(11) EP 4 202 119 A1

(12)

EUROPEAN PATENT APPLICATION

(43) Date of publication: 28.06.2023 Bulletin 2023/26

(21) Application number: 21217014.6

(22) Date of filing: 22.12.2021

(51) International Patent Classification (IPC):

(52) Cooperative Patent Classification (CPC):
 D21H 17/375; D21H 17/28; D21H 17/52;
 D21H 17/55; D21H 17/56; D21H 21/18;

D21H 21/20; D21H 27/10

(84) Designated Contracting States:

AL AT BE BG CH CY CZ DE DK EE ES FI FR GB GR HR HU IE IS IT LI LT LU LV MC MK MT NL NO PL PT RO RS SE SI SK SM TR

Designated Extension States:

BA ME

Designated Validation States:

KH MA MD TN

(71) Applicant: Billerud Aktiebolag (publ) 169 27 Solna (SE)

(72) Inventors:

- ATHLEY, Karin Norrköping (SE)
- WAHLSTRÖM, Lars Norrköping (SE)
- (74) Representative: Kransell & Wennborg KB P.O. Box 27834
 115 93 Stockholm (SE)

Remarks:

Amended claims in accordance with Rule 137(2) EPC.

(54) RECLABLE PAPER OF HIGH WET STRENGTH

(57) There is provided a paper comprising polyamino amide epichlorohydrin (PAE) resin and glyoxylated polyacrylamide (G-PAM), wherein the amount of PAE resin is 0.15-1.75 kg/tonne dry paper.

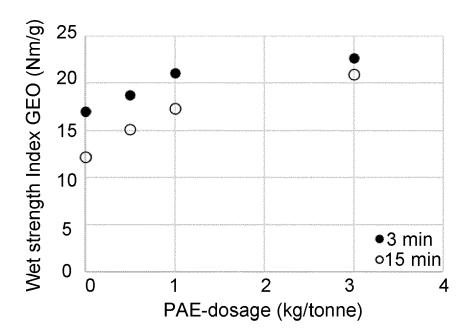


Fig. 1

EP 4 202 119 A

Description

TECHNICAL FIELD

[0001] The present disclosure relates to the field of paper and in particular recyclable paper having relatively high wet strength.

BACKGROUND

[0002] Paper for some applications must retain some strength also if wetted. Hence, various "wet-strength agents" have been developed for papermaking. A common drawback of the use of such wet-strength agents is that it is difficult to repulp and hence recycle the paper.

SUMMARY

15

20

30

50

[0003] An objective of the present disclosure is to provide a paper of relatively high wet strength that is recyclable.

[0004] Accordingly, the present disclosure provides a paper (preferably a kraft paper) comprising polyamino amide

epichlorohydrin (PAE) resin and glyoxylated polyacrylamide (G-PAM), wherein the amount of PAE resin is 0.15-1.75 kg/tonne dry paper.

BRIEF DESCRIPTION OF THE DRAWINGS

[0005]

Figure 1 shows the geometrical wet tensile strength index (Nm/g) measured after 3 or 15 minutes of wetting time for paper sheets comprising different amounts (kg/tonne) of PAE resin.

Figure 2 shows the recyclability (%) according to PTS Method PTS-RH 021/97 for paper sheets comprising different amounts (kg/tonne) of PAE resin.

[0006] Further details of the paper sheets are given in the Examples section below.

DETAILED DESCRIPTION

[0007] As a first aspect of the present disclosure, there is provided a paper comprising polyamino amide epichlorohydrin (PAE) resin and glyoxylated polyacrylamide (G-PAM). The amount of PAE resin is 0.15-1.75 kg/tonne dry paper.

[0008] The amount of G-PAM is preferably 0.5-6.0 kg/tonne dry paper and more preferably 1.0-5.0 kg/tonne dry paper, such as 1.5-4.0 kg/tonne dry paper.

[0009] The amount of PAE resin is preferably 0.25-1.50 kg/tonne dry paper, more preferably 0.30-1.25 kg/tonne dry paper, such as 0.30-1.10 kg/tonne dry paper or 0.40-1.25 kg/tonne dry paper.

[0010] The paper of any one of the preceding claims, wherein said G-PAM is cationic G-PAM.

[0011] In the papermaking, the PAE resin and the G-PAM is typically added in the wet end. The PAE resin and the G-PAM are thus wet end chemicals.

[0012] The paper is preferably a kraft paper, such as a machine glazed (MG) kraft paper.

machine. Examples of hydrophobic size are AKD, ASA and rosin size.

[0013] The paper may be bleached or unbleached. Bleached paper is typically easier to repulp and may therefore be preferred.

[0014] The grammage of the paper is typically 35-200 g/m² and preferably 50-140 g/m². In one embodiment, the grammage is 50-110 g/m². In another embodiment, the grammage is 60-120 g/m². In yet another embodiment, the grammage is 35-55 g/m². In the present disclosure, grammage is measured according to ISO 536:2019.

[0015] The teachings of the present disclosure are not intended for tissue paper, such as bath, towel, facial or napkin paper. Accordingly, the paper of the present disclosure typically has a density of at least 600 kg/m³, such as 600-1000 kg/m³. In the present disclosure, density is measured according to ISO 534:2011.

[0016] To improve the performance in wet conditions, the water absorbency of the paper is preferably relatively low. Hence the Cobb 60 s value of at least one side of the paper is preferably below 40 g/m², more preferably below 30 g/m², such as below 25 g/m². In one embodiment, both sides of the paper have a Cobb 60 s value below 40 g/m², such as below 30 g/m², such as below 25 g/m². In the present disclosure, Cobb 60 s is measured according to ISO 535:2014. **[0017]** To obtain a relatively low Cobb 60 s value, hydrophobic size may be added, typically in the wet end of the paper

[0018] Typically, not only the wet tensile strength, but also the dry tensile strength is of interest in the application in which the paper is used. In one embodiment, the (dry) geometrical tensile index measured according to ISO 1924-3:2005 is at least 50 Nm/g, such as 50-150 Nm/g.

[0019] The geometrical wet tensile index measured according to ISO 1924-3:2005 (using a wetting time of 10 min) of the paper is typically at least 10 Nm/g, such as at least 15 Nm/g, such as at least 16 Nm/g.

[0020] The paper is preferably formed from a softwood pulp or a mixture of a softwood pulp and a hardwood pulp. Softwood pulp typically provides better strength and hardwood typically provides better surface properties.

[0021] In one embodiment, at least 75% by dry weight of the fibres of the paper are virgin fibres and preferably at least 90% by dry weight of the fibres of the paper are virgin fibres. Virgin fibres provide better strength than recycled fibres.

[0022] Starch is often added as a strength agent in papermaking. However, the above-mentioned addition of G-PAM allows for a relatively low amount of starch. In one embodiment, the paper of the present disclosure comprises less than 3.5 kg/tonne dry paper of starch, such as 1-3 kg/tonne dry paper of starch.

[0023] The starch is preferably cationic starch.

[0024] Strength may be impaired by the addition of inorganic filler to the paper pulp. A high amount of filler corresponds to a high ash content. The paper of the present disclosure preferably has an ash content of less than 7.0% by dry weight, such as less than 5.5% by dry weight.

[0025] As a second aspect of the present disclosure there is provided a package comprising a paper according to the first aspect of the present disclosure. In one embodiment, the package is a Quick Service Restaurant food package. In this embodiment, the package is typically a bag or a wrap. In another embodiment, the package is a medical device package, in particular a medical device package that will undergo or has undergone gas or steam sterilization. Further, the package may be a sack, such as a potato sack or a sack for a hydraulic binder (e.g. a cement sack), a garbage bag, a pouch for foods (e.g. fruits or vegetables) or an e-commerce bag.

[0026] In the Quick Service Restaurant food package, the grammage of the paper is typically in the range of 35-55 g/m². [0027] As a third aspect of the present disclosure, there is provided a coated barrier paper comprising a paper substrate, which is a paper according to the first aspect, and a barrier coating provided on the paper substrate.

EXAMPLES

10

30

35

40

45

[0028] Lab sheets were produced in a dynamic sheet former. The target grammage was 80 g/m². The lab procedure started with the preparation of a fiber suspension, which was a mixture bleached softwood kraft pulp and bleached hardwood kraft pulp in a dry weight ratio of 70:30. Both pulps were taken from the production of MG paper in Billerud-Korsnäs' mill in Skärblacka. The pulps were diluted with tap water to obtain a fibre consistency of 12.5 g/l and the pH was adjusted 8.0. Furnishes for sheet forming were then prepared according to Table 1.

Table 1. "PAE" was Kymene GHP20 from Solenis, which is a polyamino amide epichlorohydrin resin. "AKD" was FennoSize 364M from Kemira. "G-PAM" was Fennobond 3150E from Kemira, which is a cationic glyoxylated polyacrylamide. "Silica" was FennoSil 442 from Kemira. "Starch" was Solbond PC₃₅ from Solenis which is a cationic starch with degree of substitution 35%.

Step	Additive	Fibre consist. (g/l)	Dosage (g/kg dry fibre)	Stirring time (s)
1	Fibre	12.5		
2	PAE	12.5	Acc. to Table 2	30
3	AKD	12.5		30
4	G-PAM	12.5	Acc. to Table 2	15
5	Tap water	5		15
5	Alum	5	4	Added with the tap water
5	Sulphuric acid	5	To obtain pH 6	Added with the tap water
6	Starch	5	Acc. to Table 2	20
7	Silica	5	0.25	10

55

Table 2.

	PAE (kg/tonne)	G-PAM (kg/tonne)	Starch (kg/ton)
Series 1	3	0	4
Series 2	1	2.8	2
Series 3	0.5	2.8	2
Series 4	0	2.8	2

10

15

5

[0029] Ten sheets per series were made. Pressing was carried out by two passes through a nip press (5 bar). Drying was carried out in a "STFI dryer" at 130 °C for 10 minutes.

[0030] To simulate the curing that paper typically undergoes in the paper machine and after the paper machine, but before the paper is used by the customer, the sheets were cured at 105 °C for 2 * 30 minutes.

[0031] After curing, sheets were tested for the following properties: dry tensile strength (ten stripes per series in MD and seven stripes per series in CD except for series 2 for which only two stripes could be tested due to restricted amount of paper); COBB 60 s; air resistance (Gurley); and wet tensile strength measured after wetting times of 1, 3, 5, and 15 minutes (ten stripes per series in MD and seven stripes per series in CD except for series 2 for which only two stripes could be tested due to restricted amount of paper).

[0032] Tensile index values were calculated by dividing the tensile strength values by the grammage. Geometrical tensile index values were calculated as the geometrical mean of the tensile index in MD and the tensile index in CD.

[0033] Further, sheets were sent to Karlstad University for recyclability testing in accordance with PTS Method PTS-

RH 021/97.

[0034] Results of the tests are presented in the tables 3 and 4 below.

25

30

35

Table 3. "WS₃" means average geometrical wet tensile strength index measured after 3 minutes of wetting time. "WS₅" means average geometrical wet tensile strength index measured after 5 minutes of wetting time. "WS₁₅" means average geometrical wet tensile strength index measured after 15 minutes of wetting time.

Series	PAE (kg/ tonne)	G-PAM (kg/ tonne)	Recyclability* (%)	WS ₃ (Nm/g)	WS5 (Nm/g)	WS ₁₅ (Nm/g)
1	3.0	0	46,5	22.7	21.6	21.0
2	0	2.8	99.7	17.0	14.9	12.2
3	0.5	2.8	98.0	18.8	17.1	15.1
4	1.0	2.8	93.7	21.1	18.3	17.3

^{*} Recyclability (%) means the percentage suitable for recycling or usable in papermaking. It corresponds to the total mass of the sample (50g oven-dry $\stackrel{\land}{=}$ 100%) less the total reject.

40

[0035] The results of Table 3 are also presented in figures 1 and 2. Notably, figure 1 shows a sharp increase in the wet strength when 0.5 kg/tonne and 1.0 kg/tonne PAE is added, but a more modest increase when the amount is higher. Conversely, figure 2 shows a relatively modest decrease of the recyclability when 0.5 kg/tonne and 1.0 kg/tonne PAE is added, and a sharper decrease beyond those amounts. Consequently, figures 1 and 2 together show that the optimal amount of PAE is from about 0.25 kg/tonne to about 1.5 kg/tonne when high wet strength is to be combined with high recyclability, provided that G-PAM has been added.

[0036] Further properties of the sheets are presented in table 4 below.

[0037] Table 4. Average values for Cobb, Gurley and dry strength. The tests behind the results in this table were carried out between the first and the second curing, i.e. after curing at 105 °C for 1 * 30 minutes.

55

Series	PAE (kg/ tonne)	G-PAM (kg/ tonne)	Cobb 60s (g/m²)	Gurley (s)	Geometrical dry tensile strength index (Nm/g)
1	3.0	0	21.4	18.5	99.3
2	0	2.8	19.3	24.2	107.2
3	0.5	2.8	18.9	22.6	105.9
4	1.0	2.8	18.9	15.4	110.0

Claims

- 1. A paper comprising polyamino amide epichlorohydrin (PAE) resin and glyoxylated polyacrylamide (G-PAM), wherein the amount of PAE resin is 0.15-1.75 kg/tonne dry paper.
- 2. The paper of claim 1, wherein the amount of G-PAM is 0.5-6.0 kg/tonne dry paper, preferably 1.0-5.0 kg/tonne dry paper, such as 1.5-4.0 kg/tonne dry paper.
- 3. The paper of claim 1 or 2, wherein the amount of PAE resin is 0.25-1.50 kg/tonne dry paper, preferably 0.30-1.25 10 kg/tonne dry paper, such as 0.30-1.10 kg/tonne dry paper or 0.40-1.25 kg/tonne dry paper.
 - 4. The paper of any one of the preceding claims, wherein said G-PAM is cationic G-PAM.
 - 5. The paper of any one of the preceding claims, which is a kraft paper, such as a machine glazed (MG) kraft paper.
 - **6.** The paper of any one of the preceding claims, which is bleached.
 - 7. The paper of any one of the preceding claims, wherein the grammage measured according to ISO 536:2019 is 35-200 g/m², such as 50-200 g/m², preferably 50-140 g/m², such as 50-110 g/m² or 60-120 g/m².
 - 8. The paper of any one of the preceding claims, wherein the density measured according to ISO 534:2011 is at least 600 kg/m³, such as 600-1000 kg/m³.
 - 9. The paper of any one of the preceding claims, wherein the Cobb 60 s value measured according to ISO 535:2014 of at least one side of the paper is below 40 g/m², preferably below 30 g/m², such as below 25 g/m².
 - 10. The paper of any one of the preceding claims, wherein the geometrical tensile index measured according to ISO 1924-3:2005 is at least 50 Nm/g, such as 50-150 Nm/g.
- 30 11. The paper of any one of the preceding claims, wherein the geometrical wet tensile index measured according to ISO 1924-3:2005 is at least 10 Nm/g, such as at least 15 Nm/g, such as at least 16 Nm/g.
 - 12. The paper of any one of the preceding claims, which is formed from a softwood pulp or a mixture of a softwood pulp and a hardwood pulp.
 - 13. The paper of any one of the preceding claims, wherein at least 75% by dry weight of the fibres of the paper are virgin fibres and optionally wherein at least 90% by dry weight of the fibres of the paper are virgin fibres.
- 14. The paper of any one of the preceding claims, wherein the paper comprises less than 3.5 kg/tonne dry paper of starch, such as 1-3 kg/tonne dry paper of starch.
 - **15.** A package comprising a paper according to any one of the preceding claims.

45 Amended claims in accordance with Rule 137(2) EPC.

- 1. A paper comprising polyamino amide epichlorohydrin (PAE) resin, glyoxylated polyacrylamide (G-PAM) and starch, wherein:
 - the amount of PAE resin is 0.15-1.75 kg/tonne dry paper;
 - the amount of starch is less than 3.5 kg/tonne dry paper, such as 1-3 kg/tonne dry paper; and
 - the dry geometrical tensile index measured according to ISO 1924-3:2005 is at least 50 Nm/g, such as 50-150
- 2. The paper of claim 1, wherein the amount of G-PAM is 0.5-6.0 kg/tonne dry paper, preferably 1.0-5.0 kg/tonne dry paper, such as 1.5-4.0 kg/tonne dry paper.
 - 3. The paper of claim 1 or 2, wherein the amount of PAE resin is 0.25-1.50 kg/tonne dry paper, preferably 0.30-1.25

5

5

15

20

25

35

40

50

kg/tonne dry paper, such as 0.30-1.10 kg/tonne dry paper or 0.40-1.25 kg/tonne dry paper.

- 4. The paper of any one of the preceding claims, wherein said G-PAM is cationic G-PAM.
- 5. The paper of any one of the preceding claims, which is a kraft paper, such as a machine glazed (MG) kraft paper.
 - **6.** The paper of any one of the preceding claims, which is bleached.

20

25

30

35

40

45

50

55

- 7. The paper of any one of the preceding claims, wherein the grammage measured according to ISO 536:2019 is 35-200 g/m², such as 50-200 g/m², preferably 50-140 g/m², such as 50-110 g/m² or 60-120 g/m².
 - **8.** The paper of any one of the preceding claims, wherein the density measured according to ISO 534:2011 is at least 600 kg/m3, such as 600-1000 kg/m3.
- **9.** The paper of any one of the preceding claims, wherein the Cobb 60 s value measured according to ISO 535:2014 of at least one side of the paper is below 40 g/m², preferably below 30 g/m², such as below 25 g/m².
 - **10.** The paper of any one of the preceding claims, wherein the geometrical wet tensile index measured according to ISO 1924-3:2005 is at least 10 Nm/g, such as at least 15 Nm/g, such as at least 16 Nm/g.
 - **11.** The paper of any one of the preceding claims, which is formed from a softwood pulp or a mixture of a softwood pulp and a hardwood pulp.
 - **12.** The paper of any one of the preceding claims, wherein at least 75% by dry weight of the fibres of the paper are virgin fibres and optionally wherein at least 90% by dry weight of the fibres of the paper are virgin fibres.
 - **13.** A package comprising a paper according to any one of the preceding claims.

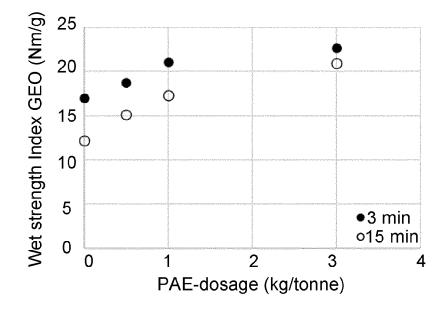


Fig. 1

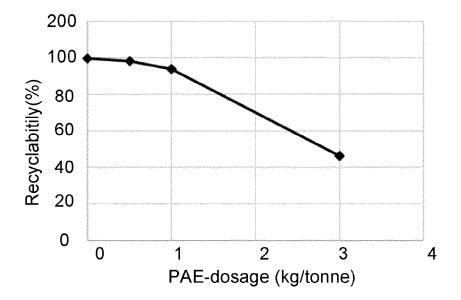


Fig. 2



EUROPEAN SEARCH REPORT

Application Number

EP 21 21 7014

J	
10	
15	
20	
25	
30	
35	
40	
45	
50	

	DOCUMENTS CONSID	FRED TO BE RI	ELEVANT	T				
Category	Citation of document with i		priate,	Relevant to claim		TICATION OF THE ATION (IPC)		
х	US 5 427 652 A (DAF ET AL) 27 June 1995 * claims 1-8; examp	(1995-06-27)	AM B [US]	1,15	INV. D21H2: D21H1'	7/37		
x	WO 95/21298 A1 (MEA 10 August 1995 (199 * claims 1-17; exam	5-08-10)		1,15	D21H1 D21H1 D21H2	7/55 7/56		
x	WO 99/50500 A1 (CAI 7 October 1999 (199 * claims 1-9; examp	9-10-07)	s])	1,15				
x	WO 2016/085836 A1 (2 June 2016 (2016-0 * claims 1-12; exam	6-02)	C [US])	1,15				
x	US 2017/314204 A1 (2 November 2017 (20 * claims 1-37 *		ET AL)	1,15				
A	WO 2015/075318 A1 (28 May 2015 (2015-0	-	I])	1-15	TECHN SEARC	ICAL FIELDS HED (IPC)		
	* the whole documen	t * 			D21H			
A	US 2010/269995 A1 (ET AL) 28 October 2 * the whole documen	010 (2010-10-		1-15				
A	WO 01/77437 A1 (SCA SANDBERG SUSSAN [SE 18 October 2001 (20 * the whole document]; ANDREASSON 01-10-18)		1-15				
A	EP 0 835 957 A2 (FC 15 April 1998 (1998 * the whole document	-04-15)	[US])	1-15				
	The present search report has	been drawn up for all c	laims					
	Place of search	Date of comple	etion of the search		Examine			
	Munich	3 June	2022	Kaı	clsson,	Lennart		
X : part Y : part doci	ATEGORY OF CITED DOCUMENTS icularly relevant if taken alone icularly relevant if combined with anotument of the same category inological background	her E	: theory or principle : earlier patent doc after the filing dat) : document cited in : document cited fo	e underlying the cument, but puble n the application or other reasons	invention ished on, or			
O : non	-written disclosure rmediate document	8			& : member of the same patent family, corresponding document			

55

ANNEX TO THE EUROPEAN SEARCH REPORT ON EUROPEAN PATENT APPLICATION NO.

EP 21 21 7014

5

This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report. The members are as contained in the European Patent Office EDP file on The European Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

03-06-2022

10	Patent document cited in search report	Publication date	Patent far member		Publication date
	US 5427652 A	27-06-1995	US 54276	52 A	27-06-1995
			US 54663	37 A	14-11-1995
			WO 95212	97 A1	10-08-1995
15	WO 9521298 A1	10-08-1995	AU 17429	95 A	21-08-1995
			WO 95212	98 A1	10-08-1995
	WO 9950500 A1	07-10-1999	AU 32173	99 A	18-10-1999
			US 60773	94 A	20-06-2000
20			WO 99505	00 A1	07-10-1999
	WO 2016085836 A1	02-06-2016	BR 1120170112		26-12-2017
				68 A1	02-06-2016
			CN 1056964		22-06-2016
25			CN 1071097		29-08-2017
20				13 A1	04-10-2017
			KR 201700866		26-07-2017
			US 20182985		18-10-2018
			WO 20160858	36 A1	02-06-2016
30	US 2017314204 A1	02-11-2017	NONE		
	WO 2015075318 A1	28-05-2015	AU 20143517		09-06-2016
				93 A1	28-05-2015
			CN 1058997		24-08-2016
				51 A1	28-09-2016
35			ES 29072	76 T 3	22-04-2022
			JP 20165392	56 A	15-12-2016
			KR 201600894	33 A	27-07-2016
			KR 202200385		28-03-2022
				88 A	25-01-2019
40				51 T3	25-04-2022
			PT 30717		21-02-2022
				87 C1	18-09-2018
			RU 20161190		25-12-2017
			US 20151442		28-05-2015
45			WO 20150753	18 A1	28-05-2015
70	US 2010269995 A1	28-10-2010	BR PI10141		05-04-2016
			CN 1024143		11-04-2012
				12 A1	29-02-2012
				76 B2	04-11-2015
			JP 20125248	47 A	18-10-2012
50	,		US 20102699		28-10-2010
2045	3		WO 20101235	80 A1	28-10-2010
ORM P0459					
Ö)				

55

page 1 of 2

For more details about this annex : see Official Journal of the European Patent Office, No. 12/82

ANNEX TO THE EUROPEAN SEARCH REPORT ON EUROPEAN PATENT APPLICATION NO.

EP 21 21 7014

This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report. The members are as contained in the European Patent Office EDP file on The European Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

03-06-2022

AU 4297101 A 23-10- BR 0109841 A 03-06- CN 1422347 A 04-06- DE 60102082 T2 23-12- EP 1282741 A1 12-02- ES 2215887 T3 16-10- HU 0300410 A2 28-06- MX PA02009106 A 12-03- PL 357809 A1 26-07- SK 14682002 A3 01-04- WO 0177437 A1 18-10-	AU 4297101 A 23-10- BR 0109841 A 03-06- CN 1422347 A 04-06- DE 60102082 T2 23-12- EP 1282741 A1 12-02- ES 2215887 T3 16-10- HU 0300410 A2 28-06- MX PA02009106 A 12-03- PL 357809 A1 26-07- SK 14682002 A3 01-04- WO 0177437 A1 18-10- EP 0835957 A2 15-04-1998 DE 69724211 T2 09-06- EP 0835957 A2 15-04- US 6419789 B1 16-07- US 2002074100 A1 20-06- US 2006032595 A1 16-02-		ed in search report		date		member(s)	date
BR 0109841 A 03-06- CN 1422347 A 04-06- DE 60102082 T2 23-12- EP 1282741 A1 12-02- ES 2215887 T3 16-10- HU 0300410 A2 28-06- MX PA02009106 A 12-03- PL 357809 A1 26-07- SK 14682002 A3 01-04- WO 0177437 A1 18-10- EP 0835957 A2 15-04-1998 DE 69724211 T2 09-06- EP 0835957 A2 15-04- US 6419789 B1 16-07- US 2002074100 A1 20-06- US 2006032595 A1 16-02-	BR 0109841 A 03-06- CN 1422347 A 04-06- DE 60102082 T2 23-12- EP 1282741 A1 12-02- ES 2215887 T3 16-10- HU 0300410 A2 28-06- MX PA02009106 A 12-03- PL 357809 A1 26-07- SK 14682002 A3 01-04- WO 0177437 A1 18-10- EP 0835957 A2 15-04-1998 DE 69724211 T2 09-06- EP 0835957 A2 15-04- US 6419789 B1 16-07- US 2002074100 A1 20-06- US 2006032595 A1 16-02-	WO	0177437	A1	18-10-2001	AT		15-03-2
CN 1422347 A 04-06- DE 60102082 T2 23-12- EP 1282741 A1 12-02- ES 2215887 T3 16-10- HU 0300410 A2 28-06- MX PA02009106 A 12-03- PL 357809 A1 26-07- SK 14682002 A3 01-04- WO 0177437 A1 18-10- EP 0835957 A2 15-04-1998 DE 69724211 T2 09-06- EP 0835957 A2 15-04- ES 2202558 T3 01-04- US 6419789 B1 16-07- US 2002074100 A1 20-06- US 2006032595 A1 16-02-	CN 1422347 A 04-06- DE 60102082 T2 23-12- EP 1282741 A1 12-02- ES 2215887 T3 16-10- HU 0300410 A2 28-06- MX PA02009106 A 12-03- PL 357809 A1 26-07- SK 14682002 A3 01-04- WO 0177437 A1 18-10- EP 0835957 A2 15-04-1998 DE 69724211 T2 09-06- EP 0835957 A2 15-04- US 6419789 B1 16-07- US 2002074100 A1 20-06- US 2006032595 A1 16-02-							23-10-2
DE 60102082 T2 23-12- EP 1282741 A1 12-02- ES 2215887 T3 16-10- HU 0300410 A2 28-06- MX PA02009106 A 12-03- PL 357809 A1 26-07- SK 14682002 A3 01-04- WO 0177437 A1 18-10- EP 0835957 A2 15-04-1998 DE 69724211 T2 09-06- EP 0835957 A2 15-04- US 6419789 B1 16-07- US 2002074100 A1 20-06- US 2006032595 A1 16-02-	DE 60102082 T2 23-12- EP 1282741 A1 12-02- ES 2215887 T3 16-10- HU 0300410 A2 28-06- MX PA02009106 A 12-03- PL 357809 A1 26-07- SK 14682002 A3 01-04- WO 0177437 A1 18-10- EP 0835957 A2 15-04-1998 DE 69724211 T2 09-06- EP 0835957 A2 15-04- US 2002074100 A1 20-06- US 2006032595 A1 16-02-							03-06-2
EP 1282741 A1 12-02- ES 2215887 T3 16-10- HU 0300410 A2 28-06- MX PA02009106 A 12-03- PL 357809 A1 26-07- SK 14682002 A3 01-04- WO 0177437 A1 18-10- EP 0835957 A2 15-04-1998 DE 69724211 T2 09-06- EP 0835957 A2 15-04- US 6419789 B1 16-07- US 2002074100 A1 20-06- US 2006032595 A1 16-02-	EP 1282741 A1 12-02- ES 2215887 T3 16-10- HU 0300410 A2 28-06- MX PA02009106 A 12-03- PL 357809 A1 26-07- SK 14682002 A3 01-04- WO 0177437 A1 18-10- EP 0835957 A2 15-04-1998 DE 69724211 T2 09-06- EP 0835957 A2 15-04- US 6419789 B1 16-07- US 2002074100 A1 20-06- US 2006032595 A1 16-02-							
ES 2215887 T3 16-10- HU 0300410 A2 28-06- MX PA02009106 A 12-03- PL 357809 A1 26-07- SK 14682002 A3 01-04- WO 0177437 A1 18-10- EP 0835957 A2 15-04-1998 DE 69724211 T2 09-06- EP 0835957 A2 15-04- ES 2202558 T3 01-04- US 6419789 B1 16-07- US 2002074100 A1 20-06- US 2006032595 A1 16-02-	ES 2215887 T3 16-10- HU 0300410 A2 28-06- MX PA02009106 A 12-03- PL 357809 A1 26-07- SK 14682002 A3 01-04- WO 0177437 A1 18-10- EP 0835957 A2 15-04-1998 DE 69724211 T2 09-06- EP 0835957 A2 15-04- ES 2202558 T3 01-04- US 6419789 B1 16-07- US 2002074100 A1 20-06- US 2006032595 A1 16-02-							
HU 0300410 A2 28-06- MX PA02009106 A 12-03- PL 357809 A1 26-07- SK 14682002 A3 01-04- WO 0177437 A1 18-10- EP 0835957 A2 15-04-1998 DE 69724211 T2 09-06- EP 0835957 A2 15-04- US 6419789 B1 16-07- US 2002074100 A1 20-06- US 2006032595 A1 16-02-	HU 0300410 A2 28-06- MX PA02009106 A 12-03- PL 357809 A1 26-07- SK 14682002 A3 01-04- WO 0177437 A1 18-10- EP 0835957 A2 15-04-1998 DE 69724211 T2 09-06- EP 0835957 A2 15-04- US 6419789 B1 16-07- US 2002074100 A1 20-06- US 2006032595 A1 16-02-							
MX PA02009106 A 12-03- PL 357809 A1 26-07- SK 14682002 A3 01-04- WO 0177437 A1 18-10- EP 0835957 A2 15-04-1998 DE 69724211 T2 09-06- EP 0835957 A2 15-04- ES 2202558 T3 01-04- US 6419789 B1 16-07- US 2002074100 A1 20-06- US 2006032595 A1 16-02-	MX PA02009106 A 12-03- PL 357809 A1 26-07- SK 14682002 A3 01-04- WO 0177437 A1 18-10- EP 0835957 A2 15-04-1998 DE 69724211 T2 09-06- EP 0835957 A2 15-04- ES 2202558 T3 01-04- US 6419789 B1 16-07- US 2002074100 A1 20-06- US 2006032595 A1 16-02-							
PL 357809 A1 26-07- SK 14682002 A3 01-04- WO 0177437 A1 18-10- EP 0835957 A2 15-04-1998 DE 69724211 T2 09-06- EP 0835957 A2 15-04- ES 2202558 T3 01-04- US 6419789 B1 16-07- US 2002074100 A1 20-06- US 2006032595 A1 16-02-	PL 357809 A1 26-07- SK 14682002 A3 01-04- WO 0177437 A1 18-10- EP 0835957 A2 15-04-1998 DE 69724211 T2 09-06- EP 0835957 A2 15-04- ES 2202558 T3 01-04- US 6419789 B1 16-07- US 2002074100 A1 20-06- US 2006032595 A1 16-02-							
SK 14682002 A3 01-04- WO 0177437 A1 18-10- EP 0835957 A2 15-04-1998 DE 69724211 T2 09-06- EP 0835957 A2 15-04- ES 2202558 T3 01-04- US 6419789 B1 16-07- US 2002074100 A1 20-06- US 2006032595 A1 16-02-	SK 14682002 A3 01-04- WO 0177437 A1 18-10- EP 0835957 A2 15-04-1998 DE 69724211 T2 09-06- EP 0835957 A2 15-04- ES 2202558 T3 01-04- US 6419789 B1 16-07- US 2002074100 A1 20-06- US 2006032595 A1 16-02-							
WO 0177437 A1 18-10-1 EP 0835957 A2 15-04-1998 DE 69724211 T2 09-06-1 EP 0835957 A2 15-04-1 ES 2202558 T3 01-04-1 US 6419789 B1 16-07-1 US 2002074100 A1 20-06-1 US 2006032595 A1 16-02-1	WO 0177437 A1 18-10- EP 0835957 A2 15-04-1998 DE 69724211 T2 09-06- EP 0835957 A2 15-04- ES 2202558 T3 01-04- US 6419789 B1 16-07- US 2002074100 A1 20-06- US 2006032595 A1 16-02-							
EP 0835957 A2 15-04-1998 DE 69724211 T2 09-06- EP 0835957 A2 15-04- ES 2202558 T3 01-04- US 6419789 B1 16-07- US 2002074100 A1 20-06- US 2006032595 A1 16-02-	EP 0835957 A2 15-04-1998 DE 69724211 T2 09-06- EP 0835957 A2 15-04- ES 2202558 T3 01-04- US 6419789 B1 16-07- US 2002074100 A1 20-06- US 2006032595 A1 16-02-							
EP 0835957 A2 15-04- ES 2202558 T3 01-04- US 6419789 B1 16-07- US 2002074100 A1 20-06- US 2006032595 A1 16-02-	EP 0835957 A2 15-04- ES 2202558 T3 01-04- US 6419789 B1 16-07- US 2002074100 A1 20-06- US 2006032595 A1 16-02-							
ES 2202558 T3 01-04- US 6419789 B1 16-07- US 2002074100 A1 20-06- US 2006032595 A1 16-02-	ES 2202558 T3 01-04- US 6419789 B1 16-07- US 2002074100 A1 20-06- US 2006032595 A1 16-02-	EP	0835957	A 2	15-04-1998			09-06-2
US 6419789 B1 16-07- US 2002074100 A1 20-06- US 2006032595 A1 16-02-	US 6419789 B1 16-07- US 2002074100 A1 20-06- US 2006032595 A1 16-02-							
US 2002074100 A1 20-06-3 US 2006032595 A1 16-02-3	US 2002074100 A1 20-06- US 2006032595 A1 16-02-							
US 2006032595 A1 16-02-	US 2006032595 A1 16-02-							
US 2007272380 AI 29-11-	US 2007272380 AI 29-11-							

page 2 of 2