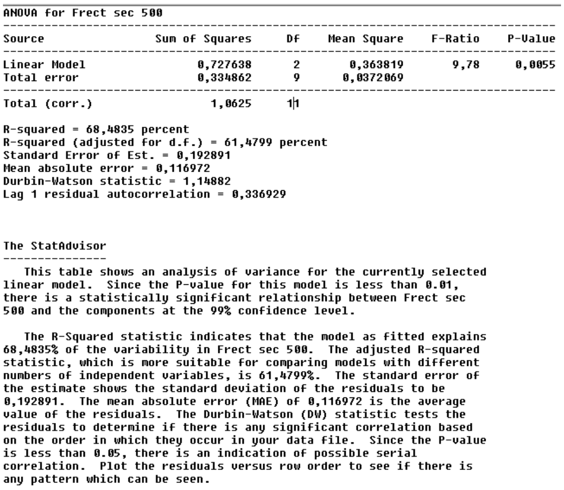
```
Optimize Response
-----------------
Goal: maximize Flexió Humit

Optimum value = 47737,0

Factor                Low                High               Optimum
-----------------------------------------------------------------------
MMA                   0,3                0,47               0,47
BA                    0,5                0,67               0,5
TBAEMA                0,03               0,2                0,03


The StatAdvisor
---------------
   This table shows the combination of factor levels which maximizes
Flexió Humit over the indicated region.  Use the Analysis Options
dialog box to indicate the region over which the optimization is to be
performed.  You may set the value of one or more factors to a constant
by setting the low and high limits to that value.
```

Taula 54. Resultat de l'anàlisi d'optimització



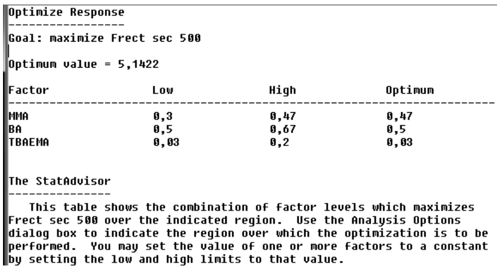
Gràfic 15. Diagrama de superfície de resposta

- **FREC SEC 500**



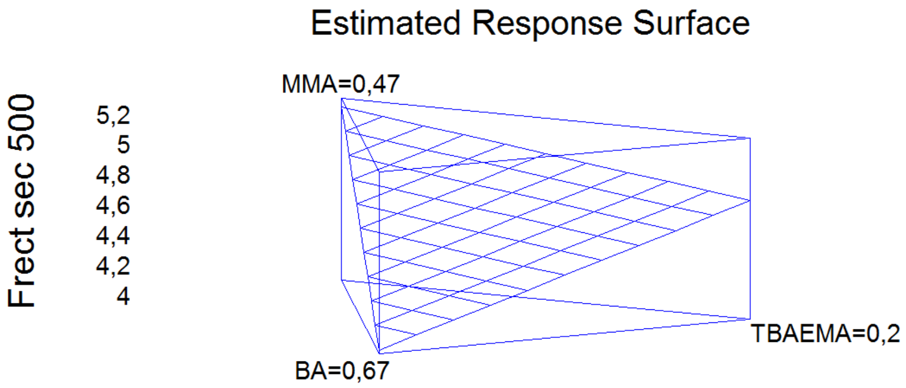
```
ANOVA for Frect sec 500
--------------------------------------------------------------------------------
Source                  Sum of Squares     Df    Mean Square    F-Ratio    P-Value
--------------------------------------------------------------------------------
Linear Model                  0,727638      2       0,363819       9,78     0,0055
Total error                   0,334862      9      0,0372069
--------------------------------------------------------------------------------
Total (corr.)                   1,0625     11

R-squared = 68,4835 percent
R-squared (adjusted for d.f.) = 61,4799 percent
Standard Error of Est. = 0,192891
Mean absolute error = 0,116972
Durbin-Watson statistic = 1,14882
Lag 1 residual autocorrelation = 0,336929


The StatAdvisor
---------------
   This table shows an analysis of variance for the currently selected
linear model.  Since the P-value for this model is less than 0.01,
there is a statistically significant relationship between Frect sec
500 and the components at the 99% confidence level.

   The R-Squared statistic indicates that the model as fitted explains
68,4835% of the variability in Frect sec 500.  The adjusted R-squared
statistic, which is more suitable for comparing models with different
numbers of independent variables, is 61,4799%.  The standard error of
the estimate shows the standard deviation of the residuals to be
0,192891.  The mean absolute error (MAE) of 0,116972 is the average
value of the residuals.  The Durbin-Watson (DW) statistic tests the
residuals to determine if there is any significant correlation based
on the order in which they occur in your data file.  Since the P-value
is less than 0.05, there is an indication of possible serial
correlation.  Plot the residuals versus row order to see if there is
any pattern which can be seen.
```

Taula 55. Resultat de l'anàlisi de variància



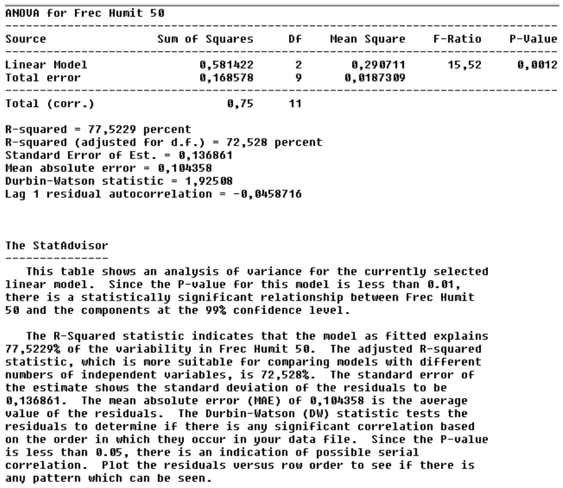
```
Optimize Response
-----------------
Goal: maximize Frect sec 500

Optimum value = 5,1422

Factor               Low                High               Optimum
-----------------------------------------------------------------------
MMA                  0,3                0,47               0,47
BA                   0,5                0,67               0,5
TBAEMA               0,03               0,2                0,03


The StatAdvisor
---------------
   This table shows the combination of factor levels which maximizes
Frect sec 500 over the indicated region.  Use the Analysis Options
dialog box to indicate the region over which the optimization is to be
performed.  You may set the value of one or more factors to a constant
by setting the low and high limits to that value.
```

Taula 56. Resultat de l'anàlisi d'optimització



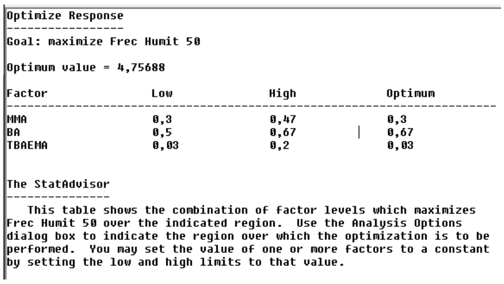
Gràfic 16. Diagrama de superfície de resposta

- ## FREC HUMIT 50



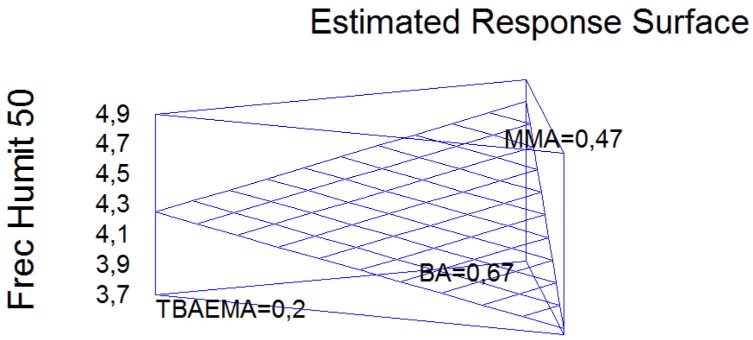
```
ANOVA for Frec Humit 50
--------------------------------------------------------------------------------
Source                Sum of Squares     Df    Mean Square    F-Ratio     P-Value
--------------------------------------------------------------------------------
Linear Model                0,581422      2       0,290711      15,52      0,0012
Total error                 0,168578      9      0,0187309
--------------------------------------------------------------------------------
Total (corr.)                   0,75     11

R-squared = 77,5229 percent
R-squared (adjusted for d.f.) = 72,528 percent
Standard Error of Est. = 0,136861
Mean absolute error = 0,104358
Durbin-Watson statistic = 1,92508
Lag 1 residual autocorrelation = -0,0458716



The StatAdvisor
---------------
   This table shows an analysis of variance for the currently selected
linear model.  Since the P-value for this model is less than 0.01,
there is a statistically significant relationship between Frec Humit
50 and the components at the 99% confidence level.

   The R-Squared statistic indicates that the model as fitted explains
77,5229% of the variability in Frec Humit 50.  The adjusted R-squared
statistic, which is more suitable for comparing models with different
numbers of independent variables, is 72,528%.  The standard error of
the estimate shows the standard deviation of the residuals to be
0,136861.  The mean absolute error (MAE) of 0,104358 is the average
value of the residuals.  The Durbin-Watson (DW) statistic tests the
residuals to determine if there is any significant correlation based
on the order in which they occur in your data file.  Since the P-value
is less than 0.05, there is an indication of possible serial
correlation.  Plot the residuals versus row order to see if there is
any pattern which can be seen.
```

Taula 57. Resultat de l'anàlisi de variància



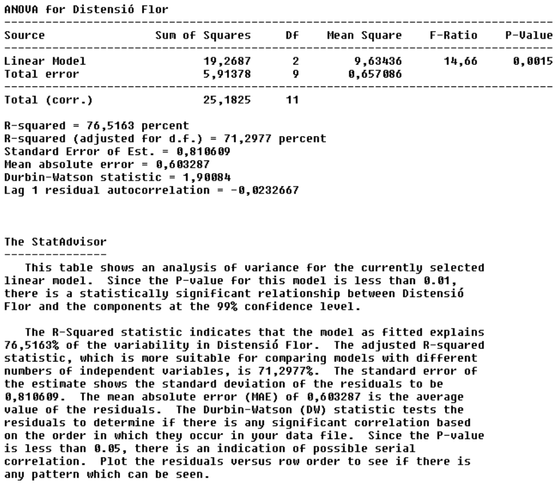
```
Optimize Response
-----------------
Goal: maximize Frec Humit 50

Optimum value = 4,75688

Factor               Low              High             Optimum
-----------------------------------------------------------------------
MMA                  0,3              0,47             0,3
BA                   0,5              0,67         |   0,67
TBAEMA               0,03             0,2              0,03


The StatAdvisor
---------------
   This table shows the combination of factor levels which maximizes
Frec Humit 50 over the indicated region.  Use the Analysis Options
dialog box to indicate the region over which the optimization is to be
performed.  You may set the value of one or more factors to a constant
by setting the low and high limits to that value.
```

Taula 58. Resultat de l'anàlisi d'optimització



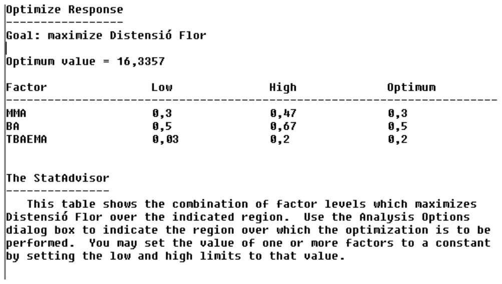
Gràfic 17. Diagrama de superfície de resposta

DISTENSIÓ FLOR



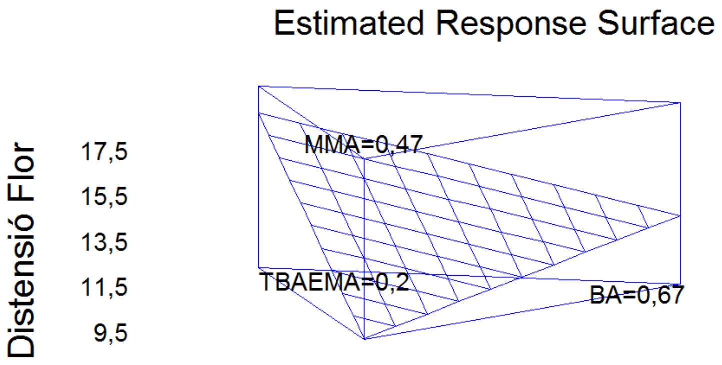
```
ANOVA for Distensió Flor
--------------------------------------------------------------------------------
Source                  Sum of Squares     Df    Mean Square    F-Ratio    P-Value
--------------------------------------------------------------------------------
Linear Model                   19,2687      2        9,63436      14,66     0,0015
Total error                    5,91378      9       0,657086
--------------------------------------------------------------------------------
Total (corr.)                  25,1825     11

R-squared = 76,5163 percent
R-squared (adjusted for d.f.) = 71,2977 percent
Standard Error of Est. = 0,810609
Mean absolute error = 0,603287
Durbin-Watson statistic = 1,90084
Lag 1 residual autocorrelation = -0,0232667


The StatAdvisor
---------------
   This table shows an analysis of variance for the currently selected
linear model.  Since the P-value for this model is less than 0.01,
there is a statistically significant relationship between Distensió
Flor and the components at the 99% confidence level.

   The R-Squared statistic indicates that the model as fitted explains
76,5163% of the variability in Distensió Flor.  The adjusted R-squared
statistic, which is more suitable for comparing models with different
numbers of independent variables, is 71,2977%.  The standard error of
the estimate shows the standard deviation of the residuals to be
0,810609.  The mean absolute error (MAE) of 0,603287 is the average
value of the residuals.  The Durbin-Watson (DW) statistic tests the
residuals to determine if there is any significant correlation based
on the order in which they occur in your data file.  Since the P-value
is less than 0.05, there is an indication of possible serial
correlation.  Plot the residuals versus row order to see if there is
any pattern which can be seen.
```

Taula 59. Resultat de l'anàlisi de variància



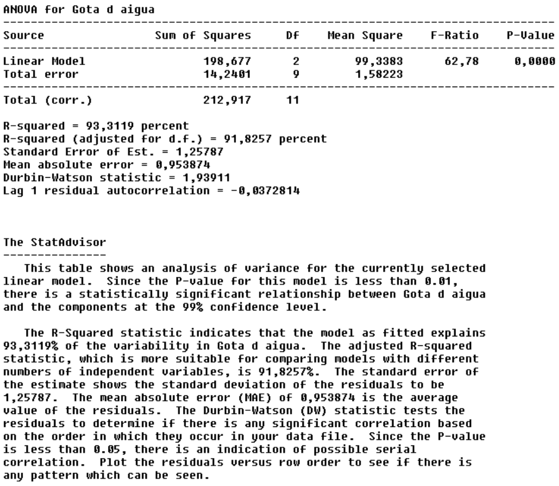
```
Optimize Response
-----------------
Goal: maximize Distensió Flor

Optimum value = 16,3357

Factor                Low              High             Optimum
-----------------------------------------------------------------------
MMA                   0,3              0,47             0,3
BA                    0,5              0,67             0,5
TBAEMA                0,03             0,2              0,2


The StatAdvisor
---------------
   This table shows the combination of factor levels which maximizes
Distensió Flor over the indicated region.  Use the Analysis Options
dialog box to indicate the region over which the optimization is to be
performed.  You may set the value of one or more factors to a constant
by setting the low and high limits to that value.
```

Taula 60. Resultat de l'anàlisi d'optimització



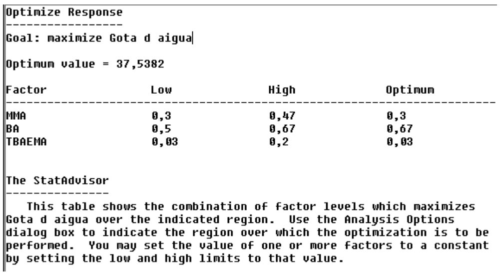
Gràfic 18. Diagrama de superfície de resposta

- **GOTA D'AIGUA**



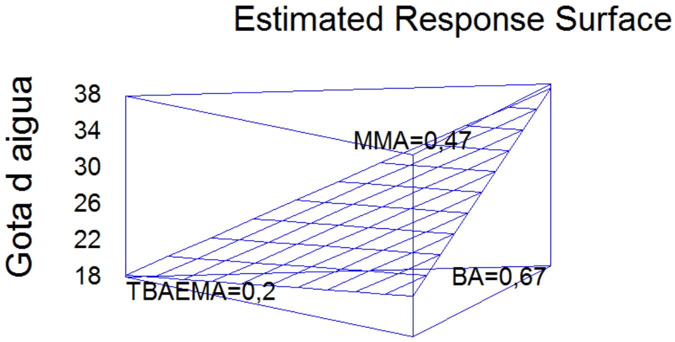
```
ANOVA for Gota d aigua
--------------------------------------------------------------------------------
Source                   Sum of Squares     Df    Mean Square    F-Ratio    P-Value
--------------------------------------------------------------------------------
Linear Model                    198,677      2        99,3383      62,78     0,0000
Total error                     14,2401      9        1,58223
--------------------------------------------------------------------------------
Total (corr.)                   212,917     11

R-squared = 93,3119 percent
R-squared (adjusted for d.f.) = 91,8257 percent
Standard Error of Est. = 1,25787
Mean absolute error = 0,953874
Durbin-Watson statistic = 1,93911
Lag 1 residual autocorrelation = -0,0372814


The StatAdvisor
---------------
   This table shows an analysis of variance for the currently selected
linear model.  Since the P-value for this model is less than 0.01,
there is a statistically significant relationship between Gota d aigua
and the components at the 99% confidence level.

   The R-Squared statistic indicates that the model as fitted explains
93,3119% of the variability in Gota d aigua.  The adjusted R-squared
statistic, which is more suitable for comparing models with different
numbers of independent variables, is 91,8257%.  The standard error of
the estimate shows the standard deviation of the residuals to be
1,25787.  The mean absolute error (MAE) of 0,953874 is the average
value of the residuals.  The Durbin-Watson (DW) statistic tests the
residuals to determine if there is any significant correlation based
on the order in which they occur in your data file.  Since the P-value
is less than 0.05, there is an indication of possible serial
correlation.  Plot the residuals versus row order to see if there is
any pattern which can be seen.
```

Taula 61. Resultat de l'anàlisi de variància



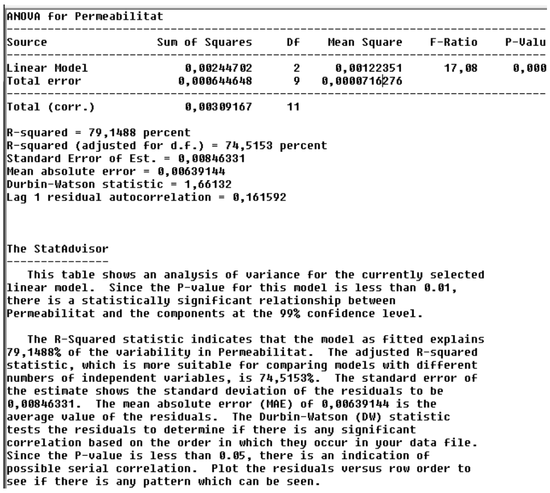
```
Optimize Response
-----------------
Goal: maximize Gota d aigua

Optimum value = 37,5382

Factor               Low                High               Optimum
-----------------------------------------------------------------------
MMA                  0,3                0,47               0,3
BA                   0,5                0,67               0,67
TBAEMA               0,03               0,2                0,03


The StatAdvisor
---------------
   This table shows the combination of factor levels which maximizes
Gota d aigua over the indicated region.  Use the Analysis Options
dialog box to indicate the region over which the optimization is to be
performed.  You may set the value of one or more factors to a constant
by setting the low and high limits to that value.
```

Taula 62. Resultat de l'anàlisi d'optimització



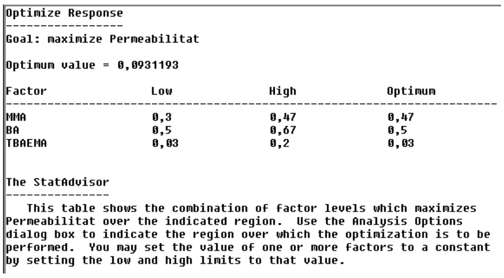
Gràfic 19. Diagrama de superfície de resposta

PERMEABILITAT AL VAPOR D'AIGUA



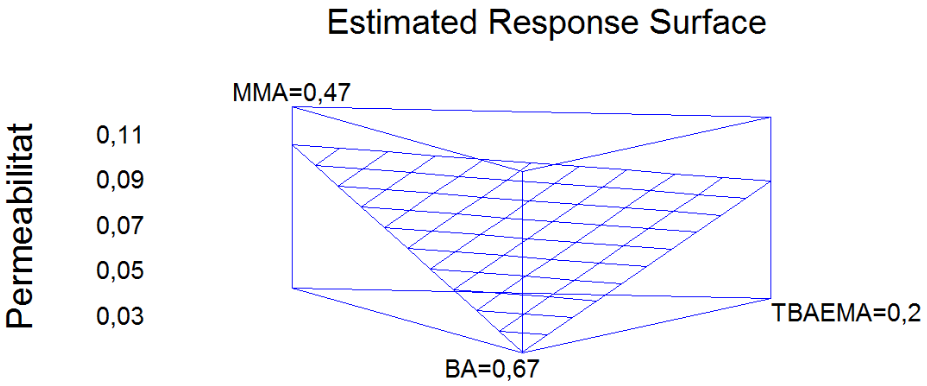
```
ANOVA for Permeabilitat
--------------------------------------------------------------------------------
Source                 Sum of Squares     Df     Mean Square     F-Ratio     P-Valu
--------------------------------------------------------------------------------
Linear Model               0,00244702      2      0,00122351       17,08      0,000
Total error               0,000644648      9    0,0000716276
--------------------------------------------------------------------------------
Total (corr.)              0,00309167     11

R-squared = 79,1488 percent
R-squared (adjusted for d.f.) = 74,5153 percent
Standard Error of Est. = 0,00846331
Mean absolute error = 0,00639144
Durbin-Watson statistic = 1,66132
Lag 1 residual autocorrelation = 0,161592



The StatAdvisor
---------------
   This table shows an analysis of variance for the currently selected
linear model.  Since the P-value for this model is less than 0.01,
there is a statistically significant relationship between
Permeabilitat and the components at the 99% confidence level.

   The R-Squared statistic indicates that the model as fitted explains
79,1488% of the variability in Permeabilitat.  The adjusted R-squared
statistic, which is more suitable for comparing models with different
numbers of independent variables, is 74,5153%.  The standard error of
the estimate shows the standard deviation of the residuals to be
0,00846331.  The mean absolute error (MAE) of 0,00639144 is the
average value of the residuals.  The Durbin-Watson (DW) statistic
tests the residuals to determine if there is any significant
correlation based on the order in which they occur in your data file.
Since the P-value is less than 0.05, there is an indication of
possible serial correlation.  Plot the residuals versus row order to
see if there is any pattern which can be seen.
```

Taula 63. Resultat de l'anàlisi de variància



```
Optimize Response
-----------------
Goal: maximize Permeabilitat

Optimum value = 0,0931193

Factor               Low                High               Optimum
-----------------------------------------------------------------------
MMA                  0,3                0,47               0,47
BA                   0,5                0,67               0,5
TBAEMA               0,03               0,2                0,03


The StatAdvisor
---------------
   This table shows the combination of factor levels which maximizes
Permeabilitat over the indicated region.  Use the Analysis Options
dialog box to indicate the region over which the optimization is to be
performed.  You may set the value of one or more factors to a constant
by setting the low and high limits to that value.
```

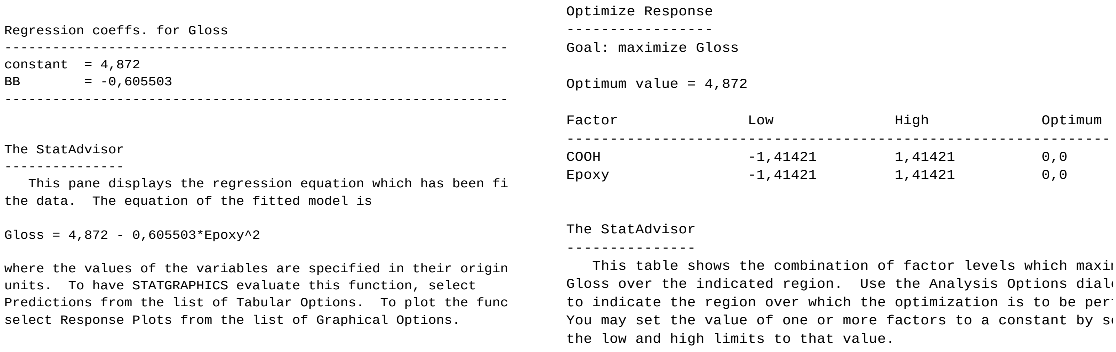
Taula 64. Resultat de l'anàlisi d'optimització



Gràfic 20. Diagrama de superfície de resposta

1.8.4 Anàlisis dels resultats de la barreja de resines epoxi-carboxíliques

- **BRILLANTOR**

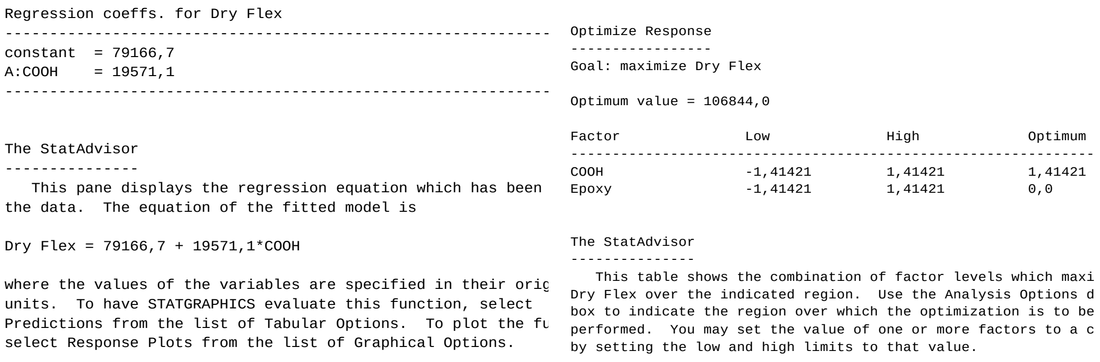


```
Regression coeffs. for Gloss
---------------------------------------------------------------
constant  = 4,872
BB        = -0,605503
---------------------------------------------------------------


The StatAdvisor
---------------
   This pane displays the regression equation which has been fi
the data.  The equation of the fitted model is

Gloss = 4,872 - 0,605503*Epoxy^2

where the values of the variables are specified in their origin
units.  To have STATGRAPHICS evaluate this function, select
Predictions from the list of Tabular Options.  To plot the func
select Response Plots from the list of Graphical Options.
```

```
Optimize Response
-----------------
Goal: maximize Gloss

Optimum value = 4,872

Factor                Low              High             Optimum
-----------------------------------------------------------------
COOH                  -1,41421         1,41421          0,0
Epoxy                 -1,41421         1,41421          0,0


The StatAdvisor
---------------
   This table shows the combination of factor levels which maxi
Gloss over the indicated region.  Use the Analysis Options dial
to indicate the region over which the optimization is to be per
You may set the value of one or more factors to a constant by s
the low and high limits to that value.
```

Taula 65. Resultat de l'anàlisi de variància i de l'anàlisi d'optimització

- **FLEXIÓ EN SEC**

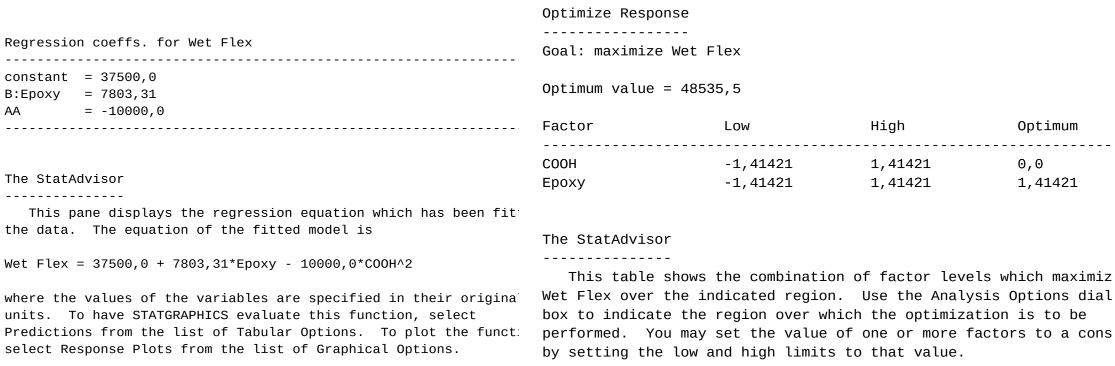


```
Regression coeffs. for Dry Flex
---------------------------------------------------------------
constant   = 79166,7
A:COOH     = 19571,1
---------------------------------------------------------------


The StatAdvisor
---------------
   This pane displays the regression equation which has been
the data.  The equation of the fitted model is

Dry Flex = 79166,7 + 19571,1*COOH

where the values of the variables are specified in their orig
units.  To have STATGRAPHICS evaluate this function, select
Predictions from the list of Tabular Options.  To plot the fu
select Response Plots from the list of Graphical Options.
```

```
Optimize Response
-----------------
Goal: maximize Dry Flex

Optimum value = 106844,0

Factor               Low              High             Optimum
---------------------------------------------------------------
COOH                 -1,41421         1,41421          1,41421
Epoxy                -1,41421         1,41421          0,0


The StatAdvisor
---------------
   This table shows the combination of factor levels which maxi
Dry Flex over the indicated region.  Use the Analysis Options d
box to indicate the region over which the optimization is to be
performed.  You may set the value of one or more factors to a c
by setting the low and high limits to that value.
```

Taula 66. Resultat de l'anàlisi de variància i de l'anàlisi d'optimització

- **FLEXIÓ EN HUMIT**

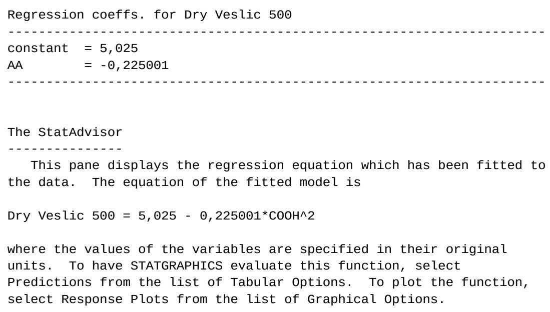


```
Regression coeffs. for Wet Flex
---------------------------------------------------------------
constant  = 37500,0
B:Epoxy   = 7803,31
AA        = -10000,0
---------------------------------------------------------------


The StatAdvisor
---------------
   This pane displays the regression equation which has been fit
the data.  The equation of the fitted model is

Wet Flex = 37500,0 + 7803,31*Epoxy - 10000,0*COOH^2

where the values of the variables are specified in their origina
units.  To have STATGRAPHICS evaluate this function, select
Predictions from the list of Tabular Options.  To plot the funct
select Response Plots from the list of Graphical Options.
```

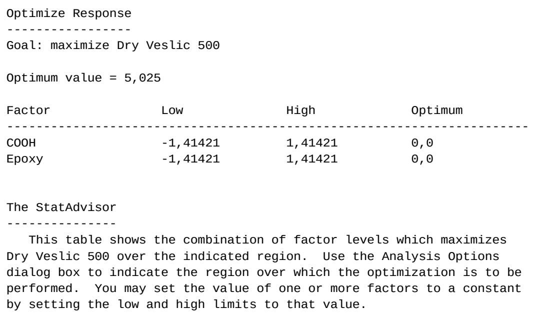
```
Optimize Response
-----------------
Goal: maximize Wet Flex

Optimum value = 48535,5

Factor                Low              High             Optimum
-----------------------------------------------------------------
COOH                  -1,41421         1,41421          0,0
Epoxy                 -1,41421         1,41421          1,41421


The StatAdvisor
---------------
   This table shows the combination of factor levels which maximiz
Wet Flex over the indicated region.  Use the Analysis Options dial
box to indicate the region over which the optimization is to be
performed.  You may set the value of one or more factors to a cons
by setting the low and high limits to that value.
```

Taula 67. Resultat de l'anàlisi de variància i de l'anàlisi d'optimització

FREC EN SEC 500



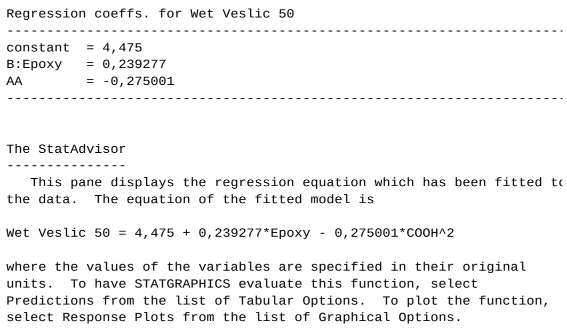
```
Regression coeffs. for Dry Veslic 500
----------------------------------------------------------------------
constant  = 5,025
AA        = -0,225001
----------------------------------------------------------------------


The StatAdvisor
---------------
   This pane displays the regression equation which has been fitted to
the data.  The equation of the fitted model is

Dry Veslic 500 = 5,025 - 0,225001*COOH^2

where the values of the variables are specified in their original
units.  To have STATGRAPHICS evaluate this function, select
Predictions from the list of Tabular Options.  To plot the function,
select Response Plots from the list of Graphical Options.
```



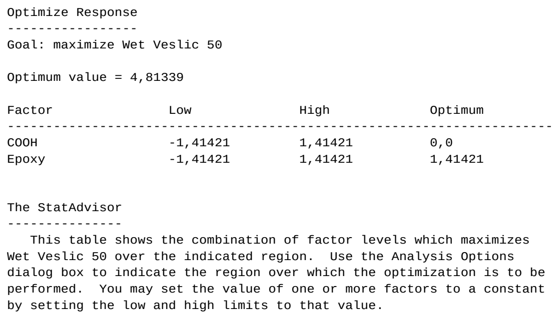
```
Optimize Response
-----------------
Goal: maximize Dry Veslic 500

Optimum value = 5,025

Factor               Low              High             Optimum
-----------------------------------------------------------------------
COOH                 -1,41421         1,41421          0,0
Epoxy                -1,41421         1,41421          0,0


The StatAdvisor
---------------
   This table shows the combination of factor levels which maximizes
Dry Veslic 500 over the indicated region.  Use the Analysis Options
dialog box to indicate the region over which the optimization is to be
performed.  You may set the value of one or more factors to a constant
by setting the low and high limits to that value.
```

Taula 68. Resultat de l'anàlisi de variància i de l'anàlisi d'optimització

FREC EN HUMIT 50



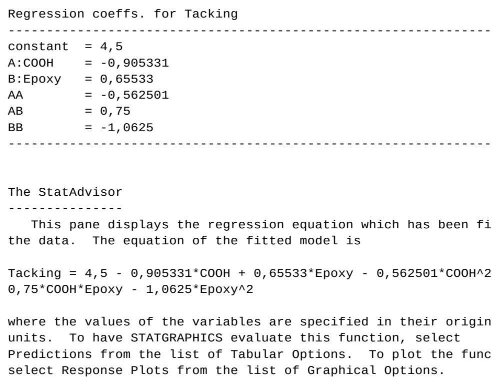
```
Regression coeffs. for Wet Veslic 50
-----------------------------------------------------------------------
constant  = 4,475
B:Epoxy   = 0,239277
AA        = -0,275001
-----------------------------------------------------------------------


The StatAdvisor
---------------
   This pane displays the regression equation which has been fitted tc
the data.  The equation of the fitted model is

Wet Veslic 50 = 4,475 + 0,239277*Epoxy - 0,275001*COOH^2

where the values of the variables are specified in their original
units.  To have STATGRAPHICS evaluate this function, select
Predictions from the list of Tabular Options.  To plot the function,
select Response Plots from the list of Graphical Options.
```



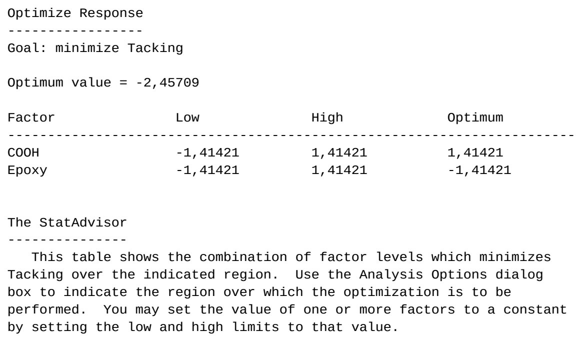
```
Optimize Response
-----------------
Goal: maximize Wet Veslic 50

Optimum value = 4,81339

Factor               Low              High             Optimum
-----------------------------------------------------------------------
COOH                 -1,41421         1,41421          0,0
Epoxy                -1,41421         1,41421          1,41421


The StatAdvisor
---------------
   This table shows the combination of factor levels which maximizes
Wet Veslic 50 over the indicated region.  Use the Analysis Options
dialog box to indicate the region over which the optimization is to be
performed.  You may set the value of one or more factors to a constant
by setting the low and high limits to that value.
```

Taula 69. Resultat de l'anàlisi de variància i de l'anàlisi d'optimització

TACKING



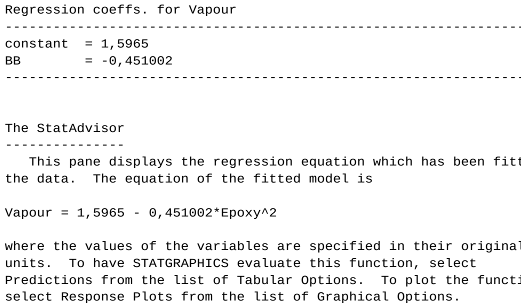
```
Regression coeffs. for Tacking
----------------------------------------------------------------
constant  = 4,5
A:COOH    = -0,905331
B:Epoxy   = 0,65533
AA        = -0,562501
AB        = 0,75
BB        = -1,0625
----------------------------------------------------------------


The StatAdvisor
---------------
   This pane displays the regression equation which has been fi
the data.  The equation of the fitted model is

Tacking = 4,5 - 0,905331*COOH + 0,65533*Epoxy - 0,562501*COOH^2
0,75*COOH*Epoxy - 1,0625*Epoxy^2

where the values of the variables are specified in their origin
units.  To have STATGRAPHICS evaluate this function, select
Predictions from the list of Tabular Options.  To plot the func
select Response Plots from the list of Graphical Options.
```



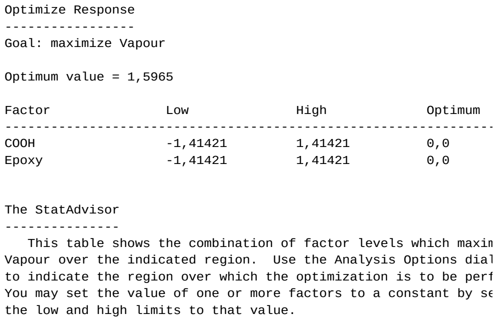
```
Optimize Response
-----------------
Goal: minimize Tacking

Optimum value = -2,45709

Factor               Low              High             Optimum
-----------------------------------------------------------------------
COOH                 -1,41421         1,41421          1,41421
Epoxy                -1,41421         1,41421          -1,41421


The StatAdvisor
---------------
   This table shows the combination of factor levels which minimizes
Tacking over the indicated region.  Use the Analysis Options dialog
box to indicate the region over which the optimization is to be
performed.  You may set the value of one or more factors to a constant
by setting the low and high limits to that value.
```

Taula 70. Resultat de l'anàlisi de variància i de l'anàlisi d'optimització

PERMEABILITAT AL VAPOR D'AIGUA



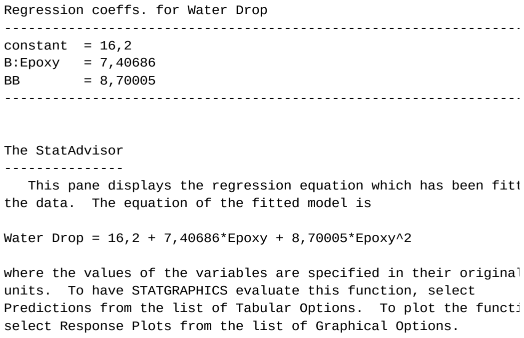
```
Regression coeffs. for Vapour
-----------------------------------------------------------------
constant  = 1,5965
BB        = -0,451002
-----------------------------------------------------------------


The StatAdvisor
---------------
   This pane displays the regression equation which has been fitt
the data.  The equation of the fitted model is

Vapour = 1,5965 - 0,451002*Epoxy^2

where the values of the variables are specified in their original
units.  To have STATGRAPHICS evaluate this function, select
Predictions from the list of Tabular Options.  To plot the functi
select Response Plots from the list of Graphical Options.
```



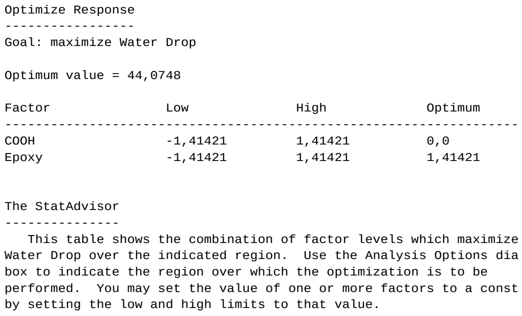
```
Optimize Response
-----------------
Goal: maximize Vapour

Optimum value = 1,5965

Factor               Low              High             Optimum
-----------------------------------------------------------------
COOH                 -1,41421         1,41421          0,0
Epoxy                -1,41421         1,41421          0,0


The StatAdvisor
---------------
   This table shows the combination of factor levels which maxim
Vapour over the indicated region.  Use the Analysis Options dial
to indicate the region over which the optimization is to be perf
You may set the value of one or more factors to a constant by se
the low and high limits to that value.
```

Taula 71. Resultat de l'anàlisi de variància i de l'anàlisi d'optimització

RESISTÈNCIA A LA GOTA D'AIGUA



```
Regression coeffs. for Water Drop
-----------------------------------------------------------------
constant  = 16,2
B:Epoxy   = 7,40686
BB        = 8,70005
-----------------------------------------------------------------


The StatAdvisor
---------------
   This pane displays the regression equation which has been fitt
the data.  The equation of the fitted model is

Water Drop = 16,2 + 7,40686*Epoxy + 8,70005*Epoxy^2

where the values of the variables are specified in their original
units.  To have STATGRAPHICS evaluate this function, select
Predictions from the list of Tabular Options.  To plot the functi
select Response Plots from the list of Graphical Options.
```

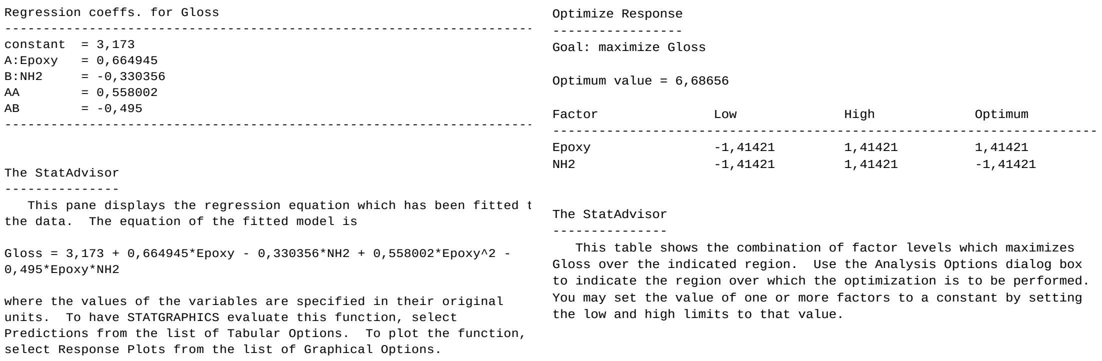


```
Optimize Response
-----------------
Goal: maximize Water Drop

Optimum value = 44,0748

Factor               Low              High             Optimum
-------------------------------------------------------------------
COOH                 -1,41421         1,41421          0,0
Epoxy                -1,41421         1,41421          1,41421


The StatAdvisor
---------------
   This table shows the combination of factor levels which maximize
Water Drop over the indicated region.  Use the Analysis Options dia
box to indicate the region over which the optimization is to be
performed.  You may set the value of one or more factors to a const
by setting the low and high limits to that value.
```

Taula 72. Resultat de l'anàlisi de variància i de l'anàlisi d'optimització

1.8.5 Anàlisis dels resultats de la barreja de resines epoxi-amíniques

- **BRILLANTOR**

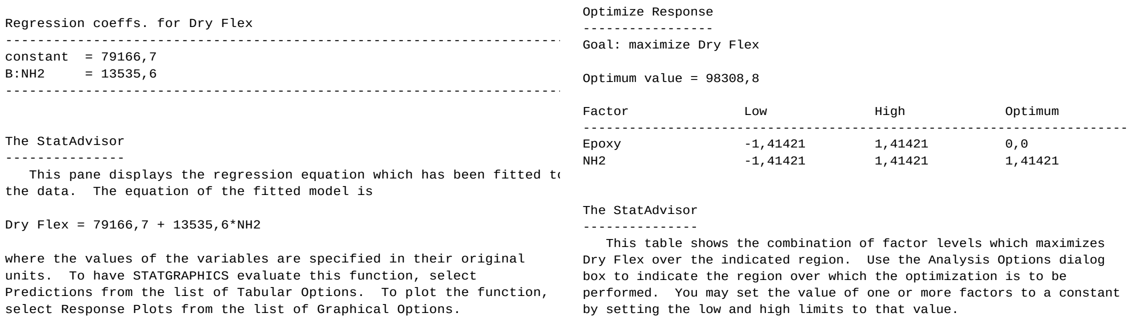


```
Regression coeffs. for Gloss
----------------------------------------------------------------------
constant  = 3,173
A:Epoxy   = 0,664945
B:NH2     = -0,330356
AA        = 0,558002
AB        = -0,495
----------------------------------------------------------------------


The StatAdvisor
---------------
   This pane displays the regression equation which has been fitted t
the data.  The equation of the fitted model is

Gloss = 3,173 + 0,664945*Epoxy - 0,330356*NH2 + 0,558002*Epoxy^2 -
0,495*Epoxy*NH2

where the values of the variables are specified in their original
units.  To have STATGRAPHICS evaluate this function, select
Predictions from the list of Tabular Options.  To plot the function,
select Response Plots from the list of Graphical Options.
```

```
Optimize Response
-----------------
Goal: maximize Gloss

Optimum value = 6,68656

Factor               Low              High             Optimum
-----------------------------------------------------------------------
Epoxy                -1,41421         1,41421          1,41421
NH2                  -1,41421         1,41421          -1,41421


The StatAdvisor
---------------
   This table shows the combination of factor levels which maximizes
Gloss over the indicated region.  Use the Analysis Options dialog box
to indicate the region over which the optimization is to be performed.
You may set the value of one or more factors to a constant by setting
the low and high limits to that value.
```

Taula 73. Resultat de l'anàlisi de variància i de l'anàlisi d'optimització

- **FLEXIÓ EN SEC**

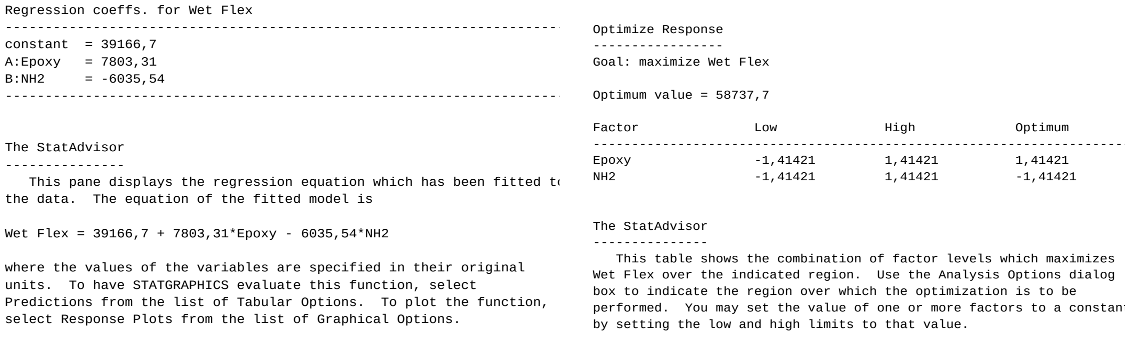


```
Regression coeffs. for Dry Flex
----------------------------------------------------------------------
constant  = 79166,7
B:NH2     = 13535,6
----------------------------------------------------------------------


The StatAdvisor
---------------
   This pane displays the regression equation which has been fitted t
the data.  The equation of the fitted model is

Dry Flex = 79166,7 + 13535,6*NH2

where the values of the variables are specified in their original
units.  To have STATGRAPHICS evaluate this function, select
Predictions from the list of Tabular Options.  To plot the function,
select Response Plots from the list of Graphical Options.
```

```
Optimize Response
-----------------
Goal: maximize Dry Flex

Optimum value = 98308,8

Factor               Low              High             Optimum
-----------------------------------------------------------------------
Epoxy                -1,41421         1,41421          0,0
NH2                  -1,41421         1,41421          1,41421


The StatAdvisor
---------------
   This table shows the combination of factor levels which maximizes
Dry Flex over the indicated region.  Use the Analysis Options dialog
box to indicate the region over which the optimization is to be
performed.  You may set the value of one or more factors to a constant
by setting the low and high limits to that value.
```

Taula 74. Resultat de l'anàlisi de variància i de l'anàlisi d'optimització

- **FLEXIÓ EN HUMIT**

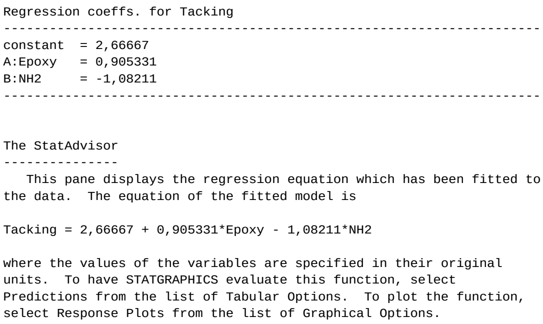


```
Regression coeffs. for Wet Flex
----------------------------------------------------------------------
constant  = 39166,7
A:Epoxy   = 7803,31
B:NH2     = -6035,54
----------------------------------------------------------------------


The StatAdvisor
---------------
   This pane displays the regression equation which has been fitted t
the data.  The equation of the fitted model is

Wet Flex = 39166,7 + 7803,31*Epoxy - 6035,54*NH2

where the values of the variables are specified in their original
units.  To have STATGRAPHICS evaluate this function, select
Predictions from the list of Tabular Options.  To plot the function,
select Response Plots from the list of Graphical Options.
```

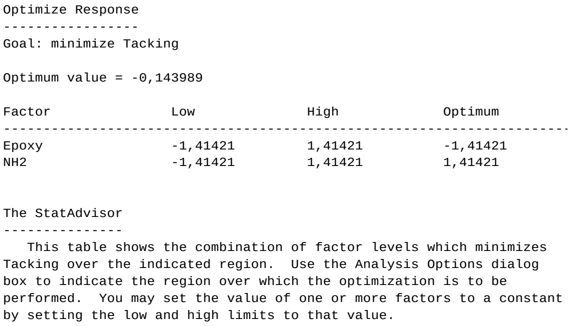
```
Optimize Response
-----------------
Goal: maximize Wet Flex

Optimum value = 58737,7

Factor               Low              High             Optimum
-----------------------------------------------------------------------
Epoxy                -1,41421         1,41421          1,41421
NH2                  -1,41421         1,41421          -1,41421


The StatAdvisor
---------------
   This table shows the combination of factor levels which maximizes
Wet Flex over the indicated region.  Use the Analysis Options dialog
box to indicate the region over which the optimization is to be
performed.  You may set the value of one or more factors to a constan
by setting the low and high limits to that value.
```

Taula 75. Resultat de l'anàlisi de variància i de l'anàlisi d'optimització

- **TACKING**



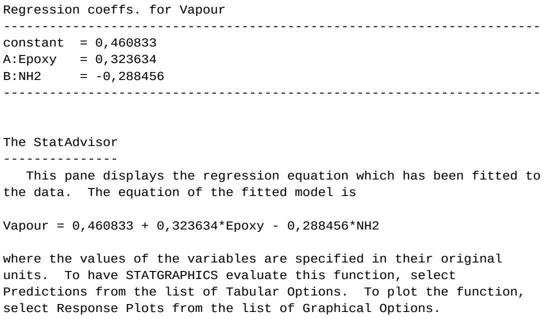
```
Regression coeffs. for Tacking
----------------------------------------------------------------------
constant  = 2,66667
A:Epoxy   = 0,905331
B:NH2     = -1,08211
----------------------------------------------------------------------


The StatAdvisor
---------------
   This pane displays the regression equation which has been fitted to
the data.  The equation of the fitted model is

Tacking = 2,66667 + 0,905331*Epoxy - 1,08211*NH2

where the values of the variables are specified in their original
units.  To have STATGRAPHICS evaluate this function, select
Predictions from the list of Tabular Options.  To plot the function,
select Response Plots from the list of Graphical Options.
```



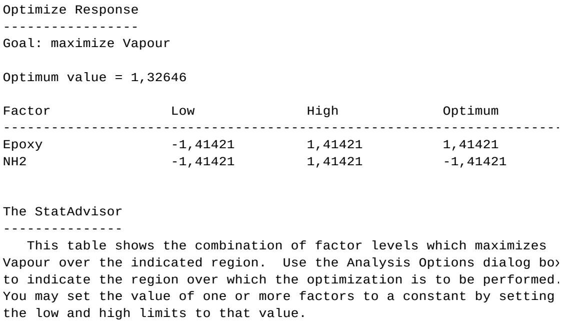
```
Optimize Response
-----------------
Goal: minimize Tacking

Optimum value = -0,143989

Factor               Low              High             Optimum
-----------------------------------------------------------------------
Epoxy                -1,41421         1,41421          -1,41421
NH2                  -1,41421         1,41421          1,41421


The StatAdvisor
---------------
   This table shows the combination of factor levels which minimizes
Tacking over the indicated region.  Use the Analysis Options dialog
box to indicate the region over which the optimization is to be
performed.  You may set the value of one or more factors to a constant
by setting the low and high limits to that value.
```

Taula 76. Resultat de l'anàlisi de variància i de l'anàlisi d'optimització

- **PERMEABILITAT AL VAPOR D'AIGUA**



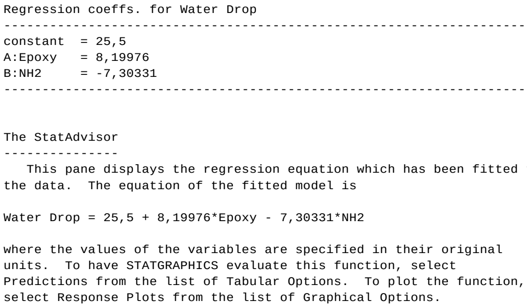
```
Regression coeffs. for Vapour
----------------------------------------------------------------------
constant  = 0,460833
A:Epoxy   = 0,323634
B:NH2     = -0,288456
----------------------------------------------------------------------


The StatAdvisor
---------------
   This pane displays the regression equation which has been fitted to
the data.  The equation of the fitted model is

Vapour = 0,460833 + 0,323634*Epoxy - 0,288456*NH2

where the values of the variables are specified in their original
units.  To have STATGRAPHICS evaluate this function, select
Predictions from the list of Tabular Options.  To plot the function,
select Response Plots from the list of Graphical Options.
```



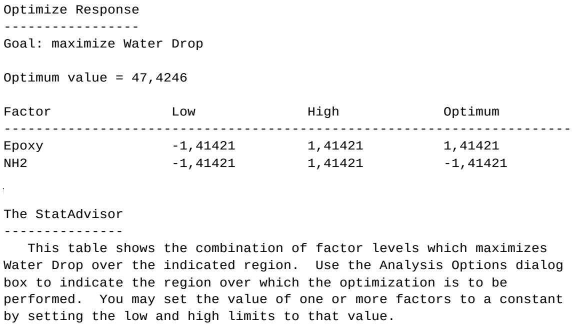
```
Optimize Response
-----------------
Goal: maximize Vapour

Optimum value = 1,32646

Factor               Low              High             Optimum
-----------------------------------------------------------------------
Epoxy                -1,41421         1,41421          1,41421
NH2                  -1,41421         1,41421          -1,41421


The StatAdvisor
---------------
   This table shows the combination of factor levels which maximizes
Vapour over the indicated region.  Use the Analysis Options dialog bo
to indicate the region over which the optimization is to be performed.
You may set the value of one or more factors to a constant by setting
the low and high limits to that value.
```

Taula 77. Resultat de l'anàlisi de variància i de l'anàlisi d'optimització

- **RESISTÈNCIA A LA GOTA D'AIGUA**



```
Regression coeffs. for Water Drop
----------------------------------------------------------------------
constant  = 25,5
A:Epoxy   = 8,19976
B:NH2     = -7,30331
----------------------------------------------------------------------


The StatAdvisor
---------------
   This pane displays the regression equation which has been fitted
the data.  The equation of the fitted model is

Water Drop = 25,5 + 8,19976*Epoxy - 7,30331*NH2

where the values of the variables are specified in their original
units.  To have STATGRAPHICS evaluate this function, select
Predictions from the list of Tabular Options.  To plot the function,
select Response Plots from the list of Graphical Options.
```



```
Optimize Response
-----------------
Goal: maximize Water Drop

Optimum value = 47,4246

Factor               Low              High             Optimum
-----------------------------------------------------------------------
Epoxy                -1,41421         1,41421          1,41421
NH2                  -1,41421         1,41421          -1,41421


The StatAdvisor
---------------
   This table shows the combination of factor levels which maximizes
Water Drop over the indicated region.  Use the Analysis Options dialog
box to indicate the region over which the optimization is to be
performed.  You may set the value of one or more factors to a constant
by setting the low and high limits to that value.
```

Taula 78. Resultat de l'anàlisi de variància i de l'anàlisi d'optimització

1.9 ANNEX 9. Càlculs i preparació de les barreges de resines

Una vegada sintetitzades les resines seleccionades, caracteritzades amb els diferents grups funcionals estudiats en aquest treball (carboxílics, epoxi, amínics), es continua amb el disseny de les barreges de resines, amb l'objectiu d'aconseguir un sistema bicomponent que proporcioni bones prestacions, sense la necessitat o utilització de reticulants externs. Així doncs, es treballa en direcció a una "clean production", ja que els reticulants són de caràcter nociu durant la seva manipulació.

Aquest projecte està centrat, principalment, en l'estudi de les resines epoxi, i és per aquest motiu, que aquestes resines seran el denominador comú en les barreges dutes a terme. D'aquesta manera s'analitzaran, d'una banda, els resultats del sistema bicomponent dels grups epoxi i carboxílics, i de l'altra, els resultats del sistema bicomponent dels grups epoxi i amínics.

1.9.1 Ajust Estequiomètric de les barreges

Per a poder dur a terme les barreges, cal tenir en compte que en els sistemes bicomponent, les reaccions es produeixen equivalent a equivalent. Per aquest motiu, en primer lloc, es calculen els equivalents que hi ha en 100g de resina. Una vegada obtingut aquest valor, ja podrem arribar a calcular la relació dels grams necessaris de cada una de les dues resines, per a que es produeixi la reacció entre elles.

Amb aquesta relació ja coneguda, es podrà introduir l'experiment a l'Statgraphics per tal d'obtenir un disseny amb un nombre de barreges suficientment representatiu com per extreure'n conclusions fiables.

- *Càlcul del Pes Equivalent i dels Equivalents de monòmer*:

$$Pes\ equivalent = \frac{Massa\ molecular}{n^{\underline{o}}\ carregues\ positives} \qquad Equivalent = \frac{g\ Solut}{Pes\ equivalent}$$

	Monòmer	**Nº Càrregues$^+$ dels monòmers**	**% de Monòmer**	**% sòlids de la resina**	**PES EQUIVALENT (g)**	**EQUIVALENTS / 100g Resina**
Resina CARBOXÍLICA	MAA	1 grup **carboxil** = 1 Càrrega $^+$	6.75%	**35%**	$\frac{86.09}{1\text{càrrega}^+}=86.09g$	$\frac{6.75gMAA}{86.09g}=0.078eq\text{Ácid}$
Resina EPOXI	GMA	1 grup **epoxi** = 1 Càrrega $^+$	1.01%		$\frac{142.15}{1\text{càrrega}^+}=142.15g$	$\frac{1.01gMAA}{142.15g}=0.0071eqEpoxi$
Resina AMÍNICA	TBAEMA	1 grup **amínic** = 1 Càrrega $^+$	1.01%		$\frac{185.26}{1\text{càrrega}^+}=185.26g$	$\frac{1.01gMAA}{185.26g}=0.0055eqAmina$

Taula 79. Resum dels càlculs dels equivalents de cada grup funcional, en cada 100g de resina

* Per cada 100g de resina carboxílica a 35% de sòlids, tenim 0.079 equivalents d'àcid.

* Per cada 100g de resina epoxídica a 35% de sòlids, tenim 0.0071 equivalents d'epòxid.

* Per cada 100g de resina amínica a 35% de sòlids, tenim 0.0055 equivalents d'amina.

	Equivalents / 100g Resina	**Resultat**
Autoreticulació Epoxi - Carboxílica	0.0071 Eq Epoxi	**100g de resina epoxi 35% sòlids, reaccionaran amb 9g de resina carboxílica 35% sòlids.**
	0.078 Eq Àcid	
Autoreticulació Epoxi – Amínica	0.0071 Eq Epoxi	**100g de resina amínica 35% sòlids, reaccionaran amb 75g de resina epoxi 35% sòlids.**
	0.0055 Eq Amina	

Taula 80. Quantitat necessària de cada resina (reacció equivalent a equivalent)

Càlcul dels nivells utilitzant l'Statgraphics:

Ja definida la quantitat necessària de resina, per a que es produeixi la reacció de reticulació entre les parelles de resines caracteritzades amb els grups funcionals objecte d'aquest estudi, cal fer ús de l'eina d'anàlisi estadístic (Statgrapics) per tal d'establir quines i quantes barreges de resines cal fer, amb l'objectiu d'arribar a obtenir uns resultats representatius de les reaccions.

El tipus d'anàlisi que s'ha establert és el de Superfície de Resposta que permet, de manera visual, inspeccionar la resposta en certa zona dels nivells dels factors d'interès, i també avaluar la seva sensibilitat als factors de tractament. Aquest tipus d'anàlisi estadístic es pot utilitzar en estudis de processos fonamentals, com per exemple podria ser la interacció entre nitrogen i fòsfor en el creixement de les plantes. El disseny de superfícies de resposta i l'estratègia d'anàlisi, suposa que la mitja de la variable de resposta està en funció dels nivells de factors quantitatius representats per les variables. Els models polinomials s'utilitzen com a aproximacions pràctiques a la funció de resposta verdadera o real. Els models polinomials que s'acostumen a utilitzar en l'anàlisi de superfícies de resposta, són el model lineal, i el model quadràtic. Per entendre millor tot aquests conceptes, anirem explicant els diferents passos que s'ha seguit per a dur a terme l'anàlisi mitjançant el sistema escollit: superfície de resposta.

Com s'ha comentat, l'experiment consta de factors i variables que cal tenir clares abans de començar. En aquest les variables i factors són:

- Procés a optimitzar: barreja entre resines amb grups funcionals diferents, per arribar a l'"autoreticulació" sense reticulants externs, i obtenint bons resultats una vegada aplicada aquesta barreja en forma de top sobre l'acabat de la pell.
- FACTORS: 2 factors en cada cas (Epoxi i Carboxílic; Epoxi i Amínic).
- VARIABLES: fent referència als assajos de laboratori realitzats, una vegada aplicat el top de resina sobre la pell (el top format per la barreja de cada una de les parelles de resines).
- Tipus de disseny: centralitzat, ortogonal i rotacional.

Ja introduït el disseny, obtenim els diferents nivells: ***-1.414 / -1 / 0 / 1 / 1.414***. Anomenem nivells als valors que corresponen a la quantitat de resina necessària en les diferents barreges. Aquest nivells són valors codificats, amb els que treballa el programa estadístic, i per tant, cal descodificar-los per quantificar amb un valor real els grams de resina necessaris en les diferents barreges.

El nivell 0 correspondrà als valors de quantitat de resina calculats anteriorment (9:100 en el cas de Carboxílica – Epoxi ; i 100:75 en el cas de Amínica – Epoxi). Pel que fa als altres valors, a continuació, s'adjunten els càlculs:

→ **Carboxílica - Epoxi**:

- Relació del punt central 0 = **9:100**.
- Punt més baix o mínim (-1.414) = **0**.
- Punt més alt o màxim (1.414) = el **doble** del punt central, en aquest cas **18:200**.

	-1.414	-1	0	1	1.414
R-COOH	**0**		***9***		**18**
R-EPOXI	**0**		***100***		**200**

Taula 81. Taula de nivells amb els punts centrals, punts mínims, i punts màxims

→ **Amínica - Epoxi** :

- Relació del punt central 0 = **100:75**.
- Punt més baix o mínim (-1.414) = **0**.
- Punt més alt o màxim (1.414) = el **doble** del punt central, en aquest cas **200:150**.

	-1.414	-1	0	1	1.414
R-AMIN	**0**		***100***		**200**
R-EPOXI	**0**		***75***		**150**

Taula 82. Taula de nivells amb els punts centrals, punts mínims, i punts màxims

En relació als nivells que falten, el càlcul es fa de la manera següent:

	-1.414	-1	0	1	1.414
R-COOH	**0**		***9***		**18**
R-EPOXI	**0**	$\frac{100-0}{1.414}=x$ $100-x=29$	***100***	$\frac{200-100}{1.414}=x$ $100+x=171$	**200**

Taula 83. Exemple de càlcul dels nivells

Així doncs, els *nivells* complets de cada una de les barreges de resines, queden de la següent manera:

	-1.414	-1	0	1	1.414
R-COOH	0	3	***9***	15	18
R-EPOXI	0	29	***100***	171	200

	-1.414	-1	0	1	1.414
R-AMIN	0	29	***100***	171	200
R-EPOXI	0	22	***75***	128	150

Taula 84. Taules de nivells de cada una de les parelles de resines

Després d'introduir el disseny en el programa estadístic, i descodificar-ne els nivells obtinguts, observem les diferents barreges que se'ns proposen. Considerant fer la barreja amb un volum total de 75mL, descodifiquem els resultats obtenint els valors següents:

			BARREJA REAL	
Test	**Resina COOH**	**Resina EPOXI**	**Qtat R.COOH (mL)**	**Qtat R.EPOXI (mL)**
1	3	29	7	68
2	3	171	1	74
3	15	29	26	49
4	15	171	6	69
5	9	0	75	0
6	9	200	3	72
7	0	100	0	75
8	18	100	11	64
9	9	100	6	69
10	9	100	6	69
11	9	100	6	69
12	9	100	6	69

			BARREJA REAL	
Test	**Resina AMIN**	**Resina EPOXI**	**Qtat R.AMIN (mL)**	**Qtat R.EPOXI (mL)**
1	29	22	43	32
2	29	128	14	61
3	171	22	66	9
4	171	128	43	32
5	100	0	75	0
6	100	150	30	45
7	0	75	0	75
8	200	75	55	20
9	100	75	43	32
10	100	75	43	32
11	100	75	43	32
12	100	75	43	32

Taula 85. Proporció de resina necessària en cada barreja; valors codificats i descodificats

Aquestes taules corresponen a la proporció de resina necessària en cada barreja. Les dues darreres columnes de cada taula, inclouen els valors de la proporció necessària ja descodificats (mL).

1.10 ANNEX 10. Documentació consultada: articles, patents, i fitxes de seguretat

S'adjunta la primera pàgina de cada article i/o patent, seguint l'ordre de la bibliografia inscrita.

EMULSION POLYMERS IN LEATHER FINISHING*

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ABSTRACT

In the introductory sections, a brief description of emulsions and emulsion stability is presented. The details of preparation of an acrylic emulsion are contrasted to the manufacturing techniques employed in making lacquer emulsions. The development of high molecular weight urethane emulsion polymers borrows from each of the preceding polymers: the acrylics and the lacquer.

The theory of reactive emulsion polymers is discussed. The film formation process is briefly reviewed and a means for preparing a cross-linked system for highly reactive materials is described.

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The leather industry has a number of interesting features. One of the most intriguing is that many leather finishes are now and have been aqueous based products. During the past decade, a variety of new solvent systems have been employed for industrial use, and the leather industry has gone in a similar direction. Today, with pollution considerations and spiraling solvent costs, water finishes are being re-examined by the coatings manufacturers.

All the known types of emulsions are used in the preparation of leather finishes. In this paper, I should like to review the technology of emulsion polymer preparation.

Definitions

An emulsion can be defined as a system of two immiscible liquids, one of which is dispersed in the other. The most simple case is oil and water. When a mixture of the two is shaken vigorously, one is dispersed into the other in fine droplets. The continuous phase is generally the system present in the greater amount, and the dispersed phase in the smaller amount.

The droplets are spherical. Liquids possess a property called surface tension† which tends to reduce the free surface area to a minimum. Since a sphere has

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*Presented at the 14th Congress of the IULCTS at Barcelona, Spain, October 20–24, 1975. Reprinted from *Congress Papers*, Vol. II, 111–120 (1975).

†Surface tension is the mathematical equivalent of surface free energy.

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A. Q. E. I. C.
XXVIII ASAMBLEA NACIONAL
DE LA ASOCIACION QUIMICA ESPAÑOLA
DE LA INDUSTRIA DEL CUERO
SEVILLA - MAYO 1979

TOPS ACUOSOS REACTIVOS PARA ACABADOS DE CUERO

Por:
Luis Labastida
Manuel Salarich
De: Stahl Ibérica, S. A.

HIGH PERFORMANCE ACRYLIC RESINS*

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Introduction

The availability of acrylic resins for the manufacture of leather dates back well over fifty years. Some of the earliest acrylics developed for the base coating of leather are still in use today, a tribute to the performance capabilities achieved through acrylic technology. In the last decade, as new technology was developed, new products such as ultra-fine impregnation resins(1), ambient cure base coat resins(2,3), and water reducible topcoats(4,5) have appeared. In this paper, some of the more recently developed materials for the finishing of leather are described with emphasis on the performance of soft, but tough acrylic basecoat emulsions. They offer a superior balance of elasticity/tensile strength with freedom from tack normally associated with typical basecoat resins. Also covered are acrylic topcoat materials which are comparable to nitrocellulose lacquer emulsions in application performance and physical requirements.

PROPERTIES OF POLYMERS USED FOR FINISHING LEATHER

Some of the conventional resins which were developed years ago are still used today, and are customarily characterized for their strongest property or contribution to film properties. Properties such as thermoplasticity, adhesion, penetration, and hold out which influence the selection of specific resins. To achieve the balance of properties required by a finish, it is customary to blend resins. Table I shows film properties of three conventional resins along with their blends. Of significance is the low tensile strength of the softer resins as compared to the hard resin H, although elongations at yield are not much different. This leads to a low modulus at 100% elongation which is an indicator of lower toughness. When blended with hard resin, both tensile strength and modulus pick up to more satisfactory levels, while flexibility of the film still reflects the low T_{300} values.

The T_{300} value of a torsion modulus profile is an approximation of the softening point of a polymer in degrees centigrade. Taking the T_{300} value and relating it to the modulus at 30°C, characterizes the polymer's behavior from the low temperature range to ambient temperatures. The modulus curve provides valuable information by which to compare polymers and predict key performance properties.

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* Presented at the 79th Annual Meeting of the American Leather Chemists Association at Hershey, Pennsylvania, June 23, 1983.

(4)

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ACABADOS ACRILICOS *

por

J. M.ª BLASCO**; J. P. BUSANYA***; L. M.ª LABASTIDA**** y E. SOLANAS*****

RESUMEN

Se describe la historia cronológica en el descubrimiento y desarrollo de las emulsiones acrílicas desde 1910 a nuestros días, en el acabado del cuero.

Se hace una breve introducción a los métodos y conceptos de polimerización de los monómeros acrílicos, así como, de estos mismos y de las propiedades finales más sobresalientes que cabe esperar en los polímeros en función de los co-monómeros que intervienen. Los aspectos físico-químicos del polímero en emulsión son analizados en función del uso y método de aplicación, a la vez que, se discute la funcionalidad y lo que cabe esperar de ella. La correlación entre las propiedades de los polímeros acrílicos y posibilidades de uso, en función de los resultados a obtener y el proceso mecánico de aplicación.

Se relacionan los diferentes componentes de un acabado acrílico y cómo pueden éstos afectar las propiedades finales de éste.

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* Ponencia presentada en la Jornada Monográfica sobre Acabados de la XXXV Asamblea de AQEIC, Lorca Murcia, del 1 al 3 de mayo de 1986.

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**** Luis M.ª Labastida Acemar, nacido en Barcelona en 1947. Estudió Ingeniería Química en la Universidad Politécnica de Barcelona y estudios de Ciencias Químicas en la Universidad de Barcelona. Diplomado en Polímeros, desde 1970 pertenece a Stahl Ibérica desempeñando hasta 1981 la Dirección de Investigación de la División Polyvinyl de Stahl Ibérica, y desde esta fecha la Dirección de Investigación de Stahl Ibérica, en sus distintas Divisiones.

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CROSS-LINKED POLYMERS TO PROVIDE BETTER PROPERTIES IN LEATHER FINISHING

by

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Abstract

The physical properties of a polymer system are dependent upon the molecular weight of the polymer. In the coatings industry, reactive systems are employed to provide cross-linking to achieve the performance level being sought. In leather finishing, the times and temperatures are generally insufficient to obtain significant cross-linking without having pot life problems.

In this paper, the entire subject will be reviewed and existing approaches along with novel compositions will be discussed. Both solution systems and emulsion systems will be described.

Introduction

The requirements for a leather finish today are far more stringent than they were twenty years ago. It is necessary to pass a variety of tests involving flexibility, adhesion, light stability and so on. Thus, the R & D in the coatings industry today involves improving performance.

In many coating areas, reactive systems are employed. The films cast undergo a thermally catalyzed reaction and the final film is described as a cross-linked system. This cross-linked polymer is of extremely high molecular weight; in fact, it can be considered as a single molecule. The formation of this cross-linked entity permits obtaining properties that are far superior to those of low molecular weight polymers. As examples, properties, such as solvent resistance and detergent resistance, are difficult to achieve using linear polymers.

The subject of this paper is cross-linking. The material to be discussed is tabulated below:

1. Chemistry of Cross-Linking
2. Properties of Cross-Linked Polymers
3. Viscosity Considerations Relating to Application Properties
4. Pot Life or Stability
5. Film Formation
6. Some Compositions of Cross-Linking Systems in Current Use

Presented at the 82nd Annual Meeting of the American Leather Chemists Association June 25, 1986 at the Pinehurst Hotel, Pinehurst, North Carolina.

CONFERENCIAS

XLVI

ASAMBLEA NACIONAL DE LA ASOCIACIÓN QUÍMICA ESPAÑOLA DE LA INDUSTRIA DEL CUERO Y JORNADAS TECNICAS HISPANO-LUSAS

ESTORIL (Portugal)

1 al 4 de Mayo 1997

(7) [PAPER, 1999 pag. 683-684
ISSN-0032-3934]

Reaccions epoxi amb altres
grups químics!

KINETIC MODELING OF COMPETITIVE CROSSLINKING REACTIONS FOR CYCLOALIPHATIC EPOXIDES WITH HYDROXYL- AND CARBOXYL-FUNCTIONALIZED ACRYLIC COPOLYMERS (LOW pH AND TEMPERATURE EFFECTS)

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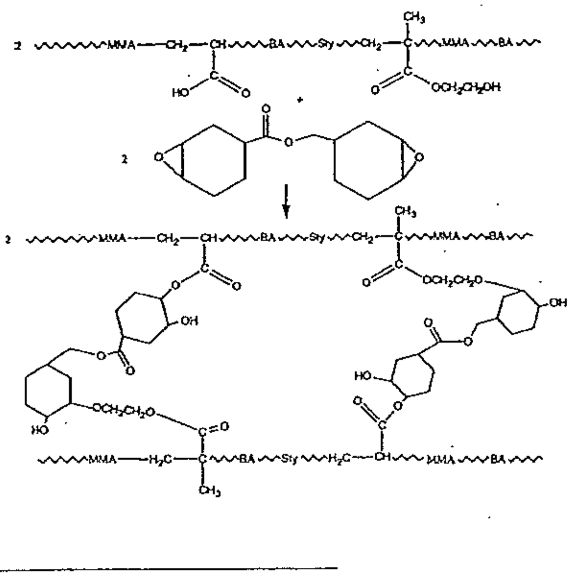
Introduction

Epoxy resins are high performance thermosetting resins that contain at least one epoxide group per molecule[1]. They have many applications in industry. The U.S. alone produces more than 500 million pounds of epoxy resins each year[2]. This can be attributed to their many advantages, such as mechanical strength, flexibility, and protection of substrates from corrosion, to name a few[3]. The focus of this study is to examine the kinetics involved in the crosslinking reactions of hydroxyl- and carboxyl-functionalized copolymers with cycloaliphatic epoxides.

Epoxy resins can be crosslinked with a variety of different compounds, such as hydroxyl- and carboxyl-functionalized copolymers. This makes epoxides an ideal choice for crosslinking copolymers containing both functional groups. The competitive reaction of cyclohexene oxide with methanol and acetic acid, as shown in *Scheme 1*, is representative of the crosslinking reactions of cycloaliphatic epoxides with hydroxyl- and carboxyl-functionalized copolymers, respectively. The reactions can be either base- or acid- catalyzed[4]. This study will focus on the kinetics of the competitive reaction of cyclohexene oxide with methanol and acetic acid without catalyst. This represents the low pH portion of the reaction. The temperature dependence of the reaction will also be explored, carrying out the reaction under three different temperatures.

The kinetics of the competitive reaction will be explored to determine rate constants, energies of activation, and possible reaction mechanisms. This has been addressed in the past, but not as extensively under the conditions of interest[4]. This is important since the linkages that form with either functional group will give different properties. The ester linkages formed by reaction of cycloaliphatic epoxides with carboxyl-functionalized copolymers have better photo-oxidative stability, whereas the ether linkages that result from reaction with hydroxyl-functionalized copolymers give better hydrolysis resistance[4]. A desirable coating has a combination of both ether and ester linkages. The kinetic parameters will give insight into how to achieve an optimum combination of these linkages.



Scheme 1. Proposed crosslinking reaction of cycloaliphatic epoxides with hydroxyl- and carboxyl-functionalized acrylic copolymers[4].

Experimental

Materials. Cyclohexene oxide ($C_6H_{10}O$, CE) (98 %), methanol (CH_3OH, MeOH) (99.9%), acetic acid (CH_3COOH, HOAc) (99.8 %), and dichloroethane (ClC_2H_4Cl, DCE) (99.9 %) were all purchased from Aldrich and distilled before use.

Instrumentation. A 5890 HEWLETT PACKARD Series II gas chromatograph with FID detector and HP 3396 Series II integrator was used to analyze and record quantitative data for the reactions. The separation column used was an intermediate polar capillary column (DB17, 30 M × 0.53 mm ID, J & W Scientific). The optimum column separation was obtained using an initial temperature of 60 °C, and then at a ramp rate of 10 °C/min to reach the final temperature. The final temperature was set to 250 °C in order for all the components to elute out of the column. The carrier gas used was hydrogen, and the splitting ratio was set to 100:1, with a purge of 3.0 mL/mm. The detector operation temperature was set to 280 °C and injection temperature was set to 280 °C. The pH value of the reaction solutions was directly measured with a Corning pH Meter 320. The temperature of each reaction was controlled using a constant temperature water bath MD 20 LAUDA (± 0.1 °C).

Basic procedure for kinetic studies. The competitive reactions at low pH were performed at varying ratios of cyclohexene oxide, methanol, and acetic acid. All reactions took place at a constant pH of 2.05 with dichloroethane as the solvent. The reactions were performed at three temperatures (25 °C, 35 °C, and 45 °C) in order to construct an Arrhenius plot. The experimental conditions used to determine reaction orders are shown in *Table 1*.

Table 1. Reaction Conditions

Reaction Order Determination for	Cyclohexene Oxide (M)	Methanol (M)	Acetic Acid (M)	pH	Temp. (°C)
Cyclohexene Oxide	0.5	2.0	2.0	2.05	35
	0.25	2.0	2.0	2.05	35
	0.125	2.0	2.0	2.05	35
Methanol	2.0	0.5	2.0	2.05	35
	2.0	0.25	2.0	2.05	35
	2.0	0.125	2.0	2.05	35
Acetic Acid	2.0	2.0	0.5	2.05	35
	2.0	2.0	0.25	2.05	35
	2.0	2.0	0.125	2.05	35
---	2.0	1.0	1.0	2.05	25, 35, 45

The total volume for all reactions was 10 mL. All reactions were performed in a 50 mL flask equipped with a stir bar and septum. Methanol and acetic acid were added to dichloroethane and allowed to equilibrate in the constant-temperature water bath for ~5 minutes. At this time, cyclohexene oxide was added to the reaction flask using a syringe. All reactions were carried out for 120 hours, taking aliquots out for GC analysis at 4, 8, 12, 24, 36, 48, 72, 96, and 120 hours.

Sampling and GC analysis. An internal standard method was used to calculate concentrations of reactants and products. The internal standard chosen was cyclohexanol. At each time interval, a 100 μL aliquot was taken out of the reaction flask through the septum and diluted with 100 μL of an internal standard solution (0.2667 M, in dichloroethane). Each sample was then immediately injected into the gas chromatograph for quantitative analysis.

Kinetic equations. The rate of reaction can be described either by the disappearance of a reactant or by the appearance of product. Since the competitive reactions occur concomitantly, the reaction rate was derived by the formation of products[4]. In a reaction of reactants A and B giving product P, the rate equation for formation of product can be shown as follows:

$$d[P]/dt = k[A]^n \times [B]^m \qquad (1)$$

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(8)

Macromolecules 2001, 34, 415–424

Atom Transfer Radical Copolymerization of Methyl Methacrylate and *n*-Butyl Acrylate

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Received July 10, 2000

ABSTRACT: Copolymers of methyl methacrylate and *n*-butyl acrylate were prepared using atom transfer radical polymerization catalyzed by CuBr/4,4′-di(5-nonyl)-2,2′-bipyridine (dNbpy), CuBr/*N,N,N,N′,N″*-pentamethyldiethylenetriamine (PMDETA), and CuBr/tris[2-(dimethylamino)ethyl]amine (Me_6TREN). Point estimates and 95% joint confidence intervals of the reactivity ratios were calculated for the copolymerizations catalyzed by CuBr/dNbpy ($r_{MMA} = 2.52$, $r_{BA} = 0.265$) and those catalyzed by CuBr/PMDETA ($r_{MMA} = 3.15$, $r_{BA} = 0.37$) using previously described least-squares calculations. Semilogarithimic plots of monomer conversion vs time were nearly linear. Plots of M_n vs monomer conversion were linear, and polydispersity decreased with increasing monomer conversion. Copolymerizations were simulated using Predici 5.3.2 in order to better understand the experimentally observed results.

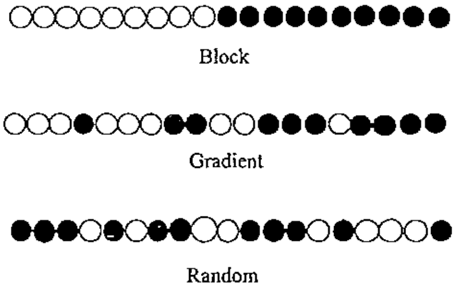
Introduction

The past several years have witnessed significant growth ~~in the~~ field of controlled/living radical polymerization (CRP).[1,2] Atom transfer radical polymerization (ATRP) is one CRP technique in particular that has proven to be very robust and a versatile method for the polymerization of a wide range of monomers.[1–6] Additionally, ATRP has been shown to be effective for the synthesis of well-defined polymers with many different types of architectures and sequential compositions.[7–12] There are also several reports of the use of ATRP to prepare a novel class of materials, gradient copolymers.[13–15]

Gradient copolymers are copolymers in which the instantaneous composition of the polymer changes continuously from one end of the chain to the other.[14–16] As shown in Scheme 1, this is in contrast to block copolymers, which have no change in composition until the crossover from one block to the other, and random copolymers, which have no continuous change in instantaneous composition. To achieve this continuous change in instantaneous composition, all chains must be initiated simultaneously and must survive until the end of the polymerization. Therefore, a living (ionic) or controlled/living radical polymerization technique must be employed, as the significant presence of chain breaking reactions would lead to heterogeneity in composition as well as in molecular weight.

Two types of gradient copolymers can be synthesized: controlled gradients and spontaneous gradients. ① Controlled gradient copolymers are prepared in semibatch copolymerizations, with controlled addition of one monomer to the other during the course of the polymerization. This results in composition gradients that are manipulated by the forced change in monomer feed composition. ② Spontaneous gradients are formed in batch copolymerizations, where the significance of the composition gradient is determined exclusively by the reactivity ratios of the monomer pair and the initial monomer feed concentration. Our group has previously reported on the semibatch copolymerizations of styrene/acrylonitrile[17] and styrene/*n*-butyl acrylate,[18] the batch copolymerizations of styrene/acrylonitrile[17] and styrene/*n*-butyl acrylate,[19] and preliminary results for methyl methacrylate/*n*-butyl acrylate[20] using ATRP. Reports from other laboratories have also demonstrated the usefulness of ATRP in the synthesis of gradient or statistical copolymers.[21–26]

Scheme 1. Schematic Representation of the Composition in Block, Gradient, and Statistical Copolymers, in Which the Open Circles Denote Monomer 1 and the Closed Circles Denote Monomer 2



It is the purpose of this paper to compare the results of the ATRP batch copolymerization of methyl methacrylate (MMA) with *n*-butyl acrylate (*n*-BA) using different catalytic systems. It has previously been shown that both of these monomers can be homopolymerized in a controlled fashion by ATRP,[27,28] and we now intend to show that they can also be simultaneously copolymerized in a well-controlled manner using different copper/ligand complexes. This monomer pair is of interest due to the large differences in the monomer reactivity ratios in a free radical process.[24,25,29–33] It is anticipated that this difference will lead to a large spontaneous gradient in instantaneous composition along the growing polymer chain. Living or controlled/living polymerizations will allow the formation of a continuous gradient in composition along the chain, which is in direct contrast with conventional radical polymerization. In conventional radical polymerizations, a large difference in reactivity ratios would lead to one monomer being preferentially consumed first, resulting in polymer chains with high MMA content during the initial stages of the polymerization and polymers with high *n*-BA content at the later stages of the polymerization. In a

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Emulsion copolymerizations of methyl acrylate with methyl methacrylate and with acrylonitrile

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Abstract—Emulsion copolymerizations of methyl acrylate (MA) with methyl methacrylate (MMA) and with acrylonitrile (AN) were carried out employing sodium lauryl sulphate (SLS) as a surfactant, potassium persulphate (KPS) as an initiator, and water as a dispersion medium at 70°C. The copolymers were characterized by FTIR, NMR, and TG/DTA techniques. Another series of emulsion copolymerization experiments were carried out employing HYOXYD X400 as a co-surfactant, along with SLS, and keeping all the other experimental conditions the same. The reactivity ratios of these monomer pairs were evaluated by employing the graphical procedures by Fineman–Ross (F–R) and Kelen–Tüdös (K–T). The K–T method yielded the reactivity ratios 0.95 ± 0.05 (r_{MA}) and 2.05 ± 0.05 (r_{MMA}), for the monomer pair MA/MMA, and 1.84 ± 0.05 (r_{MA}) and 0.98 ± 0.05 (r_{AN}) for the monomer pair MA/AN. When HYOXYD *X*400 was employed as a co-surfactant in the emulsion copolymerization experiments, the reactivity ratios were 0.75 ± 0.05 (r_{MA}) and 2.17 ± 0.05 (r_{MMA}) for the monomer pair MA/MMA, and 1.23 ± 0.05 (r_{MA}) and 0.89 ± 0.05 (r_{AN}) for the monomer pair MA/AN. These reactivity ratios obtained for the monomer pairs MA/MMA and MA/AN deviate from the values obtained by bulk and solution copolymerization methods reported in the literature.

Keywords: Emulsion copolymerization; reactivity ratios; characterization.

1. INTRODUCTION

Copolymerization is the most general and powerful method of effecting systematic changes in polymer properties, and is widely used in the production of commercial polymers and in fundamental investigations of structure–property relationships. The reactivity ratios for copolymerizing monomers have become important in predicting the types of monomers that will combine to give polymers and in determining the regularity of such polymeric materials.

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[*] To whom correspondence should be addressed.

LA POLIMERIZACIÓN CONTINUA EN EMULSIÓN DE COPOLÍMEROS VINÍLICOS

José A. Acle
Technical Consultant

RESUMEN

La polimerización continua en emulsión presenta una oscilación cíclica de propiedades debida a las alteraciones en la formación de las partículas y en la conversión a lo largo del proceso, sin embargo, estas variaciones periódicas se pueden eliminar con un pre-polímero formado por una pequeña cantidad de monómero y todo el tensioactivo ó coloide que interviene en la reacción. Este pre-polímero, que corresponde al periodo de pre-nucleación en el que se forman las partículas primarias, se utiliza como un aditivo en la continua para que las partículas solo crecerán sin posibilidad de formarse otras nuevas.

La polimerización continua de los monómeros vinílicos líquidos puede realizarse en reactores convencionales o tubulares. Los primeros son los mas utilizados, con tiempos de residencia largos (3-4 horas) y en los que se producen las oscilaciones descritas, mientras que los tubulares se usan mucho menos, generalmente no tienen oscilaciones periódicas, los tiempos de residencia son cortísimos (4-8 min) y se pueden utilizar con los monómeros gaseosos que se polimerizan a presión, si se consigue una buena conversión por medio de sistemas catalíticos redox de alta eficacia, y por la unión de varios reactores en serie.

INTRODUCCIÓN

Los polímeros en emulsión han tenido un desarrollo espectacular en los últimos treinta años La gran diversificación de estos productos y sus muchas posibilidades de aplicación (adhesivos, pinturas, recubrimientos de papel, textiles, etc.,) han aumentado

⑩

ENHANCED ACRYLIC TECHNOLOGY FOR AUTOMOTIVE TOPCOAT FINISHES.

by
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ABSTRACT

The Rohm and Haas Company has developed a novel acrylic polymer technology for use in topcoat finishing formulations. Products making use of this unique polymer technology can deliver performance comparable to many polyurethane based materials, while providing the aesthetics and cost efficiency that can only be achieved with acrylic polymers. The key enabler in this technology is an acrylic bound-duller that provides comparable gloss, "jetness" and performance properties, at comparable loadings to many commercially available polyurethane based dulling materials. The technology is inherently solvent free, and is compatible with older acrylic ~~and PUD~~ resin components as well as conventional cross linking chemistries. This new technology will provide leather finishers with additional options in high quality finishes with enhanced economics.

INTRODUCTION

Because of numerous possibilities in the chemical structure and particle morphology, acrylic emulsion polymers continue to be optimized and employed in a broad array of coating applications.[1-5] The key applications include; architectural paints for interior and exterior decorative and structural surfaces, industrial coatings for factory-applied coatings on metal, plastic, wood, and cementitious substrates as well as field applied coatings for industrial maintenance (structural steel and traffic paint), building products for construction applications, adhesives and sealants, graphic arts, textile/non-woven fabrics and leather (wet end and finishing). In each of these applications acrylic chemistry is broad enough to provide a balance of aesthetic value, performance and cost effectiveness. Continual demands for cost effective materials with improved properties drive innovation to provide new acrylic technologies in all of the markets described above. Although performance criteria are different for all of the applications, the inherent properties of all acrylic polymers are light stability, low solvent demand and outstanding film formation and pigment binding.

At present, for leather applications, acrylic polymers play a large role in wetend re-tan and basecoat finishing applications. For wetend applications acrylics provide softening, lightfastness and strength to the crust.[6-9] In basecoats, acrylic polymers are used to provide outstanding print, cut-through resistance, flexibility, soft feel and aesthetic value, water and chemical resistance.[10] This superior performance is generally provided with a cost advantage over other binder technologies.

Ultimately, it is our goal to deliver an acrylic technology, for use in topcoats, that would provide not only key wear and appearance properties, but also softer feel/handle and enhanced economics. Although, there are already acrylics especially designed for top coat use and these commercial acrylic materials provide acceptable wear properties they typically come with a sacrifice in softness. Historically, when the key criterion is high wear performance, acrylics have played a secondary role for topcoats. Typically, polyurethane polymers are considered the polymer of choice for higher wear applications in topcoat formulations. It has been challenging for a single polymer technology to balance soft feel, good wear performance with cost effective economics. Recent advances in acrylic polymer technology have changed that paradigm.

Wear performance is measured by the ability of the leather finish to resist abrasive rubbing. Passing key wear tests such as the Pilling, Wyzenbeek and Gakushin abrasion tests require that the finish be resistant to extensive rubbing against a rough, abrasive surface such as "duck" cloth, while Taber testing requires the finish to be resistance to an abrasive, stone-like wheel. In most cases, in addition to the abrasion of the cloth or wheel, there is also a force applied (weight) to stress the finish and hasten the failure. Under these testing conditions it is very typically the hardest of surfaces that have the best resistance to wear. Finish hard-

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*A **Technical Note** based on a presentation at the 100th annual meeting of the American Leather Chemists Association at the Chase Park Plaza, St, Louis, Missouri on June 16 - 20, 2004

⑪

El acabado del cuero con polímeros carbonil funcionales *

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Resumen

El objetivo de esta comunicación ha sido el estudio eminentemente práctico bajo el punto de vista de aplicación, de una serie de polímeros en base acuosa, con funcionalidad carbonílica reticulable con compuestos amínicos.

En el estudio se comparan estos polímeros con otros tipos, tanto en base acuosa como en base solvente, para conocer el máximo posible alcanzable de las prestaciones a conferir al acabado del cuero.

Con los resultados obtenidos se puede ampliar el abanico de utilización de polímeros, tanto en capas de fondo como en tops.

Estos polímeros se consideran interesantes para "shoe finishing", por sus características intrínsecas de ser auto brillante y auto reticulables.

En el estudio práctico se contempla:
- *Grado de reticulación de los polímeros vs. Propiedades*
- *Grado de reticulación de los polímeros vs. Tiempo*
- *Envejecimiento del acabado*
- *Comparación de sus propiedades (flexiones y frotes secos y húmedos) vs. Polímeros acrílicos convencionales, poliuretanos en dispersión acuosa lacas nitrocelulosa*

Palabras clave: polímeros, funcionalidad carbonílica, reticulación, acabado, cuero.

Summary «Leather finishing with carbonyl functional polymers»

The aim of this communication is the eminently practical study under application point of view, of some water based carbonyl-functional polymers crosslinkable with amino derivatives.

In this study, these polymers have been compared with other types of polymers, water based and solvent based, to know the maximum performance possible for leather finishing.

With the obtained results it is possible to extend the range of utilization of polymers as base coat and top coat.

These polymers are considered to be interesting for "shoe finishing", due to their glossy and self-crosslinking properties.

In the practical study it is contemplated:
- *Degree of reticulation of the polymer vs. Properties*
- *Degree of reticulation of the polymers vs. Curing time*
- *Ageing of finished article*
- *Comparison of their properties (flexes and dry and wet rubs) vs. Common acrylic polymer, water based polyurethane and nitrocellulose lacquers.*

Keywords: polymers, carbonyl functional, self-crosslinking, leather finishing

1. Introducción

La seguridad en el trabajo y la protección medioambiental son dos asignaturas presentes hoy en día en cualquier aspecto de la actividad industrial y una preocupación constante de una forma totalmente responsable para la industria del curtido.

La preocupación creciente desde hace varias décadas, para obtener productos para el acabado del cuero respetuosos con el medioambiente y para aumentar la seguridad en los espacios de trabajo y la salud de los trabajadores, fue una de las causas que condujeron a la sustitución progresiva de los polímeros en base solvente a polímeros en base acuosa.

Sin duda alguna uno de los aspectos relevantes en el estudio de dichos sustitutos fue la investigación de polímeros capaces de conseguir las altas prestaciones físicas exigidas por el mercado del cuero diseñándose para este propósito polímeros con grupos funcionales capaces de reaccionar entre ellos para así aumentar las resistencias físicas sin perder las propiedades intrínsecas de flexibilidad y elasticidad.

Recibido: Mayo 2006.

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*Comunicación presentada en el 55 Congreso de la Asociación Química Española de la Industria del Cuero (AQEIC), Elche (Alicante), del 4 al 7 de Mayo del 2006.

⑫

Macromolecules 2006, 39, 8999–9009 8999

Cp_2TiCl-Catalyzed Epoxide Radical Ring Opening: A New Initiating Methodology for Graft Copolymer Synthesis

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ABSTRACT: The first example of the use of epoxides in radical grafting copolymerizations was exemplified by the grafting of poly(methyl methacrylate) (PMMA), poly(butyl methacrylate) (PBMA), poly(butyl acrylate) (PBA), and poly(styrene) (PSt) from poly(glycidyl methacrylate) (PGMA) and from copolymers of GMA with MMA and St as well as by the iterative synthesis of mixed arm graft copolymers such as (PGMA-*g*-PMMA)-*g*-PSt and ((PGMA-*g*-PMMA)-*g*-PSt)-*g*-PBMA with a wide range of molecular weights and compositions. The grafting was demonstrated by a combination of gel permeation chromatography (GPC), NMR, and differential scanning calorimetry (DSC) investigations. The polymerization is initiated by the Cp_2TiCl-catalyzed radical ring opening of the epoxide group of GMA and is optionally controlled by $CuBr_2$/bipyridyl. This methodology does not require any epoxide protection/deprotection steps and provides the typical advantages of radical polymerizations and convenient access to complex macromolecular architectures.

Introduction

Graft copolymers contain side-chain branches emanating from different points along the polymer backbone.[1] Variations in the nature of the main chain and side chains and in the length and polydispersity of the backbone and branches as well as in graft density[2] determine the properties and the associated complexity of the synthetic effort. Well-defined graft copolymers can be prepared by the "onto", "through", and "from" major grafting protocols. In the "grafting onto" process, end-functionalized polymer chains are attached to the main chain of another polymer via coupling reactions with functional groups along its backbone.[3] However, difficulties associated with the poor control over the quantitative coupling have confined the "grafting onto" method to a narrow range of applications. The "grafting through" approach is based on the synthesis of a well-defined macromonomer, followed by its copolymerization with a low molecular weight comonomer.[4] Although control over length and polydispersity can be achieved for both backbone and side chains, the grafting density is controlled by the reactivity ratios. The "grafting from" process is based on the synthesis of a macroinitiator containing suitable initiating groups along the backbone.[5] The high initiator efficiency, the ability to manipulate initiator distribution along the main chain, and the side chain length control afforded by living polymerization techniques make the "grafting from" process the preferred option in the synthesis of well-defined graft copolymers. The ability of living radical polymerization (LRP) to control molecular weight and polydispersity and its multiple advantages over ionic living polymerizations have enabled it to become one of the most efficient and robust synthetic methods in modern polymer chemistry. The molecular weight (M_n) and polydispersity (M_w/M_n) control in LRP is afforded by the reversible termination of growing chains with persistent radicals[6] or degenerative transfer (DT) agents[7] and proceeds mechanistically via either atom transfer (ATRP),[7,8,14] dissociation–combination (DC),[9,10] or degenerative transfer (DT)[7,11–13] processes. The wide applications of LRP in the synthesis of complex, well-defined macromolecular structures[14–16] including block, graft, hyperbranched, and multiarm stars copolymers have also stimulated extensive efforts in the development of novel initiators and catalytic systems.

Currently, in addition to applications in α-olefin coordination polymerizations[17] and organometallic reactions,[18] there is increased interest in the radical organic chemistry of early transition metals.[19] One of the most successful examples, the paramagnetic $Cp_2Ti(III)Cl$[20] complex, is inexpensively available from the reduction of $Cp_2Ti(IV)Cl_2$ with Zn.[21] Cp_2TiCl is a very mild one-electron-transfer agent which catalyzes a variety of radical reactions,[22] including the radical ring opening (RRO) of epoxides[23] and the single electron transfer (SET)[24] reduction of carbonyls and their pinacol coupling.[25] These reactions can occur enantioselectively[26] even under aqueous conditions.[27] We have recently extended the use of Cp_2TiCl to radical polymer chemistry and demonstrated the first examples of an early transition metal catalyzed living radical polymerization of styrene initiated by epoxide RRO,[28] aldehyde SET reduction,[29] or peroxides.[30] The effect of ligands,[31] reducing agents,[32] solvents, and additives[33] as well as reagent ratios and temperature was also investigated. This study revealed the superiority of sandwich metallocenes over alkoxide and half-sandwich ligands and the relatively weak influence of the Cp substituents. Gratifyingly, the most promising catalyst (Cp_2TiCl_2) was also the least expensive one.[34] Furthermore, Ti alkoxides generated in situ by epoxide RRO catalyze the living ring-opening polymerization of cyclic esters.[35]

Typical LRP initiators for metal catalyzed polymerizations are based either on redox processes involving late transition metal complexes and activated alkyl halides or on thermal systems.[14] Thus, living grafting copolymerization by ATRP via the "grafting from" method requires the presence of activated halides along the polymer backbone. Consequently, the main chain cannot be synthesized directly in a controlled fashion via ATRP, unless the halide is masked[36] at the expense of the increase in the number of synthetic steps. Therefore, a wider

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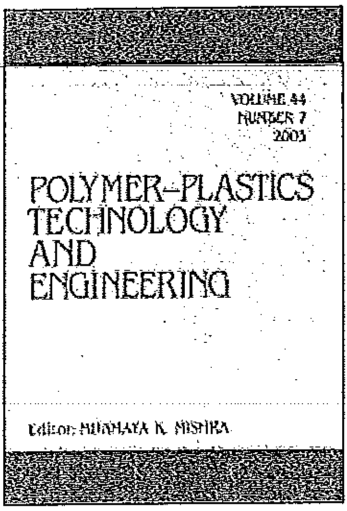

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Modification of Natural Leather by Grafting Emulsion Copolymerization Technique

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⑮

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Synthesis of alkali-soluble copolymer (butyl acrylate/acrylic acid) and its application in leather finishing agent

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ABSTRACT

Alkali-soluble copolymer (butyl acrylate/acrylic acid) was synthesized via solution polymerization and used as the emulsifier to prepare acrylic resin for leather finishing agent. The influence of the synthetic conditions, such as the contents of acrylic acid and the initiator types on the properties of P(BA/AA) was investigated in detail. Fourier Transform Infrared Spectrometer (FTIR) indicated that the polymerization reaction of P(BA/AA) was complete without "—C=C—" absorption peak. Differential Scanning Calorimeters (DSC) analysis confirmed that the glass transition temperature (T_g) of P(BA/AA) was −44 °C, Transmission Electron microscope (TEM) indicated that the copolymer latex particles dispersed evenly and were less than 100 nm. Moreover, in contrast to acrylic resin prepared with sodium dodecylsulfate (SDS) and alkylphenol ethoxylates (OP-10) as the emulsifiers, the applied properties of light leather finished by acrylic resin in use of P(BA/AA) as the emulsifier were measured: The air permeability increased by 18.5% as well as the water-resistance by 28.08% and the wet rub fastness by half class, respectively.

1. Introduction

With the improvement of the environment safeguards and the finishing materials requirement, water-soluble finishing materials has become the research hotspot in the leather finishing agent. However, the water-solubility finishing agent prepared via the conventional emulsion polymerization also exhibited the disadvantages [1]: the large use of small molecular surfactants pollute the environment and limit the finishing materials properties such as the freezing stability, the mechanical stability, the wet and dry rubbing resistance and so on. In order to avoid these disadvantages, The polymer surfactants was applied to the emulsion polymerization [2–9]. Liu et al. [3] reported that the alkali-soluble copolymer P(methyl methacrylate/ethyl acrylate/methacrylic) was used to synthesize the emulsion polymerization of *n*-butyl methacrylate as the surfactants and the nucleus-shell latexes of 40–80 nm were obtained. Sun et al. [4] prepared a novel macromolecule surfactant of PS21-acrylamide/acrylic acid/MMA copolymer via the homogeneous polymerization. Muller et al. [5] used polyelectrolyte block copolymers to prepare the emulsion of 100 nm. Doug et al. [6] studied the preparation of PBA emulsion in the presence of polystyrene/α-methylphenylene/AA copolymer; Satoshi et al. [7] investigated the kinetic process of styrene emulsion polymerization with MMA/methacrylic acid copolymer as the emulsifiers.

Although the results that the properties of the macromolecule emulsifiers are better than low-molecular-weight emulsifier have been reported already, the macromolecule emulsifiers usually have be used after the purification. In a word, the process seems to be time-consuming. If the mixture of the macromolecule emulsifiers with the excellence emulsifier property was obtained without the separation, this will be useful for the industry manufacture.

In this paper, the mixture of the macromolecule emulsifiers synthesized via solution polymerization without any purification ways was employed to be copolymerized with

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CROSS-LINKED POLYMERS FOR AQUEOUS FINISHING. BINDERS CROSSLINKED WITH POLYAZIRIDINE. PART I: BEHAVIOUR OF POLYURETHANE

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Abstract

Aqueous finishing emerges as an alternative to solvent based finishes, which have to be limited due to their toxicity, negative effects on the environment and in terms of safety.

It is necessary, in order to be able to introduce aqueous systems progressively, to use cross-linkers that react with polymers. Otherwise, it is not possible to obtain the physical properties required for most leather goods.

The present study aims to look into the behaviour of polyurethane binders cross-linked with polyaziridine: the amount of cross-linker used, the type of polymer employed, the type of tannage and retannage effected and the evolution of the reaction over time.

A comparison of results shows that the type of tannage and retannage, as well as the physical characteristics of the polyurethane used are influential factors in the behaviour of polyurethane binders cross-linked with polyaziridine. Different amounts of polyaziridine are needed with regard to the type of tannage and retannage effected and to the physical characteristics of the polyurethane nsed.

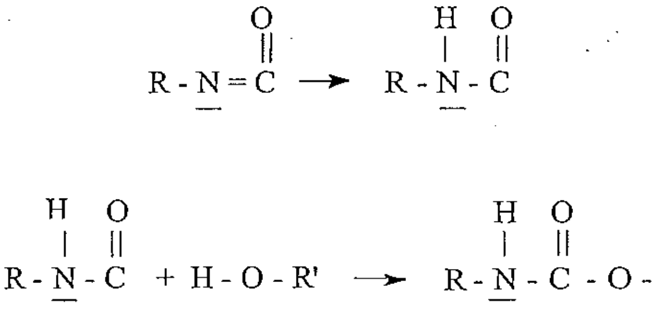
Introduction

One of the main environmental problems in the process of leather finishing is the production of atmospheric emissions and wastewaters. Therefore, it is necessary to eliminate, or at least, to reduce the content of solvent in finish recipes.

Aqueous finishing emerges as an alternative to solvent based finishes, which have to be limited due to their toxicity, negative effects on the environment and in terms of safety. As compared to solvent based finishes, the aqueous finish presents advantages such as: inflammable products do not have to be used, nor stored, environmental and working conditions are healthier, and atmosphere emissions of volatile organic compounds (VOC) are reduced. Also, it allows for waste products to be greatly reduced and this leads to savings.

Polymeric binders are products capable of forming, on drying, a film on the surface they are applied to and represent the basic element in an aqueous leather finish formulation. Three types of polymeric binders with a different chemical base are used in leather finishing: acrylic, butadiene and polyurethane. All of them have specific properties according to their chemical base. Hence it is essential to choose the most suitable polymer in order to obtain the desirable effect. In this study, we focused on polyurethane binders.

Polyurethane binders contain carbamate or urethane groups in their structural unity and are formed by the reaction of an isocyanate group (R - N = C = O) with a hydroxyl group (OH). The reaction of the isocyanate group starts by breaking the double bond located between the nitrogen and the carbon, reacting with products which contain active hydrogens. The hydrogen's donor group binds to the carbon, whereas the hydrogen binds to the nitrogen. Its structure and reaction is shown in Figure 1.



$$\mathrm{R-\underline{N}=\overset{\overset{\displaystyle O}{\|}}{C}} \longrightarrow \mathrm{R-\overset{\overset{\displaystyle H}{|}}{\underline{N}}-\overset{\overset{\displaystyle O}{\|}}{C}}$$

$$\mathrm{R-\overset{\overset{\displaystyle H}{|}}{\underline{N}}-\overset{\overset{\displaystyle O}{\|}}{C}} + \mathrm{H-O-R'} \longrightarrow \mathrm{R-\overset{\overset{\displaystyle H}{|}}{\underline{N}}-\overset{\overset{\displaystyle O}{\|}}{C}-O-}$$

Figure 1. Structure of isocyanate and its reaction.

Chemical products

The basic chemical products in a polyurethane synthesis are the following: diisocyanates, polyols (polyethers or polyesters), solvents (organic or aqueous) and catalysers.

Diisocyanates are compounds which have two isocyanate groups of the aromatic or aliphatic type.

Polyols are compounds which at least have two hydroxyl groups. When condensed with water, polyethers are derivates of ethylene oxide and propylene. Polyesters are products resulting from the polycondensation of saturated carboxylic acids with saturated diols or triols.

The solvents used during the polymerisation must maintain both the isocyanate and polyurethane formed in solution. Mixtures of solvents which do not contain active hydrogens and water are generally used, such as xylene, toluene, ethyl acetate, butyl acetate, acetone, methylethylketone, methyl-isobutylketone, dimethylformamide or isopropanol.

Catalysers are used in the production of aliphatic polyurethanes due to their low reactivity. They are usually metallic salts of organic compounds such as dibutyl tin dilaureate, zinc octanate and tertiary amines of the triethylamine or N-methylformolin type.

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NUBUCK FINISHING WITH WATER-BASED CARBONYL-FUNCTIONAL POLYMERS

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Summary

The process of leather finishing causes environmental pollution. It produces both wastewaters and emissions to atmosphere.[1] The present paper details the study of a new coating formulation intended to minimize the negative environmental effects of this process. This study tests an alternative method of nubuck finishing imitating full grain using using water-based carbonyl-functional polymers and micro-dispersed pigments. These new polymers can achieve very good physical properties without losing the inherent properties of flexibility and elasticity. The application of these new products together with micro-dispersed pigments enables the reduction of the amount of products needed and, therefore, makes it possible to reduce wastewaters and atmospheric emissions.

The leather finished using this new coating formulation is soft, with a very natural handle; with only 0.02mm thick coating, all the necessary requirements for leather goods are met and, most importantly, a great capacity for covering defects is achieved.

Introduction

The process of leather finishing causes environmental pollution. On the one hand, the process results in wastewaters, and on the other it produces atmospheric emissions.[1] In order to reduce the pollution in finishing processes, there has been a significant decrease in the use of solvent-based finishes during the last two decades.[2]

An alternative to the use of solvent-based products is an aqueous finish. The problem with aqueous finishes is that they are unable by themselves to provide the physical properties required for most leather goods.[3] Cross-linkers are employed to solve this problem. Cross-linkers are chemical compounds that react with the functional groups of the polymer chains, linking them and forming a branched polymer. Suitable cross-linkers for leather finishing are those which allow the cross-linking mechanism to start at room temperature and in a fairly short time.[3-7] Unfortunately, the cross-linkers used at present are toxic as they react irreversibly with both the reactive groups of the binders and the reactive groups of our cellular substance. In other words, they tend to cause allergic reactions to the skin. Some mutagenic tests have given moderately positive results and indicate cases of allergy.[8]

Due to the need of new aqueous cross-linking systems, acrylic polymers with carbonylfunctional groups are being developed. The carbonyl group can react readily with multi-functional diamines or polyamines, hydrazides or semicarbazides contained in the finishing product itself.

The cross-linking mechanism of water-based carbonyl-functional polymers has been published in literature[9-10] as shown in Figure 1.

$$2RR'C = O + H_2N\text{-}R''\text{-}NH_2 \rightarrow RR'C = N\text{-}R''\text{-}N = CRR + 2\,H_2O$$

Figure 1. Cross-linking mechanism of water based carbonyl-functional polymers.

Emulsions of water-based acrylic polymers with carbonyl-functional groups form stable systems with multi-functional amines since they are in different phases. A cross-linking mechanism is established during film formation owing to the fact that water is produced in the condensation reaction. This cross-linking system is rapid at room temperature without the disadvantage of toxicity typical of cross-linkers such as polyaziridine. This allows for the design of water-based, self-cross-linkable acrylic polymers. These polymers can achieve very good physical properties without losing their inherent properties of flexibility and elasticity.[9]

This paper tests an alternative method of nubuck finishing imitating full grain by using a water-based acrylic polymer with carbonyl-functional groups. The application of this new product together with micro-dispersed pigments allows us to obtain a high capacity in covering defects while preserving the natural appearance of the leather.

Micro-dispersed pigments are aqueous pigment preparations with anionic charge containing small amounts of protective colloid, free of casein and other film-forming substances. These micro pigments can be used to formulate finishes with a natural appearance, excellent ageing resistance and very high fastness. The finished leather has a very fine grain.

Exeimental ocedure

Material

The tests were carried out on Spanish chrome tanned cattle hides shaved at 1.1-1.2mm. The hides were first retanned using synthetics, tannins and resins; then fatliquored using phosphate ester, lanolin and beef tallow. Finally, the hides were dried (vacuum-air) and buffed twice with a 320 grit buffing paper. The basecoats used were applied (in three applications) using forced air spraying. The base coated crusts were then sprayed with a top coat and pressed.

Finally, leathers were milled and toggled.

The chemicals employed in the operation are those normally used in the leather finishing industry. The chemical products used in finishing process are:

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nº 4

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CROSS-LINKED POLYMERS FOR AQUEOUS FINISHING. BINDERS CROSS-LINKED WITH POLYAZIRIDINE. PART II: BEHAVIOUR OF ACRYLIC BINDERS

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Abstract

Due to the current stringent regulations regarding emissions into the environment, coating recipes commonly used in leather finishing comprise water-based products. However, in order to obtain the physical properties required for most leather goods, the use of reactive cross-linkers is needed.

The present study aims to look into the behaviour of acrylic binders cross-linked with polyaziridine: the amount of cross-linker used, the type of polymer employed, the type of tannage and retannage and the evolution of the reaction over time.

A comparison of results shows that the type of tannage and retannage, as well as the physical characteristics of the acrylic binder used are influential factors in the behaviour of acrylic binders cross-linked with polyaziridine. Different amounts of polyaziridine are needed with regard to the type of tannage and retannage and to the physical characteristics of the acrylic binder used.

Introduction

Polymeric binders are products capable of forming, on drying, a film on the surface to which they are applied and represent the basic element in an aqueous leather finish. Three types of polymeric binders with a different chemical base are mainly used in leather finishing: acrylic, butadiene and polyurethane. All of them have specific properties according to their chemical base. Hence, it is essential to choose the most suitable polymer in order to obtain the desirable effect. In this study, we focused on acrylic binders.

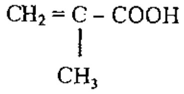
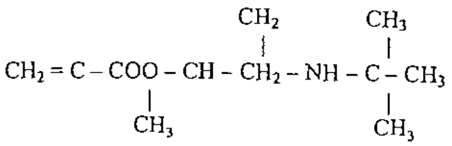
Acrylic binders are formed from the polymerisation of monomers containing carboxylic groups, amide groups or amine groups as can be seen in Table I.

The characteristics of the polymer vary depending on the base monomer, the degree of polymerisation, the molecular mass of the polymer, the type of esterification of the side chains and the number of intramolecular and intermolecular bonds.

The main properties of acrylic binders are: good light fastness, thermoplasticity, poor coverage, good adherence and low volatile organic compounds (VOC) content. However, they present low chemical resistance and low mechanical properties.[1,2]

To obtain the physical properties required for most leather goods, it is necessary to use cross-linkers that react with polymers. Cross-linkers are chemical compounds which react with the reactive groups in the polymeric

TABLE I.
Monomers most commonly employed to produce acrylic binders.

With carboxylic groups	Acrylic acid	$CH_2=CH-COOH$
	Metacrylic acid	 $CH_2=C(CH_3)-COOH$
With amide groups	Acrylamide	$CH_2=CH-CONH_2$
	Methylolacrylamide	$CH_2=CH-CONH-CH_2OH$
	Terbutylaminoethyl-metacrylate	 $CH_2=C(CH_3)-COO-CH(CH_2)-CH_2-NH-C(CH_3)_2-CH_3$

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(9) (18)

JSLTC, 2009 ([redacted])

n.º 3

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BINDERS CROSS-LINKED WITH POLYAZIRIDINE. STUDY OF CROSS-LINKED POLYMERS FOR AQUEOUS FINISHING. PART III: INFLUENCE OF A CATIONIC PRE-BOTTOM

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Abstract

The leather industry needs innovative products and processes to satisfy the requirements of a continuously evolving global market. Modern leathers are processed to very stringent technical specifications so that they will meet end use requirements. In most cases, to fulfil current demands for leather with very high fastness, the use of cross-linkers is needed.

Due to the differences in the manufacturing processes of polyurethanes and acrylic binders, their structural differences, and the type of tanned and retanned leather being used, variations in cross-linking may occur. Quality discrepancies will therefore be observed in the finished leather.

This study explores the influence of a cationic pre-bottom on the cross-linking reaction of polyaziridine, as well as the influence of the type of polymer employed and the type of tannage and retannage effected on the amount of cross-linker needed to improve wet physical properties of the finishing.

A comparison of results shows that the application of a cationic pre-bottom improves fastness to wet rub, this improvement being most significant in polyurethane rather than in acrylic binders. Furthermore, the cationic pre-bottom provides better results when working with resin-synthetic-vegetable retanned leather and vegetable leathers. However, different amounts of polyaziridine are needed with regard to the content of carboxylic acid groups of the polymers being used.

Introduction

The leather industry needs innovative products and processes to satisfy the requirements of a continuously evolving global market. Modern leathers are processed to very stringent technical specifications so that they will meet end use requirements. In most cases, to fulfil current demands for leather with very high fastness, the use of cross-linkers is needed.

Polyurethane and acrylic resins are widely employed for leather finishing to give different responses to a multitude of quality standards and market trends.[1] The manufacturing processes for these products are quite different. Acrylates are produced by low pressure polymerisation whereas polyurethanes by polyaddition.[2]

Acrylic binders are made by free radical emulsion polymerisation of acrylic and methacrylic esters and acids, sometimes in combination with acrylonitrile or styrene.[3]

Polyurethanes are usually produced by a two-stage polyaddition reaction process. In the first stage, diisocyanates and polyols react to form pre-polymers, which still contain reactive terminal groups and are reacted in the second stage to produce the final product.[4]

Both types of binders can react with polyaziridine at room temperature to improve wet physical properties of the finish.[5-9] Polyaziridine can react with the carboxylic groups of acrylic and polyurethane binders.

In two previous studies,[10-11] the behaviour of polyurethane and acrylic binders cross-linked with polyaziridine was analyzed. Although polyaziridine improves wet physical properties of the finish such as wet rub fastness, float resistance and wet bending, different amounts of polyaziridine are required depending on the type of tannage and retannage effected and on the physical characteristics of the polymer used.

Each type of tanned and retanned leather has a specific overall charge and different absorption power. Hence, the aim of the present paper is to explore if a cationic pre-bottom can change the power of fixation of subsequent applications and adjust the power of absorption of the leather.

A cationic pre-bottom formulation consists of an acrylic cationic resin of very fine particle size, a cationic wax and a cationic binder. With this formulation, the anchorage will be obtained by anion/cation chemical assembly between the leather surface (if it is anionic), cationic pre-bottom and subsequently, anionic base coat.

With a cationic pre-bottom grain absorptions are improved, defects are covered, levelling and coverage are enhanced and, consequently, the leathers obtained are more natural.

Methodology

In order to study the influence of the cationic pre-bottom on the power of fixation and absorption of each type of tanned and retanned leather, the tests were carried out on three types of tannages: (1) chrome tanned leather with a chrome retannage at 33% basicity; (2) vegetable tanned leather; and (3) chrome tanned leather with an acrylic resin-synthetic-vegetable retannage.

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AQUEOUS FINISHING WITH POLYISOCYANATE CROSS-LINKED BINDERS

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Summary

Due to increasingly stringent environmental legislation the leather industry has to produce eco-friendly articles. At the same time, it needs to mannfacture fashionable and high-quality articles in order to increase competitiveness. As part of this, factories have to replace solvent-based products by water-based products in their finishing formulations to reduce the level of volatile organic compounds. The addition of a cross-linker is required in most cases in order to obtain high performance aqueous finishes. The aim of the present work is to explore the cross-linking reaction between polyisocyanate and both acrylic binders and polyurethanes.

A comparison of results shows that polyisocyanate presents a very fast reticulation [cross-linking] speed at room temperature and has a pot-life of only 4 hours. In order to obtain optimum fastness values, different amounts of polyisocyanate are required, the amounts depend on the type of leather used and on the physical characteristics of the binders employed.[1-3]

Introduction

Different types of cross-linkers are available for use in leather finishing: aziridines, isocyanates, carbodiimides and epoxies, all can react with polymeric chains, however; they have different characteristics and reaction rates.[6] We have explored in four previous studies,[7-10] the cross-linking differences between acrylic binders and polyurethanes with polyaziridine and with epoxy resin. In the present work, we focus on polyisocyanate cross-linker.

Isocyanates are polyfunctional aggregates with a high reactivity and contain the R-N=C=O group. The reaction of the isocyanate group starts by breaking the double bond located between the nitrogen and the carbon, and then reacting with products which contain active hydrogens. The hydrogen's donor group binds to the carbon, whereas hydrogen binds to the nitrogen, as can be seen in Figure 1.[6]

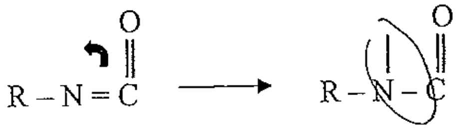

Figure 1. Reaction initiation of the isocyanate group.

Isocyanates react with carboxyl groups (-COOH), anhydride groups (-OH), water (H_2O), as well as with the amino group (-NH) and with amide groups (-NH-CO). These reactions are shown in Figure 2.[11]

Polyisocyanates are obtained by modification of diisocyanates and aliphatic or aromatic which are chosen depending on the required property. Aromatic isocyanates show a faster reaction rate than aliphatic isocyanates. Figure 3 shows the diisocyanates most commonly used in industry.[12]

We used a commercial aliphatic polyisocyanate which contains approximately 12% of NCO. This product is colourless or slightly yellowish and can be added to the finish without having to be mixed in advance.

Methodology

We designed a coating formulation in order to study the cross-linking differences between acrylic binders and polyurethanes with polyisocyanate. This formulation can be seen in Table I.

TABLE I
Coating formulations

	Base coat
100	Pigment
100	Wax
150	Binder
450	Water
200	Acrylic/polyurethane binder*
X	Polyisocyanate**
	Spray 8 wet g/ft^2
	Plate 80°C/80 bar/1"
	Top coat
100	Nitrocellulose lacquer
100	Water
5	Silicone
	Spray 2 wet g/ft^2
	Plate 80°C/80 bar/1"
	Tests after 7, 14 and 31 days' storage

* The amount of acrylic/polyurethane binder depends on the solid content.

** Addition of polyisocianate was 0-4-8-12 parts.

To assess variations in cross-linking reaction depending on the type of leather employed, tests were carried out on three types of leathers: (1) chrome tanned leather with a chrome retannage at 33% basicity; (2) vegetable tanned leather and (3) chrome tanned leather with an acrylic resin-synthetic-vegetable retannage.

The chemicals employed were those normally used in leather finishing. To evaluate the differences in cross-linking reaction depending on the type of binder used, four

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EPOXY RESIN CROSS-LINKED BINDERS IN AQUEOUS FINISHING

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Summary

Nowadays, the leather industry needs to produce a very wide range of finished articles with different levels of performance to increase their competitiveness while minimizing their environmental impact. To reduce the negative effects on the environment a strong shift from solvent to water-based recipes has taken place in leather finishing. However, most of the water-based high-performance articles require the addition of a cross-linker[1-5] to improve wet-fastness properties. The aim of the present work is to study the cross-linking reaction between an epoxy cross-linker and both acrylic binders and polyurethanes.

A comparison of results shows that epoxy resin improves wet rub fastness significantly. The cross-linking reaction evolves with time within 31 days storage. Moreover, different amounts of epoxy resin are required to obtain optimum values depending on the type of leather used and on the physical characteristics of the binders employed.

Introduction

At present, four types of cross-linkers are marketed by several companies: aziridines, isocyanates, carbodiimides and epoxies. All these products have different characteristics and interactions so that variations in cross-linking may occur, changing the properties of the final article.[6]

In three previous studies,[7-9] the behaviour of polyaziridine was analysed. This study focuses on epoxy cross-linkers; on the cross-linking reaction between an epoxy cross-linker and acrylic binders and polyurethanes: the amount of cross-linker needed, the influence of the physical characteristics of the binders, the influence of the tannage and retannage, as well as the evolution of the reaction over time.

The simplest epoxy is a three membered ring structure known as an alpha-epoxy or 1,2-epoxy. Figure 1 shows this structure.

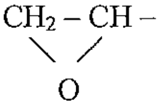

Figure 1. Structure of epoxy group.

Epoxy resins are long-chain molecules with reactive sites at either end. The epoxy molecule also contains two ring groups at its centre which are able to absorb both mechanical and thermal stresses better than linear groups and therefore, give the epoxy resin very good stiffness, toughness and heat resistance properties. Moreover, due to the absence of ester groups, the epoxy resin has particularly good water resistance. Figure 2 shows the molecular structure.

Epoxy compounds react with carboxyl groups (-COOH), anhydride groups (-OH) and amino groups (-NH) and thus, aside from being used as resins, they can also be used as cross-linking agents. The reaction of epoxy groups with carboxyl groups starts by opening the ring and two different reaction products are formed: on the one hand, an ester of the primary hydroxyl group, and on the other hand, the ester of the secondary hydroxyl group (a). Then, if the reaction temperature is high enough, a complete esterification of the reaction products takes place (b). Also, additional reactions to form ether groups can take place in presence of an excess of epoxy groups (c). These reactions are shown in Figure 3.

The reaction of epoxy groups with primary amine also starts with ring opening and one of the hydrogen atoms of the amine bonds with the oxygen of the epoxy group, forming an hydroxyl group and the primary amine is reduced to a secondary amine. In the second stage of the reaction, the secondary amine reacts with another epoxy group. These reactions are shown in Figure 4.[10-13]

In this work, we used a commercial epoxy-resin-dispersion, containing approximately 64% solids. This product is whitish; water miscible liquid of medium viscosity that prior to addition must be diluted with water (1:1).

Methodology

In order to study the behaviour of four acrylic binders and four polyurethanes cross-linked with epoxy resin, a coating formulation was designed as can be seen in Table I. First, a base coat was applied in four applications using air

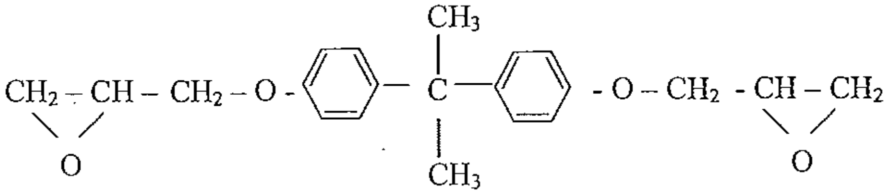

Figure 2. Epoxy molecular structure.

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Carboxyl-terminated butadiene-acrylonitrile-toughened epoxy/carboxyl-modified carbon nanotube nanocomposites: Thermal and mechanical properties

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Abstract. Carboxyl-modified multi-walled carbon nanotubes (MWCNT–COOHs) as nanofillers were incorporated into diglycidyl ether of bisphenol A (DGEBA) toughened with carboxyl-terminated butadiene-acrylonitrile (CTBN). The carboxyl functional carbon nanotubes were characterized by Fourier-transform infrared spectroscopy and thermogravimetric analysis. Furthermore, cure kinetics, glass transition temperature (T_g), mechanical properties, thermal stability and morphology of DGEBA/CTBN/MWCNT–COOHs nanocomposites were investigated by differential scanning calorimetry (DSC), dynamic mechanical analysis (DMA), universal test machine, thermogravimetric analysis and scanning electron microscopy (SEM). DSC kinetic studies showed that the addition of MWCNT–COOHs accelerated the curing reaction of the rubber-toughened epoxy resin. DMA results revealed that T_g of rubber-toughened epoxy nanocomposites lowered with MWCNT–COOH contents. The tensile strength, elongation at break, flexural strength and flexural modulus of DGEBA/CTBN/MWCNT-COOHs nanocomposites were increased at lower MWCNT-COOH concentration. A homogenous dispersion of nanocomposites at lower MWCNT–COOH concentration was observed by SEM.

Keywords: nanocomposites, thermal properties, thermosetting resin, carbon nanotubes, epoxy resin

1. Introduction

Epoxy resins are widely used as coatings, adhesives, insulating materials and matrices for fibrous composites for their rigidity, high temperature performance, chemical resistance and adhesive properties. However, the inherent brittleness due to the high crosslinking density becomes one of the biggest troubles in advanced application use in industries. Therefore, great efforts have been exerted in the modifications of epoxy resins for further improvement of multifunctional properties [1, 2].

Presently, reactive liquid rubber, thermoplastics, interpenetrating polymer networks (IPNs), thermotropic liquid crystal and nanofillers are used to toughen epoxies for improving properties of epoxy networks. The elastomeric toughening is the most frequently used and widely accepted among these methods. Recently, Tripathi and Srivastava [1, 3] reported that a two-phase morphology was observed and the best balance of mechanical properties were achieved with a concentration of carboxyl-terminated butadiene-acrylonitrile (CTBN) ranging between 15 and 20 phr (part per hundred resin) in the cured epoxy resin. Varley [4] effectively toughened the diglycidyl ether of bisphenol A (DGEBA) by both epoxy-terminated aliphatic polyesters and CTBN with a great improvement of the fracture properties whereas the addition of aminopropyl-ter-

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Effect of Carboxyl Functionalized MWCNTs on the Cure Behavior of Epoxy Resin

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ABSTRACT

Effects of carboxyl functionalized MWCNTs on the cure behavior of epoxy resin (EPIKOTE™ resin 862 and EPICURE™ curing agent W) were investigated by using differential scanning calorimeter in dynamic scan mode. Results indicated that nanotubes could initiate cure reaction at lower temperature while only 0.1 wt% samples were able to suppress the peak temperature (T_p) consistently regardless of different heating rate due to their higher reaction rate, other two batches (0.2 wt% and 0.3 wt% CNT) showed hardly any change in T_p compared to neat samples. Furthermore, addition of small amount (0.1 wt%) of CNTs significantly reduced the activation energy of the system but further addition elevated that due to their retarding effect. Also, a correlation was established between the dispersion state of CNTs in epoxy and total heat of reaction (ΔH) where lower ΔH was considered as an indication of better interfacial interaction of CNTs with matrix and hence, better dispersion. The difference in curing behavior of three different CNT percentages was discussed in terms of their steric hindrance and state of dispersion in the matrix.

Keywords: Carbon Nanotubes; DSC; Steric Hindrance; Peak Temperature; Activation Energy

1. Introduction

Owing to their unique structural integrity, carbon nanotubes (CNTs) have emerged as potential reinforcements because of their outstanding thermal, mechanical and electrical properties [1-5]. Higher specific surface area along with better stress transfer capability has made them an ideal candidate for high performance composite in structural application. Naturally, researchers have gone through a number of attempts to enhance the properties of polymer materials by incorporating CNTs. In most cases, these new composites, incorporated with CNTs exhibit excellent thermal, mechanical and electrical properties. Although numerous works have been reported on developing CNT incorporated polymeric composites, translation of properties is yet well below the theoretical expectation.

Among the various thermosetting polymers, epoxy is one of the most popular resins utilized in various industrial applications. Epoxies have found their application in coating, adhesive, aerospace and electronics industries [6-8] because of their excellent mechanical and chemical properties as well as high heat distortion temperature. The superior mechanical and chemical properties possessed by the epoxy polymer are a result of curing reaction, in which a low molecular weight resin is converted to an infinite molecular weight polymer with a three-dimensional network structure through chemical reaction or physical interlocking or both. A wide range of curing agents like aliphatic or aromatic amines, anhydrides etc. are used to initiate the cure reaction. CNTs in epoxy not only improve the load bearing and heat resistant capacity but also affect the curing process through different mechanism and alter the ultimate properties of final composites.

Even though the CNT incorporated epoxy composites have shown improvement in mechanical properties but it is not to the same degree in various studies [9-12]. For example, Allaoui *et al.* [9] found that 1 wt% CNT improved the young's modulus by 100% whereas Zhu *et al.* [10] found only a moderate improvement adding the same quantity. This variation may be attributed to the state of dispersion, orientation of CNT particles as well as the interface between the particle and the matrix. Though dispersion is regarded as one of the prerequisite to fabricate composite, there is no reasonable way to evaluate it over the whole specimen. In most recent efforts, optical microscope, scanning electron microscope

United States Patent Office

3,431,226

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3,431,226

ACRYLIC POLYMER EMULSIONS

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Claims priority, application Great Britain, Nov. 19, 1963, 45,537/63
U.S. Cl. 260—29.7 5 Claims
Int. Cl. C08f *1/13, 15/40, 37/00*

ABSTRACT OF THE DISCLOSURE

Acrylic polymer emulsions which are thickenable on addition of alkali comprising a polymer of at least one acrylic monoester, a copolymerizable acid monomer and a cross linking monomer having at least two unsaturated groups are prepared by polymerizing the monomer mixture in at least three separate stages and allowing at least 85% of the monomers added at each stage to polymerize before adding the monomers of the following stages. Latices of these polymers exhibit superior viscosity stability when thickened with alkali.

This invention relates to acrylic polymer emulsions, particularly emulsions for use in coating compositions. Such coating compositions are applied, for example, to leather and leather-like synthetic materials by known methods of padding, spraying or "curtain-coating." It is often important to the method of application that the emulsion should have a low solids content but be thickened i.e. have relatively high viscosity, and it is also important that the thickening should remain reasonably constant during at least the period of application. It is an object of this invention to provide an emulsion which can be thickened by the addition of alkali to a controlled reproducible and reasonably constant viscosity. By an "acrylic polymer emulsion" we means a dispersion of acrylic polymer particles in water.

According to the present invention an acrylic polymer emulsion which is controllably thickenable on addition of alkali comprises a polymer of at least one acrylic monomer with a copolymerizable acid and a small percentage of a cross-linking monomer.

The present invention also provides a process for preparing an acrylic polymer emulsion which is controllably thickenable on addition of alkali comprising polymerizing in emulsion at least one acrylic monomer with a minor proportion of a copolymerizable acid and a small percentage of a cross-linking monomer.

The polymerization is normally carried out in no more than five separate stages. The preferred number of stages is three, the total mixed monomers being added in proportions of 40 percent at the first stage, 30 percent at the second stage and 30 percent at the third stage. At each stage the monomers present are allowed to polymerize substantially so that at least 85 percent of the amount of monomers present has polymerized before the next addition of monomer is made. At each addition of monomers, polymerization is reinitiated by further addition of initiator.

It is preferred to use a cross-linking monomer which is at least partially water-soluble. Suitable cross-linking monomers are compounds having vinyl or allyl unsaturation containing at least two unsaturated groups capable of entering into polymerization or copolymerization. The preferred cross-linking monomers are diesters of acrylic or methacrylic acid, in particular ethylene glycol dimethacrylate and triethylene glycol dimethacrylate.

The small perecntage of cross-linking monomer which it is necessary to add will vary according to the number of polymerizable units in each molecule of the cross-linking monomer. In general, the percentage by weight of the cross-linking monomer multiplied by the number of polymerizable units in each molecule divided by its molecular weight should not exceed 0.04.

The copolymerizable acid should not be present to such an extent that it renders the emulsion alkali-soluble. It is preferred that the copolymerizable acid should amount to 8–15 percent of the total weight of mixed monomers. Suitable acids are acrylic and methacrylic acid.

The term "acrylic polymer" is used in this specification to include interpolymers of an acrylic monomer with one or more further acrylic monomers and/or with one or more nonacrylic monomers. In the latter case the polymer usually contains at least 37 percent by weight of acrylic monomers. Acrylic monomers include acrylic or methacrylic esters, acrylic or methacrylic acids or derivatives thereof. When the emulsions of this invention are used in coating compositions, pigments and fillers may be added or the emulsions may be blended with other emulsions or resins, natural oils or waxes.

It is usual when preparing an emulsion for transportation, storage etc. to produce an emulsion of fairly high solids content, i.e. of the order of 50 percent. For application as a coating composition an emulsion is diluted down to about 15 percent to 20 percent solids content. The particle size is preferably of the order of 0.1 micron, and the viscosity is substantially equal to that of water. When alkali is added thickening occurs so that the viscosity increases to give a reading on the Brookfield viscometer equivalent to a viscosity of the order of 50–200 poise. Since the thickened emulsion is a nonNewtonian fluid, the term "viscosity" as applied to it must be understood to mean no more than the property measured by the Brookfield viscometer.

The advantage of the emulsions of this invention over emulsions prepared from a polymer not containing a cross-linking monomer is that when alkali is added to the emulsions of this invention the thickening which takes place is controllable and reproducible and the thickened condition is maintained. Emusions prepared from acrylic monomers alone by a gradual addition of monomers during polymerization rather than a separate-stage process as herein disclosed were found to gel completely on addition of a very small amount of alkali in excess of the amount which caused no thickening.

Emusions prepared by a separate-stage process but not in the presence of a cross-linking monomer were found to be thickenable, but they tended to continue thickening to a gel condition or to thin down again with time.

The emulsions of this invention are thickenable on addition of alkali and not soluble in alkali. Therefore they result in a surface coating which has good water resistance and alkali resistance. This applies whether or not pigments are included in the coating composition.

The applicant's problem is illustarted in the following control experiments, and the invention is illustrated in the following examples, all parts being by weight.

Various terms used throughout the specification have been abbreviated for convenience, and these are as follows:

TDOS=Tetrasodium-N-(1,2 - dicarboxyethyl) - N - octadecyl sulphosuccinamate (35 percent active)
F10=Alkyl phenylether containing about 10 ethylene oxide units
F35=Alkyl phenylether containing about 35 ethylene oxide units
X200=Sodium salt of an alkylaryl polyether sulphonate

nited States Patent ce

3,817,946
Patented June 18, 1974

3,817,946

ACRYLIC RESINS CONTAINING EPOXY FUNCTIONALITY

Buren R. Ree, Village of Afton, Minn., assignor to Minnesota Mining and Manufacturing Company, St. Paul, Minn.

No Drawing. Filed Feb. 2, 1973, Ser. No. 329,161
Int. Cl. C08d 3/02
U.S. Cl. 260—79 4 Claims

ABSTRACT OF THE DISCLOSURE

Solid, low molecular weight, storable epoxy-functional acrylic terpolymers of glycidyl methacrylate, a lower alkyl acrylate and methyl methacrylate and the process for their preparation.

SUMMARY OF THE INVENTION

This invention relates to solid, low molecular weight, storable epoxy-functional acrylic terpolymers of glycidyl methacrylate, a lower alkyl acrylate and methyl methacrylate and the process for the preparation. These terpolymers are of particular use in thermosettable powder coating formulations.

BACKGROUND OF THE INVENTION

The powder coating technique in general involves application of a finely divided organic powder to an article and the subsequent conversion of the powder through application of heat to an adherent continuous coating. It is particularly well suited to modern assembly line operation, especially when protective and/or decorative coatings are to be applied to metal articles. The organic powder is applied to the article by one of the known methods, such as the fluidized bed and electrostatic spray methods, and then heated to cause it to fuse and flow into a continuous coating on the article being coated and to subsequently become thermoset. The thermoset coatings resulting from these powders are tough, solvent resistant and weather resistant. Unless pigments, dyes or other such materials are added, they are clear and highly glossy.

Thermosetting powder coatings have certain recognized advantages, both in the coating process and in the properties of the final coating. Thus, no solvent or primer is needed, single coat coverage is possible and there is no sagging of the film. Also, there is often an improved hardness-flexibility relationship and the films have more universal chemical and solvent resistance.

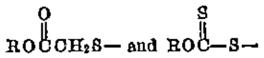
Among the classes of thermosetting coating powders are those based on epoxy, polyester and acrylic systems. These may be combined and/or modified, e.g., epoxy-acrylic and polyester-acrylic systems can be used. They can also be modified with other crosslinking components such as unsaturates and aminoplasts. More extensive information relative to the thermosetting powder coating field is provided in the review entitled "Thermosetting Polyester and Acrylic Powder Coatings," D. D. Taft, R. Hong and W. J. McKillip, publication number FC 72–942 of the Society of Manufacturing Engineers, 20501 Ford Road, Dearborn, Mich. 48128.

One economically important difficulty with powder coated surfaces is an aesthetic one, i.e., their characteristic "orange peel" appearance. This is caused by their characteristic surface configuration which gives an appearance similar to that of the peeling of an orange. Agents which have been used in the past to reduce the orange peel have had the additional effect of increasing the "blocking" of the powder (i.e., the tendency of the particles to agglomerate, resulting in failure of the powder to flow freely and thereby make coating difficult or impossible). Thus, attempts to alleviate one serious problem in powder coatings has brought on another problem which has been perhaps, more serious.

THE PRESENT INVENTION

The present invention relates to a class of solid, low molecular weight, storable epoxy-functional acrylic terpolymers which have a unique combination of properties and which provide distinct advantages to powder coating compositions in which they are incorporated. Thus, they combine (1) relatively low melt viscosities resulting, on fusion, in substantial flow and continuous coatings; (2) glass transition temperatures above the temperatures of grinding and storage, providing ease of grinding to powder form and stability (freedom from blocking) in powder form; and (3) upon application of heat in the presence of particular compounding systems, conversion to tough, adherent, solvent resistant coatings. Powder coating compositions in which they are included are thus easily used and form strongly adhering, weatherable, solvent resistant coatings. The terpolymers may also be used to make thermosetting molding powders and low viscosity, high solids, solvent based enamels.

More specifically, the invention relates to terpolymers of 5–20 parts of glycidyl methacrylate units, 5–35 parts of lower alkyl acrylate units in which the alkyl group contains from 2 to 4 carbon atoms, 50–70 parts of methyl methacrylate units, and 0.5–10 parts, preferably 2–5 parts on a weight basis (1–2 parts on a molar basis) of chain terminating groups selected from



$$\mathrm{RO\overset{O}{\overset{\|}{C}}CH_2S—}\text{ and }\mathrm{RO\overset{S}{\overset{\|}{C}}—S—}$$

wherein each R is an alkyl group containing not more than 20 carbon atoms, the sum of the four types of units being 100 parts by weight, said terpolymers having

(a) glass transition temperatures of from 20° to 70° C. (preferably from about 30° to about 50° C.)
(b) Durrans melting points of from 90° to 170° C. (preferably from about 110° to about 150° C.)
(c) melt indexes of from 1 to 30 (preferably from about 5 to about 20), when measured at a temperature of 152° C. with a piston loading of 1200 grams and an extrusion orifice of 0.0465 inch, and
(d) epoxy equivalent weights of 700–3000.

A particularly useful class of thermosetting coating powders containing the terpolymers of the invention can be defined as follows:

(1) 100 parts of the terpolymer
(2) 0–20 parts of a crosslinking agent selected from saturated aliphatic dicarboxylic acids having from 5 to 20 carbon atoms which is essentially unreactive at room temperature but which reacts rapidly above 150° C.
(3) 0–40 parts of an acid-terminated linear polyester plasticizer-curative (or plasticizer-crosslinking agent) having an equivalent weight per carboxyl group of from 100 to 1500 and
(4) 0.1 to 5 parts of a non-ionic aliphatic polyacrylate surface active agent,

provided that the ratio of total carboxyl equivalence of (2) plus (3) is 0.5–1.5:1 of the epoxy equivalence of (1).

In the compositions to be used in applications in which particularly smooth coating surfaces are desired (which are free of orange peel) it is also advantageous to add, as a surface leveling agent, 1–10 parts (based on 100 parts of terpolymer) of glyceryl tris(12-hydroxystearate). This compound is a hydrogenated castor oil having an hydroxyl equivalent weight of about 185. It is a wax with a melting point of from about 84 to 87° C. and an iodine value

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United States Patent [19]
Larsson et al.

[11] **3,896,085**
[45] **July 22, 1975**

[54] EMULSION COPOLYMERS OF ACROLEIN AND THEIR USE IN TREATING LEATHER

[75] Inventors: **Bjorn E. Larsson**, Rushland; **Stanley Le Sota**, Horsham, both of Pa.

[73] Assignee: **Rohm and Haas Company**, Philadelphia, Pa.

[22] Filed: **Apr. 12, 1973**

[21] Appl. No.: **350,573**

Related U.S. Application Data

[63] Continuation-in-part of Ser. No. 160,178, July 6, 1971, abandoned.

[52] **U.S. Cl.** .... 260/67 UA; 117/139.5; 117/142; 260/29.6 TA; 260/72 R; 260/73 R
[51] **Int. Cl.** C08f 15/12
[58] **Field of Search** .... 260/67 UA

[56] **References Cited**

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3,455,861	7/1969	Bresciani et al.	260/67 UA X
3,733,286	5/1973	Holly et al.	260/67 UA X

Primary Examiner—Lucille M. Phynes
Attorney, Agent, or Firm—Terence P. Strobaugh

[57] ABSTRACT

Emulsion copolymers containing (a) from about 0.25 to about 4% by weight acrolein, (b) from about 0.5 to about 10% by weight of an ethylenically unsaturated carboxylic acid, and (c) up to about 99.25% by weight of an alkyl acrylate, an alkyl methacrylate, or mixtures thereof are disclosed. The copolymers are useful in leather treating compositions, and particularly in base coat compositions.

7 Claims, No Drawings

United States Patent [19]

Columbus

[11] **4,032,487**

[45] **June 28, 1977**

[54] **AQUEOUS ACRYLATE-EPOXY SILANE CROSSLINKER ADHESIVE DISPERSION COMPOSITION**

[75] Inventor: Peter Spiros Columbus, Whitestone, N.Y.

[73] Assignee: Borden, Inc., Columbus, Ohio

[22] Filed: Apr. 12, 1976

[21] Appl. No.: 676,046

Related U.S. Application Data

[63] Continuation-in-part of Ser. No. 453,245, March 21, 1974, abandoned.

[52] U.S. Cl. .... 260/17 R; 156/329; 260/29.6 H; 260/29.6 TA; 260/29.15 B; 260/29.2 R; 260/29.2 N; 260/824 EP; 260/827; 428/442; 428/426; 428/494

[51] Int. Cl.² C08L 1/28

[58] Field of Search .... 260/29.6 H, 29.6 TA, 260/34, 29.15 B, 29.2 R, 29.2 N, 824 EP, 827, 17 A

[56] **References Cited**

UNITED STATES PATENTS

3,759,915	9/1973	Kotte	260/42.52

Primary Examiner—Edward M. Woodberry
Attorney, Agent, or Firm—George A. Kap; George P. Maskas; Daniel D. Mast

[57] **ABSTRACT**

An adhesive composition which comprises, for each 100 parts by weight thereof, an aqueous dispersion having pH of about 8 to 12 of the following components dispersed in 25 to 75 parts of water:

A. from 25 to 75 parts by weight of water insoluble linear polymer containing the following interpolymerized unsaturated monomers in parts by weight based on the weight of the polymer:
  1. at least 50 parts and up to 99 parts of an acrylate monomer containing from about 4 to 27 carbon atoms selected from alkyl acrylates, alkyl methacrylates, and mixtures thereof:
  2. from about 0.1 to 10 parts of a nitrogen monomer selected from vinyl amines and salts thereof; vinyl ureido monomers; vinyl compounds having heterocyclic nitrogen containing groups and halogen, hydroxyalkyl or aminoalkyl substituted derivatives thereof; amino and loweralkyl amino loweralkyl acrylates and methacrylates; and mixtures thereof;
  3. from about 0.1 to 25 parts of an acid selected from acrylic acid, methacrylic acid and mixtures thereof;

B. from a small but effective amount to effect crosslinking of the polymer to 5 parts of a silane containing both alkoxy and epoxy functionalities.

12 Claims, No Drawings

United States Patent [19]
Finn et al.

[11] 4,136,075
[45] Jan. 23, 1979

[54] ACRYLIC COPOLYMER COATINGS

[75] Inventors: William M. Finn, Decatur, Ill.; Michael C. Peck, Savannah, Ga.; Maynard R. Winstead, West Peabody, Mass.

[73] Assignee: A. E. Staley Manufacturing Company, Decatur, Ill.

[21] Appl. No.: 791,453

[22] Filed: Apr. 27, 1977

[51] Int. Cl.^{2} C08L 33/00
[52] U.S. Cl. .... 260/29.6 TA; 260/29.6 H; 260/29.6 N; 260/29.6 E; 427/338 R; 427/388 C; 427/388 D; 428/215; 428/216; 428/463
[58] Field of Search .... 260/29.6 H, 29.6 TA, 260/29.6 N, 29.6 E; 427/388 R, 388 C, 388 D; 428/215, 216, 463

[56] References Cited

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3,431,226	3/1969	Warson et al.	260/29.6 TA
3,825,514	7/1974	Drury, Jr. et al.	260/29.6 TA
3,980,602	9/1976	Jakubauskas	260/29.6 TA
3,991,216	11/1976	Christenson et al.	220/1 BC
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668832	8/1963	Canada	260/29.6 TA

Primary Examiner—Melvyn I. Marquis
Attorney, Agent, or Firm—M. Paul Hendrickson; Charles J. Meyerson

[57] ABSTRACT

Acrylic copolymer solutions having a T_g less than 0° C., a weight average molecular size ranging from about 20,000 to about 50,000 and consisting essentially of about 4–14% ethylenically unsaturated acid, about 80–95% methyl acrylate and/or ethyl acrylate and about 1–8% lower alkyl methacrylate are obtained by copolymerizing the comonomers in the presence of an organic solvent which functions as a chain terminator. Water-borne, heat-curable, internal can coating compositions may be prepared by formulating these acrylic copolymer solutions with aqueous ammonia and a water-dispersible, cross-linking reagent.

40 Claims, No Drawings

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US005723182A

United States Patent [19]

Choi et al.

[11] **Patent Number: 5,723,182**

[45] **Date of Patent: Mar. 3, 1998**

[54] **METHOD FOR COATING LEATHER**

[75] Inventors: **Chol-Yoo Choi**, Bensalem; **Patricia Marie Lesko**, Ottsville; **Katherine Sue Rice**, Glenside, all of Pa.

[73] Assignee: **Rohm and Haas Company**, Philadelphia, Pa.

[21] Appl. No.: **707,988**

[22] Filed: **Aug. 27, 1996**

[51] Int. Cl.^{6} **B05D 7/12**

[52] U.S. Cl. **427/389**; 524/558; 524/560; 524/561

[58] **Field of Search** 524/558, 560, 524/561; 427/389

[56] **References Cited**

U.S. PATENT DOCUMENTS

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4,491,612	1/1985	Fischer et al.	427/412
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5,185,387	2/1993	Klesse et al.	523/201
5,288,809	2/1994	Christner et al.	427/447

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348565	1/1990	European Pat. Off. .

Primary Examiner—Erma Cameron
Attorney, Agent, or Firm—Ronald D. Bakule

[57] **ABSTRACT**

An aqueous leather coating composition and a method for coating leather with the aqueous coating composition containing a multi-stage emulsion polymer which has been contacted with a transition metal oxide, hydroxide, or carbonate is provided. The leather coating fulfills desirable protective and aesthetic functions.

6 Claims, No Drawings

US 20010038085A1

(19) **United States**

(12) **Patent Application Publication**

(10) **Pub. No.: US 2001/0038085 A1**

Lesko et al.

(43) **Pub. Date: Nov. 8, 2001**

(54) **LEATHER COATING BINDER AND COATED LEATHER HAVING GOOD EMBOSSABILITY AND WET-FLEX ENDURANCE**

(76) Inventors: **Patricia Marie Lesko**, Ottsville, PA (US); **Frederick James Schindler**, Ft. Washington, PA (US)

Correspondence Address:
ROHM AND HAAS COMPANY
PATENT DEPARTMENT
100 INDEPENDENCE MALL WEST
PHILADELPHIA, PA 19106-2399 (US)

(21) Appl. No.: **09/870,576**

(22) Filed: **May 31, 2001**

Related U.S. Application Data

(60) Division of application No. 09/480,204, filed on Jan. 10, 2000, which is a non-provisional of provisional application No. 60/116,979, filed on Jan. 25, 1999.

Publication Classification

(51) **Int. Cl.**^{7} C14C 11/00

(52) **U.S. Cl.** **252/8.57**; 427/389

(57) **ABSTRACT**

An aqueous leather coating composition and a method for coating leather with the aqueous coating composition are provided. The aqueous coating composition contains an aqueous emulsion polymer which includes from 0.4% to 10% by weight of a copolymerized acetoacetate or acetoacetamide monomer and has a glass transition temperature from −20° C. to 10° C. In an alternative embodiment the aqueous coating composition contains an aqueous emulsion polymer which includes from 0.1% to 6% by weight of a copolymerized acetoacetate or acetoacetamide monomer and from 2% to 15% by weight of copolymerized carboxylic acid monomer and has a glass transition temperature from −40° C. to 0° C., the polymer having been contacted with a transition metal oxide, hydroxide, or carbonate at a pH less than 9 in an amount greater than 0.20 equivalent of transition metal per equivalent of copolymerized carboxylic acid monomer.

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US 20020012750A1

(19) **United States**

(12) **Patent Application Publication** (10) **Pub. No.: US 2002/0012750 A1**

Chiang et al. (43) **Pub. Date: Jan. 31, 2002**

(54) LEATHER COATING COMPOSITION

(76) Inventors: **Jin-chih Chiang**, Lansdale, PA (US); **Shang-Jaw Chiou**, Lower Gwynedd, PA (US); **Frederick James Schindler**, Ft. Washington, PA (US)

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(21) Appl. No.: **09/843,777**

(22) Filed: **Apr. 27, 2001**

Related U.S. Application Data

(63) Non-provisional of provisional application No. 60/208,130, filed on May 31, 2000.

Publication Classification

(51) **Int. Cl.^{7}** **B05D 3/02**
(52) **U.S. Cl.** **427/385.5; 521/41**

(57)

ABSTRACT

An aqueous leather coating composition containing a multistage emulsion polymer having a stage of Tg<10° C. which contains a copolymerized carboxylic acid and which has been contacted with a divalent metal oxide, hydroxide, or carbonate and a polymer stage of Tg>20° C. which has been prepared in the presence of 1% to 15% by weight based on the weight of that stage of chain transfer agent is provided. Also, provided is a method for coating leather with the composition of this invention.

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US006586516B1

(12) **United States Patent**
Kesselmayer et al.

(10) **Patent No.:** **US 6,586,516 B1**
(45) **Date of Patent:** ***Jul. 1, 2003**

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(54) AQUEOUS COATING COMPOSITIONS

(75) Inventors: **Mark Alan Kesselmayer**, Harleysville, PA (US); **Theodore Tysak**, Philadelphia, PA (US); **Matthew Stewart Gebhard**, New Britain, PA (US); **Min-Chi Tsai von Trentini**, Yardley, PA (US)

(73) Assignee: **Rohm and Haas Company**, Philadelphia, PA (US)

(*) Notice: This patent issued on a continued prosecution application filed under 37 CFR 1.53(d), and is subject to the twenty year patent term provisions of 35 U.S.C. 154(a)(2).

Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 0 days.

(21) Appl. No.: **08/720,793**

(22) Filed: **Oct. 4, 1996**

Related U.S. Application Data

(60) Provisional application No. 60/004,831, filed on Oct. 5, 1995.

(51) **Int. Cl.**^{7} **C08K 5/01**

(52) **U.S. Cl.** **524/475**; 427/385.5; 427/393; 428/463; 428/484; 428/485; 428/514; 524/477; 524/522; 524/523; 524/558

(58) **Field of Search** 524/558, 522, 524/523, 475, 477; 427/385.5, 393; 428/463, 484, 485, 514

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Primary Examiner—Judy M. Reddick
(74) *Attorney, Agent, or Firm*—Stephen E. Johnson; Kevin F. Gironda; Ronald D. Bakule

(57) **ABSTRACT**

The present invention relates to an aqueous coating composition having low volatile organic compounds (VOC) and producing a coating having excellent durability and resistance properties. The coating composition contains acetoacetate functional polymer and divalent metal ions. Coating compositions of the present invention are particularly useful in ink, polish and coating compositions where a tough, resistant coating is required. The coating composition can be used on various substrates such as floors, walls wood, metal, plastic, stone, paper, leather and concrete.

7 Claims, No Drawings

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US 20040245490A1

(19) **United States**

(12) **Patent Application Publication** (10) **Pub. No.: US 2004/0245490 A1**

Schopke et al. (43) **Pub. Date: Dec. 9, 2004**

(54) TOP COATING COMPOSITION FOR LEATHER

(76) Inventors: **Holger Schopke**, Neckargemund (DE); **Stefan Adams**, Ludwigshafen (DE); **Helfried Scheidl**, Friedelsheim (DE)

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1940 DUKE STREET
ALEXANDRIA, VA 22314 (US)

(21) Appl. No.: **10/488,106**

(22) PCT Filed: **Sep. 9, 2002**

(86) PCT No.: **PCT/EP02/10088**

(30) **Foreign Application Priority Data**

Sep. 28, 2001	(JP)	2001-299972

Publication Classification

(51) **Int. Cl.**^{7} **C14C 9/00**

(52) **U.S. Cl.** **252/8.57**; 524/507; 524/556; 526/303.1; 526/317.1; 526/319

(57) ABSTRACT

A top coating composition for leather comprises

(a) from 20 to 60% by weight of at least one dispersed copolymer A formed from

(a1) from 40 to 70% by weight of at least one ester A1 of methacrylic acid with C_{1-8}-alkanols,

(a2) from 25 to 60% by weight of at least one ester A2 of acrylic acid with C_{1-8}-alkanols,

(a3) from 0 to 3.5% by weight of at least one C_3-C_6 α,β-monoethylenically unsaturated carboxylic acid A3,

(a4) from 0 to 4% by weight of at least one further auxiliary monomer A4 such as hydroxypropyl acrylate, ureidomethacrylate, acrylamide, methacrylamide, N-methylolacrylamide, N-methylolmethacrylamide, A1, A2 and optionally A3 and/or A4 adding up to a total of 100% by weight,

(b) at least 40% by weight of water (B),

(c) from 0 to 40% by weight of polyurethane top coating composition C for leather, A, B and optionally C adding up to a total of 100% by weight and the top coating composition being free of plasticizers and synthetic waxes.

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US006844396B2

(12) **United States Patent**
Sugaya et al.

(10) **Patent No.: US 6,844,396 B2**
(45) **Date of Patent: *Jan. 18, 2005?**

(54) ACRYLIC RESIN COMPOSITION

(75) Inventors: **Takahiko Sugaya**, Houston, TX (US); **Riichi Nishimura**, League City, TX (US); **Kazuhisa Tajima**, Kobe (JP)

(73) Assignees: **Kaneka Corporation**, Osaka (JP); **Kaneka Texas Corporation**, Pasadena, CA (US)

(*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 166 days.

This patent is subject to a terminal disclaimer.

(21) Appl. No.: **10/229,277**

(22) Filed: **Aug. 28, 2002**

(65) **Prior Publication Data**

US 2004/0043235 A1 Mar. 4, 2004

(51) **Int. Cl.**^{7} C08G 63/48; C08G 63/91; C08L 31/02; C08L 33/04; C08L 51/00

(52) **U.S. Cl.** 525/70; 525/71; 525/80; 525/85; 525/222; 525/308; 525/309

(58) **Field of Search** 525/70, 71, 80, 525/85, 222, 308, 309

(56) **References Cited**

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2002/0123568 A1	* 9/2002	Tajima et al.	....	525/85

* cited by examiner

Primary Examiner—Nathan M. Nutter
(74) *Attorney, Agent, or Firm*—Westerman, Hattori, Daniels & Adrian, LLP

(57) **ABSTRACT**

An acrylic resin composition with excellent impact resistance, weatherability, processability and low gloss is provided. The acrylic resin composition comprises, (A) 10 to 40 parts by weight of a high molecular weight acrylic (co)polymer which is a one-step or multi-step polymer containing 72 to 100% by weight of methyl methacrylate and 0 to 28% by weight of a copolymerizable monomer, the (co)polymer having a specific viscosity of at least 0.5 (solvent: toluene, concentration: 0.4%, temperature: 30° C.), wherein a polymer layer obtained in one of the steps contains at least 72% by weight of methyl methacrylate, has a specific viscosity of at least 0.5 and makes up at least 55% by weight of the total weight of the high molecular weight acrylic (co)polymer (A); (B) 0 to 80 parts by weight of an acrylic (co)polymer containing 50 to 100% by weight of methyl methacrylate and 0 to 50% by weight of a copolymerizable monomer, the acrylic (co)polymer having a specific viscosity of less than 0.5; (C) 10 to 90 parts by weight of an impact modifier with a multilayer structure, wherein the total of (A), (B) and (C) is 100 parts by weight and the composition has a melt flow index of 0.35 g/10 minutes to 1.4 g/10 minutes (230° C., 3.8 kg load).

12 Claims, No Drawings

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US 20070106043A1

(19) **United States**

(12) **Patent Application Publication**

Horiuchi et al.

(10) **Pub. No.: US 2007/0106043 A1**

(43) **Pub. Date: May 10, 2007**

(54) METHOD OF PRODUCING ACRYLIC COPOLYMER

(76) Inventors: **Masayoshi Horiuchi**, Ibaraki (JP); **Toshimasa Sagawa**, Ibaraki (JP); **Hanuyoshi Tatsu**, Ibaraki (JP)

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BUTZEL LONG
350 SOUTH MAIN STREET
SUITE 300
ANN ARBOR, MI 48104 (US)

(21) Appl. No.: **10/587,640**

(22) PCT Filed: **Mar. 22, 2005**

(86) PCT No.: **PCT/JP05/05114**

§ 371(c)(1),
(2), (4) Date: **Jul. 28, 2006**

(30) **Foreign Application Priority Data**

Mar. 24, 2004 (JP) 2004-086082

Publication Classification

(51) **Int. Cl.**
C08F 220/22 (2006.01)

(52) **U.S. Cl.** 526/245; 526/303.1; 526/319

(57) ABSTRACT

In the production of an acrylic copolymer by emulsion polymerization of a monomer mixture comprising (a) 30-70% by weight of perfluoroalkylalkyl (meth)acrylate, represented by the following general formula: $CH_2{=}CRCOOR'Rf$ (where R is a hydrogen atom or a methyl group, R' is a linear or branched alkylene group having 1-8 carbon atoms, and Rf is a perfluoroalkyl group having 4-16 carbon atoms), (b) 25-60% by weight of stearyl (meth)acrylate, (c) 0.1-5% by weight of (meth)acrylamide, and (d) 0.1-5% by weight of N-methylol (meth)acrylamide, in the presence of a non-ionic and/or cationic surfactant, a polypropylene glycol-based compound having a molecular weight of 250-5,000 is used as an emulsification aid at the same time. The resulting aqueous dispersion of acrylic copolymer has distinguished preservation stability, and water and oil repellency.

1. Identificación de la sustancia o del preparado y de la sociedad o empresa

Identificación de la sustancia o del preparado:
Referencia del producto: SD0010
Denominación del producto: Sodio laurilsulfato (SDS), para biología molecular

Uso de la sustancia o el preparado:
en bioquímica, para la determinación de: sustancias tensioactivas.
Identificación de la sociedad o empresa:
Empresa:
Scharlau Chemie, S.A.
Ctra. Polinyà-Sentmenat Km. 8,2
08181 Sentmenat (Barcelona) ESPAÑA
Tel. +34 - 93 715 18 11 - FAX +34 - 93 715 31 75
email: info@scharlau.com
Internet Web Site: www.scharlau.com
Representante regional:
Scharlab, S.L.
Gato Pérez, 33. Pol. Ind. Mas d'en Cisa
08181 Sentmenat (Barcelona) ESPAÑA
Tel: +34-93 715 19 39 - FAX +34-93 715 27 65
email: scharlab@scharlab.com
Internet Web Site: www.scharlab.com
Teléfono de urgencias:
Instituto Nacional de Toxicología de Madrid. Tel: +34 - 91 562 04 20

2. Composición/información sobre los componentes

Identificación y cantidad de los componentes:
CAS: 151-21-3
Peso molecular:288,38
Numero CE:205-788-1
Formula:$C_{12}H_{25}NaO_4S$

3. Identificación de peligros

Peligros que presenta la sustancia según las directivas europeas:
Nocivo por ingestión. Irrita los ojos y la piel.

4. Primeros auxilios

Tras inhalación: Tomar aire fresco.
Tras contacto con la piel: aclarar con abundante agua. Quitar la ropa contaminada.
Tras ingestión: beber abundante agua, provocar vómitos. Avisar al médico.
Tras contacto con los ojos: aclarar con abundante agua, con los párpados bien abiertos. Si no desaparecen las molestias, llamar al oftalmólogo.

5. Medidas de lucha contra incendios

Medios de extinción adecuados: Agua, espuma, polvo.
Riesgos especiales particulares: Inflamable. En caso de incendio: posible formación de vapores tóxicos.
En caso de incendio puede formarse: sulfóxidos
Equipo de protección especial para el personal de lucha contra incendios: No permanecer en la zona de peligro sin ropa protectora adecuada y sin sistemas de respiración artificiales e independientes del ambiente.
Información adicional: Precipitar vapores emergentes con agua. Procurar que el agua de extinción no penetre en acuíferos superficiales o subterráneos.

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6. Medidas a tomar en caso de vertido accidental

Precauciones individuales: Procurar no entrar en contacto con la sustancia. Procurar que no se forme polvo; intentar no inhalar el polvo.
Precauciones para la protección del medio ambiente: No verter por el sumidero.
Procedimientos de limpieza: Recoger en seco y eliminar los residuos. Aclarar. Intentar que no se forme polvo.

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7. Manipulación y almacenamiento

Manipulación: Sin más exigencias.
Almacenamiento: Almacenar bien cerrado, seco. Almacenar entre +15°C y +25°C.

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8. Controles de exposición/protección personal

Valores límite de la exposición: (MAK, Alemania):
Controles de la exposición:
Controles de la exposición profesional: Los equipos de protección personal deben elegirse según el puesto de trabajo, en función de la concentración y cantidad de la sustancia peligrosa. El suministrador debería facilitar la estabilidad de los equipos de protección personal frente a los productos químicos.
Protección respiratoria: necesaria cuando se genera polvo.
Protección de las manos: necesaria
Protección ocular: necesaria
Protección cutánea: Se recomienda protección cutánea preventiva.
Medidas de higiene particulares: Cambiar la ropa contaminada. Lavarse las manos tras trabajar con la sustancia.

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9. Propiedades físicas y químicas

Información general:
Aspecto: polvo
Color: blanco
Olor: inodoro
Información importante en relación con la salud, la seguridad y el medio ambiente:
Valor de pH: (10 g/l H2O, 20 °C) 7,5 - 9,0
Punto/intervalo de ebullición: ---
Punto de destello: > 100 °C
Límites de explosión (bajo): ---
Límites de explosión (alto): ---
Presión de vapor: ---
Densidad (20 °C): ---
Solubilidad en agua: (20 °C): 150 g/l
Solubilidad en:
etanol (25°C): 75 g/l
Coeficiente de reparto n-octanol/agua: log P(o/w): 1,6
Viscosidad: ---
Índice de refracción: ---
Punto/intervalo de fusión: 204 - 207 °C
Punto de ignición: ---

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10. Estabilidad y reactividad

Condiciones a evitar: Calentamiento
Materias a evitar: agentes oxidantes fuertes, ácidos minerales.
Productos de descomposición peligrosos: En caso de incendio: véase capítulo 5.

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11. Información toxicológica

Toxicidad aguda:

DL$_{50}$ (oral, rata): 1288 mg/kg

Informaciones complementarias sobre toxicidad:

Tras inhalación: Irritación de las vías respiratorias.
Tras contacto con la piel: irritaciones
Tras contacto con los ojos: irritaciones
Tras ingestión: Absorción por: tracto gastrointestinal.
Tras absorber cantidades importantes: cansancio, vasodilatación.

Información adicional:

No pueden descartarse propiedades peligrosas adicionales.
Este producto debe manejarse con los cuidados especiales de los productos químicos.

12. Informaciones ecológicas

Ecotoxicidad: Tóxico para los organismos acuáticos.
Toxicidad para los peces:
Onchorhynchus mykiss CL$_{50}$: 4,3 mg/l /96h.
L. macrochirus CL$_{50}$: 4,5 mg/l /96h.
Toxicidad de dafnia: Daphnia magna CE$_{50}$: 6,3 mg/l /48h.
Toxicidad para las algas: Selenastrum capricornutum CI$_{50}$: 3,7 mg/l /8d.
Toxicidad para las bacterias: Photobacterium phosphoreum CE$_{50}$: 0,5-1,2 mg/l /30min.
Movilidad: log P(o/w): 1,6
Potencial de bioacumulación: El potencial de bioacumulación será probablemente bajo (log P(o/w) = 1 - 3).
Persistencia y degradabilidad: Biodegradabilidad: > 60% /28d (ensayo en frasco cerrado).
Biodegradabilidad: buena

Observaciones ecológicas adicionales:

¡No incorporar a suelos ni acuíferos!

13. Consideraciones relativas a la eliminación

Producto: Los criterios homogéneos para la eliminación de residuos químicos no están regulados, por ahora, en la Unión Europea. Los residuos, procedentes del uso habitual de los productos químicos, poseen, generalmente, el carácter de residuos especiales. Existen leyes y disposiciones locales que regulan la eliminación de estos residuos en los países de la UE. Para informarse sobre su caso particular, rogamos que se ponga en contacto con la Administración Pública, o bien con una Empresa autorizada para la gestión de residuos.
Envases: Se procederá según las disposiciones oficiales para eliminarlos. Los embalajes contaminados deberán ser sometidos a las mismas medidas aplicadas al producto químico contaminante. Los embalajes no contaminados serán tratados como material reciclable o como residuos domésticos.

14. Información relativa al transporte

Exento de las normas de transporte.

15. Información reglamentaria

Clasificación CE: Este producto no está incluido en el índice de sustancias peligrosas con su número de índice CE correspondiente, por lo que ha sido clasificado siguiendo el anexo VI de la directiva 2001/59/CE.
Pictograma: Xn (Nocivo)
Frases R: 22-36/38 Nocivo por ingestión. Irrita los ojos y la piel.
Frases S: 46 En caso de ingestión, acuda inmediatamente al médico y muéstrele la etiqueta o el envase.
N° de índice CE: ---

16. Otras informaciones

Motivo de la revisión: Actualización general.
Fecha: 26/03/2008

La información suministrada en esta hoja de seguridad, se basa en el estado actual de nuestros conocimientos. El propósito de esta información es únicamente describir las medidas de seguridad en el manejo del producto, y por tanto no constituye una garantía sobre las propiedades del mismo.

Fichas Internacionales de Seguridad Química

ACRILATO DE n-BUTILO

ICSC: 0400

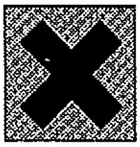
INSTITUTO NACIONAL
DE SEGURIDAD E HIGIENE
EN EL TRABAJO

Butil éster del ácido 2-propenoico
Butil 2-propenoato
$CH_2=CHCOOC_4H_9$ / $C_7H_{12}O_2$
Masa molecular: 128.2

Nº ICSC 0400
Nº CAS 141-32-2
Nº RTECS UD3150000
Nº NU 2348
Nº CE 607-062-00-3

TIPOS DE PELIGRO/ EXPOSICION	PELIGROS/ SINTOMAS AGUDOS	PREVENCION	PRIMEROS AUXILIOS/ LUCHA CONTRA INCENDIOS
INCENDIO	Inflamable.	Evitar las llamas, NO producir chispas y NO fumar. Véanse Peligros Químicos.	Polvo, AFFF, espuma, dióxido de carbono.
EXPLOSION	Por encima de 37°C pueden formarse mezclas explosivas vapor/aire.	Por encima de 37°C, sistema cerrado, ventilación y equipo eléctrico a prueba de explosión.	En caso de incendio: mantener fríos los bidones y demás instalaciones rociando con agua.
EXPOSICION		¡EVITAR LA FORMACION DE NIEBLA DEL PRODUCTO! ¡EVITAR TODO CONTACTO!	
• INHALACION	Sensación de quemazón. Tos. Jadeo. Dolor de garganta.	Ventilación, extracción localizada o protección respiratoria.	Aire limpio, reposo. Proporcionar asistencia médica.
• PIEL	Enrojecimiento. Dolor.	Guantes protectores. Traje de protección.	Quitar las ropas contaminadas. Aclarar la piel con agua abundante o ducharse. Proporcionar asistencia médica.
• OJOS	Enrojecimiento. Dolor.	Gafas ajustadas de seguridad, o protección ocular combinada con la protección respiratoria.	Enjuagar con agua abundante durante varios minutos (quitar las lentes de contacto si puede hacerse con facilidad), después proporcionar asistencia médica.
• INGESTION	Dolor abdominal. Náuseas. Vómitos. Diarrea.	No comer, ni beber, ni fumar durante el trabajo.	Enjuagar la boca. NO provocar el vómito. Dar a beber agua abundante. Proporcionar asistencia médica.

DERRAMES Y FUGAS	ALMACENAMIENTO	ENVASADO Y ETIQUETADO

Eliminar toda fuente de ignición. Recoger el líquido procedente de la fuga en recipientes tapados. Absorber el líquido residual en arena o absorbente inerte y trasladarlo a un lugar seguro. (Protección personal complementaria: Filtro respiratorio para vapores orgánicos y gases.). Traje de protección química. NO permitir que este producto químico se incorpore al ambiente.	A prueba de incendio. Mantener en lugar fresco. Mantener en la oscuridad. Separado de oxidantes fuertes. Almacenar solamente si está estabilizado.	NU (transporte): Ver pictogramas en cabecera. Clasificación de Peligros NU: 3 Grupo de Envasado NU: III CE: Nota: D símbolo Xi R: 10-36/37/38-43 S: 2-9 

VEASE AL DORSO INFORMACION IMPORTANTE

ICSC: 0400 Preparada en el Contexto de Cooperación entre el IPCS y la Comisión Europea © CE, IPCS, 2003

Fichas Internacionales de Seguridad Química

ACRILATO DE n-BUTILO

ICSC: 0400

DATOS IMPORTANTES

ESTADO FISICO: ASPECTO:
Líquido incoloro, de olor característico.

PELIGROS FISICOS:
Los vapores de la sustancia no están estabilizados y pueden polimerizar, bloqueando las válvulas.

PELIGROS QUIMICOS:
La sustancia puede polimerizar espontáneamente debido al calentamiento suave, bajo la influencia de luz y en contacto con peróxidos. Reacciona violentamente con oxidantes fuertes, originando peligro de incendio y explosión.

LIMITES DE EXPOSICION:
TLV: 2 ppm; A4; SEN; (ACGIH 2002).
MAK: 2 ppm, 11 mg/m³; Sh (sensibilización cutánea);
Categoría de limitación de pico: I(2);
Riesgo para el embarazo: grupo D (DFG 2003).

VIAS DE EXPOSICION:
La sustancia se puede absorber por inhalación y a través de la piel.

RIESGO DE INHALACION:
Por evaporación de esta sustancia a 20°C se puede alcanzar bastante rápidamente una concentración nociva en el aire.

EFFECTOS DE EXPOSICION DE CORTA DURACION:
La sustancia irrita los ojos, la piel y el tracto respiratorio. La ingestión del líquido puede dar lugar a la aspiración del mismo por los pulmones y la consiguiente neumonitis química.

EFFECTOS DE EXPOSICION PROLONGADA O REPETIDA:
El contacto prolongado o repetido puede producir sensibilización de la piel.

Punto de ebullición: 145-149°C
Punto de fusión: -64°C
Densidad relativa (agua = 1): 0.90
Solubilidad en agua, g/100 ml: 0.14
Presión de vapor, kPa a 20°C: 0.43
Densidad relativa de vapor (aire = 1): 4.42
Densidad relativa de la mezcla vapor/aire a 20°C (aire = 1): 1.01
Punto de inflamación: 36 °C c.c.
Temperatura de autoignición: 267°C
Límites de explosividad, % en volumen en el aire: 1.3-9.9
Coeficiente de reparto octanol/agua como log Pow: 2.38

La sustancia es tóxica para los organismos acuáticos.

NOTAS

Estabilizadores o inhibidores añadidos pueden influir sobre las propiedades toxicológicas de esta sustancia; consultar a un experto. Hidroquinona e hidroquinona etil éter son los estabilizadores más comunmente usados. NO llevar a casa la ropa de trabajo.

Ficha de emergencia de transporte (Transport Emergency Card): TEC (R)-30S2348
Código NFPA: H 2; F 2; R 2;

INFORMACION ADICIONAL

Los valores LEP pueden consultarse en línea en la siguiente dirección: http://www.mtas.es/insht/practice/vlas.htm	Última revisión IPCS: 2003 Traducción al español y actualización de valores límite y etiquetado: 2003 FISQ: 5-015

ICSC: 0400 **ACRILATO DE n-BUTILO**

NOTA LEGAL IMPORTANTE:	Esta ficha contiene la opinión colectiva del Comité Internacional de Expertos del IPCS y es independiente de requisitos legales. Su posible uso no es responsabilidad de la CE, el IPCS, sus representantes o el INSHT, autor de la versión española.

Fichas Internacionales de Seguridad Química

METACRILATO DE METILO (inhibido)

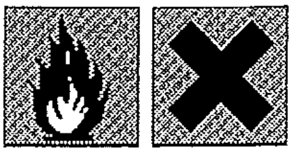
ICSC: 0300

INSTITUTO NACIONAL DE SEGURIDAD E HIGIENE EN EL TRABAJO

2-Metilpropenoato de metilo
$CH_2C(CH_3)COOCH_3$ / $C_5H_8O_2$
Masa molecular: 100.1

Nº ICSC 0300
Nº CAS 80-62-6
Nº RTECS OZ5075000
Nº NU 1247
Nº CE 607-035-00-6

TIPOS DE PELIGRO/ EXPOSICION	PELIGROS/ SINTOMAS AGUDOS	PREVENCION	PRIMEROS AUXILIOS/ LUCHA CONTRA INCENDIOS
INCENDIO	Altamente inflamable.	Evitar las llamas, NO producir chispas y NO fumar.	Espuma, polvo, dióxido de carbono.
EXPLOSION	Las mezclas vapor/aire son explosivas.	Sistema cerrado, ventilación, equipo eléctrico y de alumbrado a prueba de explosión. NO utilizar aire comprimido para llenar, vaciar o manipular.	En caso de incendio: mantener fríos los bidones y demás instalaciones rociando con agua.
EXPOSICION		¡EVITAR TODO CONTACTO!	
• INHALACION	Tos. Jadeo. Dolor de garganta.	Ventilación, extracción localizada o protección respiratoria.	Aire limpio, reposo. Proporcionar asistencia médica.
• PIEL	Enrojecimiento.	Guantes protectores. Traje de protección.	Quitar las ropas contaminadas. Aclarar y lavar la piel con agua y jabón.
• OJOS	Enrojecimiento. Dolor.	Gafas ajustadas de seguridad, o protección ocular combinada con la protección respiratoria.	Enjuagar con agua abundante durante varios minutos (quitar las lentes de contacto si puede hacerse con facilidad), después proporcionar asistencia médica.
• INGESTION	Náuseas. Vómitos. Dolor abdominal.	No comer, ni beber, ni fumar durante el trabajo.	Enjuagar la boca. Dar a beber agua abundante. Proporcionar asistencia médica.

DERRAMES Y FUGAS	ALMACENAMIENTO	ENVASADO Y ETIQUETADO
Recoger, en la medida de lo posible, el líquido que se derrama y el ya derramado en recipientes herméticos. Absorber el líquido residual en arena o absorbente inerte y trasladarlo a un lugar seguro. NO verterlo al alcantarillado. Eliminar toda fuente de ignición (Protección personal complementaria: Filtro respiratorio para vapores orgánicos y gases. Traje de protección química).	A prueba de incendio. Separado de oxidantes fuertes, bases fuertes y ácidos fuertes. Mantener en lugar fresco. Mantener en la oscuridad. Mantener en lugar bien ventilado. Almacenar solamente si está estabilizado.	NU (transporte): Ver pictograma en cabecera. Clasificación de Peligros NU: 3 Grupo de Envasado NU: II CE: Nota: D simbolo F simbolo Xi R: 11-37/38-43 S: 2-24-37-46 

VEASE AL DORSO INFORMACION IMPORTANTE

ICSC: 0300

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Fichas Internacionales de Seguridad Química

METACRILATO DE METILO (inhibido)

ICSC: 0300

DATOS IMPORTANTES

ESTADO FISICO: ASPECTO:
Líquido incoloro, de olor característico.

PELIGROS FISICOS:
El vapor se mezcla bien con el aire, formándose fácilmente mezclas explosivas. Los vapores no inhibidos, pueden polmerizar y bloquear las válvulas.

PELIGROS QUIMICOS:
La sustancia puede polimerizar debido al calentamiento suave o debido al calentamiento intenso, bajo la influencia de luz y oxidantes fuertes, con peligro de incendio o explosión. Reacciona con ácidos fuertes y bases fuertes.

LIMITES DE EXPOSICION:
TLV: 50 ppm como TWA; 100 ppm como STEL; SEN; A4; (ACGIH 2003).
MAK: 50 ppm, 210 mg/m³; Sh (sensibilización cutánea);
Categoría de limitación de pico: I(2);
Riesgo para el embarazo: grupo C (DFG 2002).

VIAS DE EXPOSICION:
La sustancia se puede absorber por inhalación, a través de la piel y por ingestión.

RIESGO DE INHALACION:
Por evaporación de esta sustancia a 20°C se puede alcanzar bastante rápidamente una concentración nociva en el aire.

EFFECTOS DE EXPOSICION DE CORTA DURACION:
La sustancia irrita los ojos, la piel y el tracto respiratorio.

EFFECTOS DE EXPOSICION PROLONGADA O REPETIDA:
El contacto prolongado o repetido puede producir sensibilización de la piel. La sustancia puede afectar al sistema nervioso periférico.

PROPIEDADES FISICAS

Punto de ebullición: 100.5°C
Punto de fusión: -48°C
Densidad relativa (agua = 1): 0.94
Solubilidad en agua, g/100 ml a 20°C: 1.6
Presión de vapor, kPa a 20°C: 3.9
Densidad relativa de vapor (aire = 1): 3.5
Temperatura crítica: 294°C (no en la ficha)
Densidad relativa de la mezcla vapor/aire a 20°C (aire = 1): 1.09
Punto de inflamación: 10°C c.a.
Temperatura de autoignición: 421°C
Límites de explosividad, % en volumen en el aire: 1.7-12.5
Coeficiente de reparto octanol/agua como log Pow: 1.38

DATOS AMBIENTALES

La sustancia es nociva para los organismos acuáticos.

NOTAS

Generalmente la sustancia contiene hidroquinona, hiroquinona metil éster, t-butilfenol de dimetilo como inhibidores de la polimerización. Estabilizadores o inhibidores añadidos pueden influir sobre las propiedades toxicológicas de esta sustancia; consultar a un experto. NO llevar a casa la ropa de trabajo.

Ficha de emergencia de transporte (Transport Emergency Card): TEC (R)-30S1247
Código NFPA: H 2; F 3; R 2;

INFORMACION ADICIONAL

Los valores LEP pueden consultarse en línea en la siguiente dirección: http://www.mtas.es/insht/practice/vlas.htm

Última revisión IPCS: 2003
Traducción al español y actualización de valores límite y etiquetado: 2003
FISQ: 5-124

ICSC: 0300 **METACRILATO DE METILO (inhibido)**

NOTA LEGAL IMPORTANTE:	Esta ficha contiene la opinión colectiva del Comité Internacional de Expertos del IPCS y es independiente de requisitos legales. Su posible uso no es responsabilidad de la CE, el IPCS, sus representantes o el INSHT, autor de la versión española.

Fichas Internacionales de Seguridad Química

ACIDO METACRILICO

ICSC: 0917

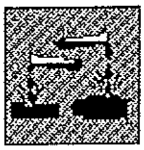
MINISTERIO DE TRABAJO Y ASUNTOS SOCIALES ESPAÑA

INSTITUTO NACIONAL DE SEGURIDAD E HIGIENE EN EL TRABAJO

ACIDO METACRILICO
Acido 2-metilpropenoico
Acido alfa-metilacrílico
$C_4H_6O_2/CH_2{=}C(CH_3)COOH$
Masa molecular: 86.09

Nº CAS 79-41-4
Nº RTECS OZ2975000
Nº ICSC 0917
Nº NU 2531 (inhibido)
Nº CE 607-088-00-5

TIPOS DE PELIGRO/ EXPOSICION	PELIGROS/ SINTOMAS AGUDOS	PREVENCION	PRIMEROS AUXILIOS/ LUCHA CONTRA INCENDIOS
INCENDIO	Combustible. Emite humos tóxicos en caso de incendio.	Evitar llama abierta.	AFFF, espuma resistente al alcohol, polvos, dióxido de carbono.
EXPLOSION	Por encima de 77°C: pueden formarse mezclas explosivas vapor/aire.	Por encima de 77°C: sistema cerrado, ventilación.	En caso de incendio: mantener fríos los bidones y demás instalaciones por pulverización con agua.
EXPOSICION			
• INHALACION	Sensación de quemazón, tos, dificultad respiratoria.	Ventilación (no si es polvo), extracción localizada o protección respiratoria.	Aire limpio, reposo, posición de semiincorporado y someter a atención médica.
• PIEL	Enrojecimiento, quemaduras cutáneas, dolor, ampollas.	Guantes protectores, traje de protección.	Quitar las ropas contaminadas, aclarar la piel con agua abundante o ducharse.
• OJOS	Enrojecimiento, dolor, pérdida de visión, quemaduras profundas graves.	Gafas ajustadas de seguridad, pantalla facial.	Enjuagar con agua abundante durante varios minutos (quitar las lentes de contacto si puede hacerse con facilidad), después consultar a un médico.
• INGESTION	Calambres abdominales, dolor abdominal, sensación de quemazón, debilidad.	No comer, beber ni fumar durante el trabajo.	Enjuagar la boca, reposo y someter a atención médica.

DERRAMAS Y FUGAS	ALMACENAMIENTO	ENVASADO Y ETIQUETADO
Recoger el líquido procedente de una fuga en recipientes herméticos de plástico o de cerámica, neutralizar cuidadosamente el residuo con solución acuosa de carbonato sódico o cal sodada, eliminar a continuación con agua abundante.	Separado de alimentos y piensos, bases fuertes. Enfriar (por debajo del punto de fusión, si es posible). Mantener en la oscuridad; mantener en una habitación bien ventilada. Almacenar sólamente si está estabilizado.	NO transportar con alimentos y piensos. símbolo C R: 34 S: (1/2-)15-26-45 Nota: D Clasificación de Peligros NU: 8 Grupo de Envasado NU: III CE: 

VEASE AL DORSO INFORMACION IMPORTANTE

ICSC: 0917

Preparada en el Contexto de Cooperación entre el IPCS y la Comisión de las Comunidades Eurpoeas © CCE, IPCS, 1994

ACIDO METACRILICO

ICSC: 0917

DATOS IMPORTANTES

ESTADO FISICO; ASPECTO
Líquido incoloro o cristales incoloros, de olor característico.

PELIGROS FISICOS

PELIGROS QUIMICOS
La sustancia se polimeriza fácilmente debido al calentamiento intenso o a la presencia de luz, oxígeno, agentes oxidantes, tales como peróxidos, o la presencia de trazas de ácido clorhídrico, con peligro de incendio o explosión. La sustancia se descompone al calentarla intensamente produciendo humos acres. La sustancia es moderadamente ácida. Ataca ciertos metales.

LIMITES DE EXPOSICION
TLV (como TWA): 20 ppm; 70 mg/m^3 (ACGIH 1990-1991).

VIAS DE EXPOSICION
La sustancia se puede absorber por inhalación del vapor o del aerosol.

RIESGO DE INHALACION
Por la evaporación de esta sustancia a 20°C se puede alcanzar bastante lentamente una concentración nociva en el aire.

EFECTOS DE EXPOSICION DE CORTA DURACION
La sustancia es corrosiva de la piel, los ojos y las membranas mucosas. El vapor de esta sustancia es corrosivo del tracto respiratorio. La inhalación del vapor puede originar edema pulmonar (véanse Notas).

EFECTOS DE EXPOSICION PROLONGADA O REPETIDA

PROPIEDADES FISICAS

Punto de ebullición: 159-163°C
Punto de fusión: 16°C
Densidad relativa (agua = 1): 1.02
Solubilidad en agua: moderada (8.9 g/100 ml a 25°C)
Presión de vapor, kPa a 25°C: alrededor de 0.13
Densidad relativa de vapor (aire = 1): 2.97
Densidad relativa de la mezcla vapor/aire a 20°C (aire = 1): 1.00
Punto de inflamación: 77°C (o.c.)°C
Coeficiente de reparto octanol/agua como log Pow: 0.93

DATOS AMBIENTALES

NOTAS

Normalmente contiene inhibidores para prevenir su propia polimerización. Los síntomas del edema pulmonar no se ponen de manifiesto a menudo hasta pasadas algunas horas y se agravan por el esfuerzo físico. Reposo y vigilancia médica son por ello imprescindibles. NO llevar a casa la ropa de trabajo. Tarjeta de emergencia de transporte (Transport Emergency Card): TEC (R)-195

Código NFPA: H 3; F 2; R 2;

INFORMACION ADICIONAL

FISQ: 1-022 ACIDO METACRILICO

ICSC: 0917 **ACIDO METACRILICO**

Fichas Internacionales de Seguridad Química

METACRILATO DE GLICIDILO — ICSC: 1679

[illegible]

Metacrilato de 2,3-epoxipropilo
Ácido metacrílico, ester 2,3-epoxipropílico
Glicidil alfa-metil acrilato
Metacrilato de 2,3-epoxi-1-propanol

CAS:	**106-91-2**	**$C_7H_{10}O_3$**
RTECS:	**OZ4375000**	**Masa molecular: 142,2**
NU:	**2810**	
CE Índice Anexo I:	**607-123-00-4**	
CE / EINECS:	**203-441-9**	

TIPO DE PELIGRO / EXPOSICIÓN	PELIGROS AGUDOS / SÍNTOMAS	PREVENCIÓN	PRIMEROS AUXILIOS / LUCHA CONTRA INCENDIOS
INCENDIO	Combustible.	Evitar las llamas.	Polvo. Dióxido de carbono. Espuma.
EXPLOSIÓN	Por encima de 61 °C pueden formarse mezclas explosivas vapor/aire.	Por encima de 61 °C, sistema cerrado, ventilación.	En caso de incendio: mantener fríos los bidones y demás instalaciones rociando con agua.
EXPOSICIÓN		**¡EVITAR TODO CONTACTO!**	
Inhalación	Tos. Dolor de garganta. Dificultad respiratoria.	Ventilación, extracción localizada o protección respiratoria.	Aire limpio, reposo. Posición de semiincorporado. Proporcionar asistencia médica.
Piel	Enrojecimiento. Dolor. Quemaduras cutáneas.	Guantes protectores. Traje de protección.	Quitar las ropas contaminadas. Aclarar con agua abundante o ducharse. Proporcionar asistencia médica.
Ojos	Enrojecimiento. Dolor. Quemaduras.	Pantalla facial o protección ocular combinada con protección respiratoria.	Enjuagar con agua abundante durante varios minutos (quitar las lentes de contacto si puede hacerse con facilidad), después proporcionar asistencia médica.
Ingestión	Dolor de garganta. Sensación de quemazón en la garganta y el pecho. Dolor abdominal.	No comer, ni beber, ni fumar durante el trabajo.	Enjuagar la boca. NO provocar el vómito. Dar a beber uno o dos vasos de agua. Proporcionar asistencia médica.

DERRAMES Y FUGAS

Protección personal complementaria: filtro respiratorio para vapores orgánicos y gases. Recoger el líquido procedente de la fuga en recipientes tapados. NO permitir que este producto químico se incorpore al ambiente.

ENVASADO Y ETIQUETADO

Clasificación UE
Símbolo: Xn
R: 20/21/22-36/38-43; S: (2-)26-28
Nota: D
Clasificación NU
Clasificación de Peligros NU: 6.1; Grupo de Envasado NU: III
Clasificación GHS
Peligro
Líquidos y vapores inflamables. Nocivo en caso de ingestión. Tóxico en contacto con la piel. Provoca irritación cutánea. Provoca irritación ocular. Puede provocar una reacción cutánea alérgica. Tóxico para los organismos acuáticos.

RESPUESTA DE EMERGENCIA

Ficha de Emergencia de Transporte (Transport Emergency Card): TEC (R)-61GT1-III

ALMACENAMIENTO

Almacenar solamente si está estabilizado. Mantener en lugar fresco. Bien cerrado. Mantener en la oscuridad. Separado de oxidantes fuertes, bases fuertes, ácidos fuertes. Separado de alimentos y piensos. Almacenar en un área sin acceso a desagües o alcantarillas. Medidas para contener el efluente de extinción de incendios.

IPCS
International Programme on Chemical Safety

WHO ILO UNEP

MINISTERIO DE TRABAJO E INMIGRACIÓN
INSTITUTO NACIONAL DE SEGURIDAD E HIGIENE EN EL TRABAJO

Preparada en el Contexto de Cooperación entre el IPCS y la Comisión Europea © CE, IPCS, 2007

VÉASE INFORMACIÓN IMPORTANTE AL DORSO

METACRILATO DE GLICIDILO | ICSC: 1679

DATOS IMPORTANTES

ESTADO FÍSICO; ASPECTO:
Líquido incoloro, de olor característico.

PELIGROS QUÍMICOS:
La sustancia puede polimerizar debido al calentamiento intenso y bajo la influencia de la luz y de peróxidos y bases. Reacciona violentamente con ácidos fuertes, bases fuertes y oxidantes fuertes, originando peligro de incendio.

LÍMITES DE EXPOSICIÓN:
TLV no establecido.
MAK no establecido.

VÍAS DE EXPOSICIÓN:
La sustancia se puede absorber por inhalación y a través de la piel y por ingestión.

RIESGO DE INHALACIÓN:
Por evaporación de esta sustancia a 20°C se puede alcanzar muy rápidamente una concentración nociva en el aire.

EFECTOS DE EXPOSICIÓN DE CORTA DURACIÓN:
La sustancia irrita gravemente los ojos, la piel y el tracto respiratorio.

EFECTOS DE EXPOSICIÓN PROLONGADA O REPETIDA:
El contacto prolongado o repetido puede producir sensibilización de la piel.

PROPIEDADES FÍSICAS

Punto de ebullición: 189°C
Punto de fusión: <-10°C
Densidad relativa (agua = 1): 1,08
Solubilidad en agua, g/100 ml a 25°C: 5 (moderada)
Presión de vapor, kPa a 25°C: 0,42
Densidad relativa de vapor (aire = 1): 4,9
Punto de inflamación: 61°C c.c.
Coeficiente de reparto octanol/agua como log Pow: 0,96

DATOS AMBIENTALES

La sustancia es tóxica para los organismos acuáticos. Se aconseja firmemente impedir que el producto químico se incorpore al ambiente.

NOTAS

La adición de estabilizadores o inhibidores puede influir sobre las propiedades toxicológicas de esta sustancia; consultar a un experto.

INFORMACIÓN ADICIONAL

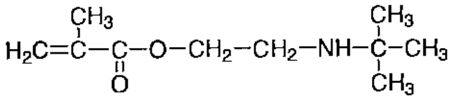
Nota legal Esta ficha contiene la opinión colectiva del Comité Internacional de Expertos del IPCS y es independiente de requisitos legales. Su posible uso no es responsabilidad de la CE, el IPCS, sus representantes o el INSHT, autor de la versión española.

Petrochemicals
Specialty Monomers

BASF
The Chemical Company

tert-Butylaminoethyl Methacrylate (TBAEMA)

tert-Butylaminoethyl Methacrylate is a monofunctional acrylate monomer with a polar tertiary amine functional group which offers water solubility and excellent adhesion properties.



$H_2C{=}C(CH_3){-}C({=}O){-}O{-}CH_2{-}CH_2{-}NH{-}C(CH_3)_2{-}CH_3$

CAS No.: 3775-90-4

EINECS-No.: 223-228-4

Molar mass: 185.3 kg/kmol

Molecular formula $C_{10}H_{19}O_2N$

Product specification

Assay (Gas chromatography)	min. 97.5 %
Water content (DIN 51777)	max. 0.2 %
Color on dispatch (APHA, DIN 53409)	max. 50
Standard stabilization (GC)	1100 ± 250 ppm MEHQ

The aforementioned data shall constitute the agreed contractual quality of the product at the time of passing of risk. The data are controlled at regular intervals as part of our quality assurance program. Neither these data nor the properties of product specimens shall imply any legally binding guarantee of certain properties or of fitness for a specific purpose.
No liability of ours can be derived therefrom.

Other properties

Appearance	colorless
Physical form	liquid
Density at 25 °C	0.914 g/cm^3
Boiling point	100–105 °C at 16 hPa
	82 °C at 13.3 hPa
Flash point	96 °C
Index of refraction, 20 °C	1.4420
Functionality, theoretical	2
T_g, homopolymer	33 °C

Labelling according to local Directives
see MSDS

Applications

tert-Butylaminoethyl Methacrylate forms homopolymers and copolymers. Copolymers of *tert*-Butylaminoethyl Methacrylate can be prepared with acrylic acid and its salts, amides and esters, and with methacrylates, acrylonitrile, maleic acid esters, vinyl acetate, vinyl chloride, vinylidene chloride, styrene, butadiene, unsaturated polyesters and drying oils, etc.

tert-Butylaminoethyl Methacrylate is also a very useful feedstock for chemical syntheses, because it readily undergoes addition reactions with a wide variety of organic and inorganic compounds.

The excellent adhesion properties of *tert*-Butylaminoethyl Methacrylate make it ideal for use in anticorrosive and automotive coatings.

tert-Butylaminoethyl Methacrylate is also used in industrial and consumer adhesives and coatings, and oil additives.

tert-Butylaminoethyl Methacrylate is a very effective dye additive in textiles.

tert-Butylaminoethyl Methacrylate is also used in photopolymer plates, photoresists, paints, rubber modifiers, dental composites and cosmetics.

tert-Butylaminoethyl Methacrylate is incorporated in flocculants or coagulants for water treatment, and is an excellent stabilizer or surface active demulsifier in oil in water separations and in liquid dispersion polymers.

tert-Butylaminoethyl Methacrylate has FDA clearance for use as basic components of single and repeat use food contact surfaces. Refer to US Code Federal Regulations Title 21: 21CFR 117.1010.

Features & Benefits

tert-Butylaminoethyl Methacrylate is a monofunctional acrylate monomer with dual methacrylic and amine reactivity. It offers excellent adhesion to metallic and plastic substrates, and low shrinkage properties.

The amine group in *tert*-Butylaminoethyl Methacrylate confers water solubility by quaternization to ammonium salts used for preparation of cationic polymers.

tert-Butylaminoethyl Methacrylate can be used to impart the following properties to polymers:

- Adhesion
- Water solubility
- Low shrinkage
- Surface active demulsifier
- Charge introduction
- Hydrophobicity

Storage & Handling

In order to prevent polymerization, *tert*-Butylaminoethyl Methacrylate has been shipped with stabilizer included. Contrary to other acrylates the presence of oxygen is not required for the stabilizer fo function effectively. Storing the product under inert atmosphere avoids coloration of the product. The storage temperature must not exceed 25 °C. Under these conditions, a storage stability of twelve months can be expected. In order to minimize the likelihood of overstorage, the storage procedure should strictly follow the "first-in-first-out" principle.

Storage tanks and pipes should be made of stainless steel or aluminium. Storage tanks, pumps and pipes should be earthed.

Safety

A Material Safety Data Sheet has been compiled for *tert*-Butylaminoethyl Methacrylate that contains up-to-date information on all questions relevant to safety.

Note

December 2011

Fichas Internacionales de Seguridad Química

PERSULFATO DE AMONIO

ICSC: 0632

MINISTERIO DE TRABAJO Y ASUNTOS SOCIALES ESPAÑA

INSTITUTO NACIONAL DE SEGURIDAD E HIGIENE EN EL TRABAJO

Peroxodisulfato de diamonio
Persulfato de diamonio

$H_8N_2O_8S_2$ / $(NH_4)_2S_2O_8$
Masa molecular: 228.2

Nº CAS 7727-54-0
Nº RTECS SE0350000
Nº ICSC 0632
Nº NU 1444
Nº CE 016-060-00-6

TIPOS DE PELIGRO/ EXPOSICION	PELIGROS/ SINTOMAS AGUDOS	PREVENCION	PRIMEROS AUXILIOS/ LUCHA CONTRA INCENDIOS
INCENDIO	No combustible pero facilita la combustión de otras sustancias. En caso de incendio se desprenden humos (o gases) tóxicos e irritantes.	NO poner en contacto con combustibles.	En caso de incendio en el entorno: están permitidos todos los agentes extintores.
EXPLOSION	Riesgo de incendio y explosión en contacto con sustancias combustibles y agentes reductores.		En caso de incendio: mantener fríos los bidones y demás instalaciones rociando con agua.
EXPOSICION		¡EVITAR LA DISPERSION DEL POLVO! ¡HIGIENE ESTRICTA!	
• INHALACION	Tos. Dificultad respiratoria. Dolor de garganta. Sibilancia.	Extracción localizada o protección respiratoria.	Aire limpio, reposo. Respiración artificial si estuviera indicada. Proporcionar asistencia médica.
• PIEL	Enrojecimiento. Sensación de quemazón. Dolor.	Guantes protectores.	Aclarar con agua abundante, después quitar la ropa contaminada y aclarar de nuevo.
• OJOS	Enrojecimiento. Dolor.	Gafas ajustadas de seguridad, o protección ocular combinada con la protección respiratoria si se trata de polvo.	Enjuagar con agua abundante durante varios minutos (quitar las lentes de contacto si puede hacerse con facilidad), después proporcionar asistencia médica.
• INGESTION	Diarrea. Náuseas. Dolor de garganta. Vómitos.	No comer, ni beber, ni fumar durante el trabajo. Lavarse las manos antes de comer.	Dar a beber agua abundante. Proporcionar asistencia médica.

DERRAMES Y FUGAS	ALMACENAMIENTO	ENVASADO Y ETIQUETADO

Barrer la sustancia derramada e introducirla en un recipiente. Eliminarlo a continuación con agua abundante. Recoger cuidadosamente el residuo, NO absorber en serrín u otros absorbentes combustibles. NO permitir que este producto químico se incorpore al ambiente. (Protección personal adicional: respirador de filtro P2 contra partículas nocivas).	Separado de sustancias combustibles y reductoras, metales en forma de polvo y bases fuertes. Mantener en lugar seco. Bien cerrado.	NU (transporte): Clasificación de Peligros NU: 5.1 Grupo de Envasado NU: III CE: símbolo O símbolo Xn R: 8-22-36/37/38-42/43 S: 2-22-24-26-37

VEASE AL DORSO INFORMACION IMPORTANTE

ICSC: 0632 Preparada en el Contexto de Cooperación entre el IPCS y la Comisión Europea © CE, IPCS, 2003

Fichas Internacionales de Seguridad Química

PERSULFATO DE AMONIO

ICSC: 0632

D A T O S I M P O R T A N T E S

ESTADO FISICO; ASPECTO
Cristales incoloros o polvo blanco.

PELIGROS QUIMICOS
La sustancia se descompone al calentarla intensamente, produciendo humos tóxicos y corrosivos, incluyendo vapores amoniacales, óxidos de nitrógeno y óxidos de azufre. La sustancia es un oxidante fuerte y reacciona con materiales combustibles y reductores. En disolución, reacciona violentamente con hierro, aluminio en polvo y sales de plata. La disolución en agua es moderadamente ácida. Reacciona violentamente con hierro, aluminio en polvo y sales de plata.

LIMITES DE EXPOSICION
TLV: 0.1 mg/m^3 (como TWA) (ACGIH 2003)
MAK: Sah (sensibilizante respiratorio y cutáneo).(DFG 2003)

VIAS DE EXPOSICION
La sustancia se puede absorber por inhalación del aerosol y por ingestión.

RIESGO DE INHALACION
La evaporación a 20°C es despreciable; sin embargo, se puede alcanzar rápidamente una concentración nociva de partículas pulverizadas o dispersadas en el aire, especialmente en forma de polvo.

EFECTOS DE EXPOSICION DE CORTA DURACION
La sustancia irrita los ojos, la piel y el tracto respiratorio. La inhalación del polvo puede originar reacciones asmáticas (véanse Notas).

EFECTOS DE EXPOSICION PROLONGADA O REPETIDA
El contacto prolongado o repetido con la piel puede producir dermatitis. El contacto prolongado o repetido puede producir sensibilización de la piel. Puede producir una reacción alérgica general, como urticaria o schock. La exposición a inhalación prolongada o repetida puede originar asma.

PROPIEDADES FISICAS

Se descompone por debajo del punto de fusión a 120°C
Densidad: 1.9 g/cm^3
Solubilidad en agua, g/100 ml a 20°C: 58.2

DATOS AMBIENTALES

La sustancia es nociva para los organismos acuáticos.

N O T A S

Los síntomas de asma no se ponen de manifiesto, a menudo, hasta pasadas algunas horas y se agravan por el esfuerzo físico. Reposo y vigilancia médica son, por ello, imprescindibles. Ninguna persona que haya mostrado síntomas de asma debe entrar nunca en contacto con esta sustancia. NO llevar a casa la ropa de trabajo. Enjuagar la ropa contaminada con agua abundante (peligro de incendio).

Ficha de emergencia de transporte (Transport Emergency Card): TEC (R)-51G02-III
Código NFPA: H 2; F 1; R 1;

INFORMACION ADICIONAL

os valores LEP pueden consultarse en línea en la siguiente irección: **http://www.mtas.es/insht/practice/vlas.htm**	Última revisión IPCS: 2001 Traducción al español y actualización de valores límite y etiquetado: 2003 FISQ: 6-157

ICSC: 0632

PERSULFATO DE AMONIO

NOTA LEGAL IMPORTANTE:	Esta ficha contiene la opinión colectiva del Comité Internacional de Expertos del IPCS y es independiente de requisitos legales. Su posible uso no es responsabilidad de la CE, el IPCS, sus representantes o el INSHT, autor de la versión española.

15A872 *ter*-**Butilo Hidroperóxido solución 3M** en isooctano PS

1. Identificación de la sustancia/preparado y de la sociedad o empresa

1.1 Identificación de la sustancia o del preparado

Denominación:
ter-Butilo Hidroperóxido solución 3M en isooctano

1.2 Uso de la sustancia o preparado:

Para usos de laboratorio, análisis, investigación y química fina.

1.3 Identificación de la sociedad o empresa:

PANREAC QUIMICA, S.A.U.
C/Garraf, 2
Polígono Pla de la Bruguera
E-08211 Castellar del Vallès
(Barcelona) España
Tel. (+34) 937 489 400
e-mail: product.safety@panreac.com
Urgencias:
Número único de teléfono para llamadas de urgencia: 112 (UE)
Tel.:(+34) 937 489 499

2. Identificación de los peligros

Peligro de fuego en contacto con materias combustibles. Inflamable. Provoca quemaduras. Nocivo por inhalación, por ingestión y en contacto con la piel. Riesgo de explosión al calentarlo en ambiente confinado. Nocivo para los organismos acuáticos, puede provocar a largo plazo efectos negativos en el medio ambiente acuático.

3. Composición/Información de los componentes

ter-Butilo Hidroperóxido solución acuosa X70% dil. 30 % - 40 %
CAS [75-91-2] Fórmula: $C_4H_{10}O_2$ M.=90,12
Número CE (EINECS): 200-915-7
R: 7-21/22-23-34
Isooctano > 50%
CAS [540-84-1] Fórmula: C_8H_{18} M.=114,23
Número CE (EINECS): 208-759-1
Número de índice CE: 601-009-00-8
R: 11-38-65-67-50/53

4. Primeros auxilios

4.1 Indicaciones generales:

En caso de pérdida del conocimiento nunca dar a beber ni provocar el vómito.

4.2 Inhalación:

Trasladar a la persona al aire libre. En caso de asfixia proceder inmediatamente a la respiración artificial. Pedir inmediatamente atención médica.

4.3 **Contacto con la piel:**

Lavar abundantemente con agua. Quitarse las ropas contaminadas. Extraer el producto con un algodón impregnado en polietilenglicol 400. Pedir inmediatamente atención médica.

4.4 **Ojos:**

Lavar con agua abundante (mínimo durante 15 minutos), manteniendo los párpados abiertos. Pedir inmediatamente atención médica.

4.5 **Ingestión:**

Beber agua abundante. Evitar el vómito (existe riesgo de perforación). Pedir inmediatamente atención médica.

5. Medidas de lucha contra incendio

5.1 **Medios de extinción adecuados:**

Agua. Espuma. Polvo seco. Dióxido de carbono (CO_2).

5.2 **Medios de extinción que NO deben utilizarse:**

5.3 **Riesgos especiales:**

Favorece la formación de incendios. Mantener alejado de sustancias combustibles. Inflamable. Mantener alejado de fuentes de ignición. En caso de incendio pueden formarse vapores tóxicos. No permitir el paso del agua de extinción a acuíferos superficiales o subterráneos. Evitar la formación de cargas electroestáticas.

5.4 **Equipos de protección:**

Ropa y calzado adecuados. Equipo de respiración autónomo.

6. Medidas a tomar en caso de vertido accidental

6.1 **Precauciones individuales:**

No inhalar los vapores. Procurar una ventilación apropiada. Evitar el contacto con la piel, los ojos y la ropa. Evacuar a toda persona no indispensable.

6.2 **Precauciones para la protección del medio ambiente:**

No permitir el paso al sistema de desagües. Evitar la contaminación del suelo, aguas y desagües.

6.3 **Métodos de recogida/limpieza:**

Recoger con materiales absorbentes (Absorbente General Panreac, Kieselguhr, etc.) o en su defecto arena o tierra secas y depositar en contenedores para residuos para su posterior eliminación de acuerdo con las normativas vigentes. Limpiar los restos con agua abundante.

7. Manipulación y almacenamiento

7.1 **Manipulación:**

Asegurar una buena ventilación y renovación de aire en el local. Manipular bajo campana extractora. Evitar la formación de cargas electrostáticas.

7.2 **Almacenamiento:**

Recipientes bien cerrados. En lugar fresco, seco y bien ventilado. Mantener alejado de sustancias inflamables, fuentes de ignición y calor. Temperatura ambiente.

8. Controles de exposición/protección personal

8.1 Medidas técnicas de protección:

Asegurar una buena ventilación y renovación de aire del local.

8.2 Control límite de exposición:

8.3 Protección respiratoria:

En caso de formarse vapores/aerosoles, usar equipo respiratorio adecuado.

8.4 Protección de las manos:

Usar guantes apropiados

8.5 Protección de los ojos:

Usar gafas apropiadas.

8.6 Medidas de higiene particulares:

Quitarse las ropas contaminadas. Usar ropa de trabajo adecuada. Lavarse manos y cara antes de las pausas y al finalizar el trabajo. No comer, beber ni fumar en el lugar de trabajo. No inhalar la sustancia.

8.7 Controles de la exposición del medio ambiente:

Cumplir con la legislación local vigente sobre protección del medio ambiente.

El proveedor de los medios de protección debe especificar el tipo de protección que debe usarse para la manipulación del producto, indicando el tipo de material y, cuando proceda, el tiempo de penetración de dicho material, en relación con la cantidad y la duración de la exposición.

9. Propiedades físicas y químicas

Aspecto:

Líquido transparente e incoloro.

Olor:

Picante

Punto de inflamación : 4°C
Densidad (20/4): 0,746
Solubilidad: Inmiscible con agua.

10. Estabilidad y reactividad

10.1 Condiciones que deben evitarse:

Temperaturas elevadas.

10.2 Materias que deben evitarse:

Agentes reductores. Acidos fuertes. Bases fuertes. Metales en polvo.

10.3 Productos de descomposición peligrosos:

Monóxido de carbono. Dióxido de carbono.

10.4 Información complementaria:

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11. Información toxicológica

11.1 Toxicidad aguda:

11.2 Efectos peligrosos para la salud:

Atendiendo a los componentes del preparado, las características peligrosas probables son las siguientes:

En contacto con la piel: Provoca quemaduras. Nocivo para la salud. Riesgo de sensibilización.

Por contacto ocular: Provoca quemaduras. Riesgo de ceguera (lesión irreversible del nervio óptico).

Por ingestión: Nocivo para la salud. Quemaduras en el aparato digestivo.

Por inhalación: Nocivo para la salud. Puede provocar espasmos, tos, laringitis, dolores de cabeza, náuseas, vómitos, inflamación y edemas en laringe, bronquios y pulmones.

No se descartan otras características peligrosas. Observar las precauciones habituales en el manejo de productos químicos.

12. Información Ecológica

12.1 Movilidad :

12.2 Ecotoxicidad :

12.2.1 - Test EC_{50} (mg/l) :

12.2.2 - Medio receptor :
Riesgo para el medio acuático = ----
Riesgo para el medio terrestre = ----
12.2.3 - Observaciones :
Datos ecotóxicos no disponibles.

12.3 Degradabilidad :

12.3.1 - Test :-----
12.3.2 - Clasificación sobre degradación biótica :
DBO_5/DQO Biodegradabilidad = -----
12.3.3 - Degradación abiótica según pH : ------
12.3.4 - Observaciones :
Datos no disponibles.

12.4 Acumulación :

12.4.1 - Test :

12.4.2 - Bioacumulación :
Riesgo = -----
12.4.3 - Observaciones :
Datos no disponibles.

12.5 Otros posibles efectos sobre el medio natural :

No permitir su incorporación al suelo ni a acuíferos. No incorporar al sumidero de aguas residuales.

13. Consideraciones sobre la eliminación

13.1 Sustancia o preparado:

En la Unión Europea no están establecidas pautas homogéneas para la eliminación de residuos químicos, los cuales tienen carácter de residuos especiales, quedando sujetos su tratamiento y eliminación a los reglamentos internos de cada país. Por tanto, en cada caso, procede contactar con la autoridad competente, o bien con los gestores legalmente autorizados para la

eliminación de residuos.
2001/573/CE: Decisión del Consejo, de 23 de julio de 2001, por la que se modifica la Decisión 2000/532/CE de la Comisión en lo relativo a la lista de residuos.
Directiva 91/156/CEE del Consejo de 18 de marzo de 1991 por la que se modifica la Directiva 75/442/CEE relativa a los residuos.
En España: Ley 10/1998, de 21 de abril, de Residuos. Publicada en BOE 22/04/98.
ORDEN MAM/304/2002, de 8 de febrero, por la que se publican las operaciones de valorización y eliminación de residuos y la lista europea de residuos. Publicada en BOE 19/02/02.

13.2 Envases contaminados:

Los envases y embalajes contaminados de sustancias o preparados peligrosos, tendrán el mismo tratamiento que los propios productos contenidos.
Directiva 94/62/CE del Parlamento Europeo y del Consejo, de 20 de diciembre de 1994, relativa a los envases y residuos de envases.
En España: Ley 11/1997, de 24 de abril, de Envases y Residuos de Envases. Publicada en BOE 25/04/97.
Real Decreto 782/1998, de 30 de abril, por el que se aprueba el Reglamento para el desarrollo y ejecución de la Ley 11/1997, de 24 de abril, de Envases y Residuos de Envases. Publicado en BOE 01/05/98.

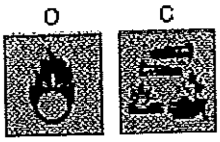
14. Información relativa al transporte

Terrestre (ADR):
Denominación técnica: PERÓXIDO ORGÁNICO DE TIPO D, LÍQUIDO (ter-Butilo Hidroperóxido solución 3M en isooctano)
ONU 3105 Clase: 5.2 Grupo de embalaje: - (D)
Marítimo (IMDG):
Denominación técnica: PERÓXIDO ORGÁNICO DE TIPO D, LÍQUIDO (ter-Butilo Hidroperóxido solución 3M en isooctano)
ONU 3105 Clase: 5.2 Grupo de embalaje: -
Aéreo (ICAO-IATA):
Denominación técnica: Peróxido orgánico de tipo D, líquido (ter-Butilo Hidroperóxido solución 3M en isooctano)
ONU 3105 Clase: 5.2 Grupo de embalaje: -
Instrucciones de embalaje: CAO 502 PAX 500

15. Información reglamentaria

15.1 Etiquetado según REACH

Símbolos:



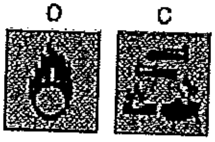
Indicaciones de peligro: Comburente Corrosivo
Frases R: 8-10-34-20/21/22-44-52/53 Peligro de fuego en contacto con materias combustibles. Inflamable. Provoca quemaduras. Nocivo por inhalación, por ingestión y en contacto con la piel. Riesgo de explosión al calentarlo en ambiente confinado. Nocivo para los organismos acuáticos, puede provocar a largo plazo efectos negativos en el medio ambiente acuático.
Frases S: 3/7/9-16-26-33-36/37/39-45 Consérvese el recipiente en lugar fresco y bien ventilado y manténgase bien cerrado. Conservar alejado de toda

llama o fuente de chispas - No fumar. En caso de contacto con los ojos, lávense inmediata y abundantemente con agua y acúdase a un médico. Evítese la acumulación de cargas electroestáticas. Usense indumentaria y guantes adecuados y protección para los ojos-la cara. En caso de accidente o malestar, acuda inmediatamente al médico (si es posible, muéstrele la etiqueta).

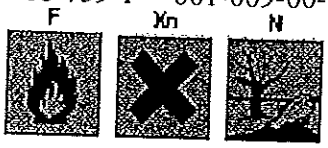
16. Otras informaciones

Respecto a la revisión anterior, se han producido cambios en los apartados: 10, 11, 14, 15.
Información de los componentes:

ter-Butilo Hidroperóxido solución acuosa X70% dil. 30 % - 40 %
CAS [75-91-2] $C_4H_{10}O_2$ M.=90,12
200-915-7



R: 7-21/22-23-34
Puede provocar incendios. Nocivo en contacto con la piel y por ingestión. Tóxico por inhalación. Provoca quemaduras.
Isooctano > 50%
CAS [540-84-1] C_8H_{18} M.=114,23
208-759-1 601-009-00-8



R: 11-38-65-67-50/53
Fácilmente inflamable. Irrita la piel. Nocivo: si se ingiere puede causar daño pulmonar. La inhalación de vapores puede provocar somnolencia y vértigo. Muy tóxico para los organismos acuáticos, puede provocar a largo plazo efectos negativos en el medio ambiente acuático.

Número y fecha de la revisión:2 14.05.09
Los datos consignados en la presente Ficha de Datos de Seguridad, están basados en nuestros conocimientos actuales, teniendo como único objeto informar sobre aspectos de seguridad y no garantizándose las propiedades y características en ella indicadas.

MERCK

Ficha de Datos de Seguridad

BRUGGOLITE

Conforme al Reglamento (CE) nro. 1907/2006 (REACH)

Fecha de emisión:	12.02.2008
Reemplaza la emisión del	12.02.2004

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1. Identificación de la sustancia o del preparado y de la sociedad o empresa

Identificación de la sustancia o del preparado

Artículo número:	106528
Denominación:	Sodio disulfito (sodio metabisulfito) para análisis ACS,Reag. Ph Eur

Utilización de la sustancia/preparación

Análisis químico
Producción química
Producción y análisis farmacéuticos

Denominación de la empresa

Empresa:	Merck KGaA * 64271 Darmstadt * Alemania * Tel: +49 6151 72-0
Teléfono de urgencias:	Instituto Nacional de Toxicología * Madrid * Tel: 91 562 04 20
Responsable de informaciones:	EHSQ/EHS-PI * e-mail: prodsafe@merck.de

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2. Identificación de peligros

Nocivo por ingestión. En contacto con ácidos libera gases tóxicos. Riesgo de lesiones oculares graves.

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3. Composición/información sobre los componentes

Sinónimos:

Sodio meta-bisulfito, Sodio metabisulfito, Sodio pirosulfito

Nr.-CAS:	7681-57-4	Número de índice CE:	016-063-00-2
PM:	190.10 g/mol	Número CE:	231-673-0
Fórmula molecular: (según Hill)	$Na_2O_5S_2$		

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4. Primeros auxilios

Tras inhalación: aire fresco.
Tras contacto con la piel: aclarar con abundante agua. Eliminar ropa contaminada.
Tras contacto con los ojos: Aclarar con abundante agua, manteniendo abiertos los párpados. Llamar inmediatamente al oftalmólogo. Tras ingestión: hacer beber agua inmediatamente (máximo 2 vasos). Llamar inmediatamente al médico.

5. Medidas de lucha contra incendios

Medios de extinción adecuados:
Adaptar a los materiales en el contorno.

Riesgos especiales:
Incombustible. En caso de incendio posible formación de gases de combustión o vapores peligrosos.
En caso de incendio pueden producirse: sulfóxidos.

Equipo de protección especial para el personal de lucha contra incendios:
Permanencia en el área de riesgo sólo con sistemas de respiracion artificiales e independientes del ambiente. Protección de la piel mediante observación de una distancia de seguridad y uso de ropa protectora adecuada .

Referencias adicionales:
Precipitar los vapores emergentes con agua. Evitar la penetración del agua de extinción en acuíferos superficiales o subterraneos.

6. Medidas a tomar en caso de vertido accidental

Medidas de precaución relativas a las personas:
Evitar la inhalación de polvo. Evitar el contacto con la sustancia.

Medidas de protección del medio ambiente:
No lanzar por el sumidero.

Procedimientos de recogida/limpieza:
Recoger en seco y proceder a la eliminación de residuos. Aclarar. Evitar la formación de polvo.

7. Manipulación y almacenamiento

Manipulación:

Sin otras exigencias.

Almacenamiento:

Bien cerrado. Seco. Temperatura de almacenamiento: sin limitaciones.

8. Controles de exposición/protección personal

Protección personal:

Los tipos de auxiliares para protección del cuerpo deben elegirse específicamente según el puesto de trabajo en función de la concentración y cantidad de la sustancia peligrosa. Debería aclararse con el suministrador la estabilidad de los medios protectores frente a los productos químicos.

Protección respiratoria:	necesaria en presencia de polvo.
Protección de los ojos:	precisa

Protección de las manos:

Para contacto pleno:

Guantes:	Caucho nitrilo
Espesor:	0.11 mm
Tiempo de penetración:	> 480 Min.

En caso de salpicaduras:

Guantes:	Caucho nitrilo
Espesor:	0.11 mm
Tiempo de penetración:	> 480 Min.

Los guantes de protección indicados deben cumplir con las especificaciones de la Directiva 89/686/EEC y con su norma resultante EN374, por ejemplo KCL 741 Dermatril® L (Sumerción), 741 Dermatril® L (Salpicaduras). Los tiempos de ruptura mencionados anteriormente han sido determinados con muestras de material de los tipos de guantes recomendados en mediciones de laboratorio de KCL según EN374.
Esta recomendación solo es válida para el producto mencionado en la ficha de datos de seguridad, suministrado por nosotros y para el fin indicado. Al disolver o mezclar en otras sustancias y cuando las condiciones difieran de las indicadas en EN374, debe dirigirse al suministrador de guantes con distintivo CE (por ejem. KCL GmbH, D-36124 Eichenzell, Internet: www.kcl.de)

Medidas de higiene particulares:
Sustituir inmediatamente la ropa contaminada. Protección preventiva de la piel. Lavar cara y manos al término del trabajo.

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9. Propiedades físicas y químicas

Estado físico:		polvo			
Color:		incoloro			
Olor:		débilmente picante			
Valor pH a 50 g/l H_2O		(20 °C)	3.5-5		
Punto de fusión			~ 150	°C	(descomposición)
Punto de ebullición			no aplicable		
Temperatura de ignición			no aplicable		
Punto de inflamación			no aplicable		
Límite de explosión	bajo		no aplicable		
	alto		no aplicable		
Densidad		(20 °C)	2.36	g/cm^3	
Densidad de amontonamiento			1000-1200	kg/m^3	
Solubilidad en Agua		(20 °C)	~ 650	g/l	
Descomposición térmica			> 150	°C	

10. Estabilidad y reactividad

Condiciones a evitar

Fuerte calefacción (descomposición).

Materias a evitar

Desprendimiento de gases o vapores peligrosos con: ácidos. Liberación de: dióxido de azufre.
Reacción exotérmica con: oxidante, nitritos, nitratos, sulfuros.

Productos de descomposición peligrosos
en caso de incendio: véase capítulo 5.

11. Información toxicológica

Toxicidad aguda

LD_{50} (dérmica, rata): >2000 mg/kg (RTECS).
LD_{50} (oral, rata): 1540 mg/kg (OECD 401).

Síntomas específicos en ensayos sobre animales:
Ensayo de irritación ocular (conejo): Irritaciones (OECD 405).
Ensayo de irritación cutánea (conejo): Sin irritación (OECD 404).

Toxicidad subaguda a crónica

Sensibilización:
Test de sensibilización (cobaya): Sin efecto sensibilizante. (Ficha de datos de Seguridad externa)

No cancerígeno en ensayos con animales. (IUCLID)
No mutágeno en experimentos con animales. (Ficha de datos de Seguridad externa)
Mutagenicidad (ensayo de células de mamífero): test micronucleus: negativo. (IUCLID)
Mutagenicidad bacteriana: test de Ames: negativo. (OECD 471)
No hay reducción de la capacidad reproductora en experimentos con animales. (IUCLID)
No teratógeno en experimentos con animales. (Ficha de datos de Seguridad externa)

Informaciones adicionales sobre toxicidad

Tras inhalación: Irritación de las mucosas, tos y dificultad para respirar.
Tras contacto con la piel: leves irritaciones.
Tras contacto con los ojos: Riesgo de lesiones oculares graves.
Tras ingestión: Irritaciones de las mucosas en la boca, garganta, esófago y tracto estomago-intestinal.

Información complementaria

El producto debe manejarse con las precauciones apropiadas para los productos químicos.

12. Informaciones ecológicas

Biodegradabilidad:
Los métodos para determinación de la biodegradabilidad no son aplicables para sustancias inorgánicas.

Efectos ecotóxicos:
Efectos biológicos:
Toxicidad para los peces: Onchorhynchus mykiss LC$_{50}$: 150-220 mg/l /96 h (DIN 38412 parte 15).
Toxicidad de dafnia: Daphnia magna CE$_{50}$: 89 mg/l /48 h (OCDE 202).
Tóxicidad para las algas: Desmodesmus subspicatus CI$_{50}$: 48 mg/l /72 h (OCDE 201).
Tóxicidad de bacterias: Ps. putida CE$_{50}$: 56 mg/l /17 h (IUCLID).
No deben esperarse interferencias en depuradoras biológicas si se maneja adecuadamente el producto.

Otras observaciones ecológicas:
DQO: 0.165 g/g (calculado) (Ficha de datos de Seguridad externa).

¡No incorporar a suelos ni acuíferos!

13. Consideraciones relativas a la eliminación

Producto:

Los productos químicos han de eliminarse siguiendo las normativas nacionales. Bajo www.retrologistik.de encontrará indicaciones sobre paises, indicaciones especificas de productos así como contactos.

Embalaje:

Los envases de productos Merck han de eliminarse siguiendo las normativas nacionales. Bajo www.retrologistik.de encontrará indicaciones especiales para las peculiaridades nacionales así como contactos

14. Información relativa al transporte

No sometido a las normas de transporte.

15. Información reglamentaria

Etiquetado según Directivas de la CEE

Pictograma:	Xn	Nocivo
Frases R:	22-31-41	Nocivo por ingestión. En contacto con ácidos libera gases tóxicos. Riesgo de lesiones oculares graves.
Frases S:	26-39-46	En caso de contacto con los ojos, lávense inmediata y abundantemente con agua y acúdase a un médico. Úsese protección para los ojos/la cara. En caso de ingestión, acúdase inmediatamente al médico y muéstresele la etiqueta o el envase.
Número CE:	231-673-0	Etiquetado CE

Etiquetado reducido(1999/45/CE,art.10,4)

Pictograma:	Xn	Nocivo
Frases R:	22-41	Nocivo por ingestión. Riesgo de lesiones oculares graves.
Frases S:	26-39	En caso de contacto con los ojos, lávense inmediata y abundantemente con agua y acúdase a un médico. Úsese protección para los ojos/la cara.

16. Otras informaciones

Razón de revisión

Cambio en el capítulo de primeros auxilios.
Cambio/completado en el capítulo 10.
Cambio en el capítulo de toxicología.
Cambio en el capítulo de ecología

Cambio de la denominación del artículo.

Revisión general.

Representante regional:

VWR International S.L. * Apartado 48 * E-08100 Mollet del Valles * Tel.: +34 (0) 93 5655 500 * Fax: +34 (0) 93 5440 000
Merck Farma y Química, S.A. * Apartado 47 * E-08100 Mollet del Vallès * Tel.: +34 (0) 93 5655 500 * Fax: +34 (0) 93 5440 000* e-mail: lifescience merck.es

Los datos suministrados en ésta ficha de seguridad se basan a nuestro actual conocimiento. Describen tan sólo las medidas de seguridad en el manejo de éste producto y no representan una garantía sobre las propiedades descritas del mismo.

Ficha de Datos de Seguridad
Según Reglamento (CE) 1907/2006

Panreac

721128 Amoníaco 20% (en NH_3) (TMA) HIPERPUR

1. Identificación de la sustancia/preparado y de la sociedad o empresa

1.1 Identificación de la sustancia o del preparado

Denominación:
Amoníaco 20% (en NH_3)

1.2 Uso de la sustancia o preparado:

Para usos de laboratorio, análisis, investigación y química fina.

1.3 Identificación de la sociedad o empresa:

PANREAC QUIMICA, S.A.U.
C/Garraf, 2
Polígono Pla de la Bruguera
E-08211 Castellar del Vallès
(Barcelona) España
Tel. (+34) 937 489 400
e-mail: product.safety@panreac.com
Urgencias:
Número único de teléfono para llamadas de urgencia: 112 (UE)
Tel.:(+34) 937 489 499

2. Identificación de los peligros

Provoca quemaduras.

3. Composición/Información de los componentes

Solución acuosa

Amoníaco 20% (en NH_3)
CAS [1336-21-6] Fórmula: NH_3
M.=17,03
Número CE (EINECS): 215-647-6
Número de índice CE: 007-001-01-2
R: 34

4. Primeros auxilios

4.1 **Indicaciones generales:**

En caso de pérdida del conocimiento nunca dar a beber ni provocar el vómito.

4.2 **Inhalación:**

Trasladar a la persona al aire libre. En caso de que persista el malestar, pedir atención médica.

4.3 **Contacto con la piel:**

Lavar abundantemente con agua. Quitarse las ropas contaminadas. En caso de irritación, pedir atención médica.

4.4 **Ojos:**

Lavar con agua abundante (mínimo durante 15 minutos), manteniendo los párpados abiertos. Pedir atención médica.

4.5 **Ingestión:**

Beber agua abundante. Evitar el vómito (existe riesgo de perforación). Pedir atención médica. No neutralizar.

5. Medidas de lucha contra incendio

5.1 **Medios de extinción adecuados:**

Los apropiados al entorno.

5.2 **Medios de extinción que NO deben utilizarse:**

5.3 **Riesgos especiales:**

En caso de incendio pueden formarse vapores de NH_3. Precipitar los vapores formados con agua. Refrigerar los recipientes con agua. Incombustible. No permitir el paso del agua de extinción a acuíferos superficiales o subterráneos.

5.4 **Equipos de protección:**

Ropa y calzado adecuados. Equipo de respiración autónomo.

6. Medidas a tomar en caso de vertido accidental

6.1 **Precauciones individuales:**

No inhalar los vapores.

6.2 **Precauciones para la protección del medio ambiente:**

6.3 **Métodos de recogida/limpieza:**

Recoger con materiales absorbentes (Absorbente General Panreac, Kieselguhr, etc.) o en su defecto arena o tierra secas y depositar en contenedores para residuos para su posterior eliminación de acuerdo con las normativas vigentes. Limpiar los restos con agua abundante. Neutralizar con acido sulfúrico diluido.

7. Manipulación y almacenamiento

7.1 Manipulación:

Sin indicaciones particulares.

7.2 Almacenamiento:

Recipientes bien cerrados. En local bien ventilado. Temperatura ambiente.

8. Controles de exposición/protección personal

8.1 Medidas técnicas de protección:

8.2 Control límite de exposición:

VLA-ED: 25 ppm ó 18 mg/m3
VLA-EC: 35 ppm ó 25 mg/m3

8.3 Protección respiratoria:

En caso de formarse vapores/aerosoles, usar equipo respiratorio adecuado. Filtro K. Filtro P_3.

8.4 Protección de las manos:

Usar guantes apropiados (neopreno, PVC, nitrilo, látex).

8.5 Protección de los ojos:

Usar gafas apropiadas.

8.6 Medidas de higiene particulares:

Quitarse las ropas contaminadas. Usar equipo de protección completo. Lavarse manos y cara antes de las pausas y al finalizar el trabajo.

8.7 Controles de la exposición del medio ambiente:

Cumplir con la legislación local vigente sobre protección del medio ambiente.

El proveedor de los medios de protección debe especificar el tipo de protección que debe usarse para la manipulación del producto, indicando el tipo de material y, cuando proceda, el tiempo de penetración de dicho material, en relación con la cantidad y la duración de la exposición.

9. Propiedades físicas y químicas

Aspecto:

Líquido transparente e incoloro.

Olor:

Característico.

Presión de vapor: (20°C) 500 hPa
Densidad (20/4): 0,92
Solubilidad: Miscible con agua

10. Estabilidad y reactividad

10.1 Condiciones que deben evitarse:

10.2 Materias que deben evitarse:

Soluciones alcalinas. Yodo. Acidos fuertes. Metales y sus aleaciones.

10.3 Productos de descomposición peligrosos:

10.4 Información complementaria:

Los gases / vapores pueden formar mezclas explosivas con el aire.

11. Información toxicológica

11.1 Toxicidad aguda:

DL_{50} oral rata: 350 mg/kg.

CL_{50} inh rata: 2000 ppm(V)/4h (anh.)

11.2 Efectos peligrosos para la salud:

Por inhalación de vapores: tos, bronquitis, edema pulmonar. En caso de formación de vapores, fuertemente irritante.
En contacto con la piel: irritaciones, quemaduras.
Por contacto ocular: quemaduras, ceguera (lesión irreversible del nervio óptico).
Por ingestión: Irritaciones en mucosas dolores de estómago, náuseas, vómitos, colapso, pérdida del conocimiento, dificultades respiratorias. Puede provocar perforación intestinal y de esófago.

12. Información Ecológica

12.1 Movilidad :

12.2 Ecotoxicidad :

12.2.1 - Test EC_{50} (mg/l) :

Peces (Salmo gairdneri) = EC_{10} 0,3 mg/l ; Clasificación : Extremadamente tóxico.

Crustáceos (Daphnia Magna) = 60 mg/l ; Clasificación : Extremadamente tóxico.

12.2.2 - Medio receptor :

Riesgo para el medio acuático = Medio

Riesgo para el medio terrestre = Bajo

12.2.3 - Observaciones :

Ecotoxicidad aguda en la zona de vertido. La ecotoxicidad se debe a la desviación del pH.

12.3 Degradabilidad :

12.3.1 - Test :-------

12.3.2 - Clasificación sobre degradación biótica :

DBO_5/DQO Biodegradabilidad = -----

12.3.3 - Degradación abiótica según pH : -------

12.3.4 - Observaciones :

12.4 Acumulación :

12.4.1 - Test :

12.4.2 - Bioacumulación :

Riesgo = -----

12.4.3 - Observaciones :

12.5 Otros posibles efectos sobre el medio natural :

No permitir su incorporación al suelo ni a acuíferos.

13. Consideraciones sobre la eliminación

13.1 Sustancia o preparado:

En la Unión Europea no están establecidas pautas homogéneas para la eliminación de residuos químicos, los cuales tienen carácter de residuos especiales, quedando sujetos su tratamiento y eliminación a los reglamentos internos de cada país. Por tanto, en cada caso, procede contactar con la autoridad competente, o bien con los gestores legalmente autorizados para la eliminación de residuos.

2001/573/CE: Decisión del Consejo, de 23 de julio de 2001, por la que se modifica la Decisión 2000/532/CE de la Comisión en lo relativo a la lista de residuos.

Directiva 91/156/CEE del Consejo de 18 de marzo de 1991 por la que se modifica la Directiva 75/442/CEE relativa a los residuos.

En España: Ley 10/1998, de 21 de abril, de Residuos. Publicada en BOE 22/04/98.

ORDEN MAM/304/2002, de 8 de febrero, por la que se publican las operaciones de valorización y eliminación de residuos y la lista europea de residuos. Publicada en BOE 19/02/02.

13.2 Envases contaminados:

Los envases y embalajes contaminados de sustancias o preparados peligrosos, tendrán el mismo tratamiento que los propios productos contenidos.

Directiva 94/62/CE del Parlamento Europeo y del Consejo, de 20 de diciembre de 1994, relativa a los envases y residuos de envases.

En España: Ley 11/1997, de 24 de abril, de Envases y Residuos de Envases. Publicada en BOE 25/04/97.

Real Decreto 782/1998, de 30 de abril, por el que se aprueba el Reglamento para el desarrollo y ejecución de la Ley 11/1997, de 24 de abril, de Envases y Residuos de Envases. Publicado en BOE 01/05/98.

14. Información relativa al transporte

Terrestre (ADR):

Denominación técnica: AMONIACO EN SOLUCIÓN acuosa con más del 10% pero no más del 35% de amoniaco

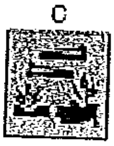
ONU 2672 Clase: 8 Grupo de embalaje: III (E)

Marítimo (IMDG):
Denominación técnica: AMONIACO EN SOLUCIÓN acuosa con más del 10% pero no más del 35% de amoniaco
ONU 2672 Clase: 8 Grupo de embalaje: III
Aéreo (ICAO-IATA):
Denominación técnica: Amoníaco en solución
ONU 2672 Clase: 8 Grupo de embalaje: III
Instrucciones de embalaje: CAO 813 PAX 819

15. Información reglamentaria

15.1 Etiquetado según REACH

Símbolos:

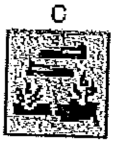


Indicaciones de peligro: Corrosivo Peligroso para medio ambiente
Frases R: 34 Provoca quemaduras.
Frases S: 26-36/37/39-45-61 En caso de contacto con los ojos, lávense inmediata y abundantemente con agua y acúdase a un médico. Usense indumentaria y guantes adecuados y protección para los ojos-la cara. En caso de accidente o malestar, acuda inmediatamente al médico (si es posible, muéstrele la etiqueta). Evítese su liberación al medio ambiente. Recábense instrucciones específicas de la ficha de datos de seguridad.
Número de índice CE: 007-001-01-2

16. Otras informaciones

Respecto a la revisión anterior, se han producido cambios en los apartados: 5.
Información de los componentes:

Amoníaco 20% (en NH_3)
CAS [1336-21-6] NH_3 M.=17,03
215-647-6 007-001-01-2



R: 34
Provoca quemaduras.

Número y fecha de la revisión:1 14.05.09
Los datos consignados en la presente Ficha de Datos de Seguridad, están basados en nuestros conocimientos actuales, teniendo como único objeto informar sobre aspectos de seguridad y no garantizándose las propiedades y características en ella indicadas.