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(54) **Aqueous suspensions for paper coating**

(57) Aqueous inorganic pigment suspensions which are useful for the coating of paper with film coaters contain a viscoelastic polysaccharide, and particularly a viscoelastic carboxymethyl cellulose, as thickener.

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Description

Technical Field

[0001] The present invention relates to aqueous inorganic pigment suspensions useful for the coating of paper and cardboard (from here ahead both indicated with the term "paper") with film coaters containing a viscoelastic polysaccharide as thickener.

[0002] It is well-known that, in order to improve the characteristics of printing paper, a coating is commonly applied on its surface; the coating, a layer of pigments and additives, makes the surface more smooth and glossy and, thanks to the resulting improved interaction with printing inks, gives the surface good characteristics of printability.

[0003] The formulations for coating, commonly called coating colours, normally consist of water, inorganic pigments (such as calcium carbonate, kaolin, titanium dioxide, chalk and mixtures thereof), polymeric binder, for example a styrene butadiene latex, and thickener; the thickener, beside acting as rheology modifier, also serves as water retention agent during the application. In addition, other additives, such as glycols or fatty acid derivatives, used as lubricants, antifoaming agents and biocides can be present.

[0004] These formulations are applied on the paper by means of specific machines (coaters).

[0005] The coaters can be stand-alone units (coating "outside machine") or integrated in a continuous apparatus (coating "in machine").

[0006] In addition to the accessory parts that guarantee the unwrapping and the wrapping of the paper sheet before and after the coating step, the coater is basically made of one or more coating heads and from units allowing the drying of the coating colour after the application (infrared oven, warm air oven, cylinders dryers).

[0007] According to their operative principle, the coating heads can fundamentally be divided in: blade coaters, film coaters (metering size press - MSP) and coaters without devices for dosing and levelling the coating colour coming into contact with the paper (curtain coaters, spray coaters).

[0008] As reported on "Paper Coating Additives", Tappi Press, 1996, pages 21-35, the thickeners more commonly used in the formulations for coating colours are cellulose ethers, like carboxymethyllcellulose (CMC) and hydroxyethylcellulose, starches and starch derivatives, and water-soluble acrylic polymers, also called "alkali swellable emulsion" or ASE.

[0009] When film coaters are used (film coaters are also often referred to as "roll coaters"), the above cited thickeners do not provide good performances, as reported by way of example in the patent WO 2006/093497.

[0010] As a matter of fact, film coaters operate at high speed.

[0011] The high speed, with traditional thickeners, results in the surface of the coating colour being rough and not uniform, or, in technical terms, showing an "orange peel" appearance; furthermore, the high speed may produce the phenomenon of "misting", which consists in an undesired nebulisation of the coating colour in the proximity of the application head.

[0012] The two phenomena, that are often concurrently present, have negative effects on the quality of the final paper and on the coating process; the misting contribute to dirtying of the coater, to dropping of the coating colour onto the paper and to scaling in the drying area, close to the coating heads.

[0013] Background Art

[0014] In order to resolve the aforesaid technical problems and with the aim to improve the performances of the suspensions for the film coating of paper, some specific thickeners for coating formulations which can be applied in particular with coaters operating at high speed have been proposed, such as for example acrylic polymers in inverse emulsion.

[0015] We mention, among the many patents on the argument, WO 2004/076743, describing a thickening composition for the coating of the paper comprising an ASE associative polyacrylate (HASE), and JP 10226993, describing coating colours comprising hydrolyzed cellulose and, among the other ingredients, a traditional carboxymethyl cellulose. Nonetheless, all the up to now proposed solutions suffer from some disadvantage.

[0016] US 5,718,756 describes paper coatings containing a fine particle size kaolin pigment and the use of conventional carboxymethyl cellulose as the thickening agent.

[0017] US 5,725,648 describes paper coating compositions containing guar and at least one other water-soluble polymer, conventional carboxymethyl cellulose being one of the preferred water-soluble polymer that can be used in the realisation of the invention.

[0018] The conventional carboxymethyl celluloses which are commonly present on the market are non-viscoelastic and impart good water retention and thickening but, as already said, they do not allow to overcome the above-mentioned problems; the associative thickeners provide low water retention despite their high thickening power; the use of inverse emulsion acrylic polymers adds in the coating colour, together with the active polymer, a significant amount of oil, that can create difficulties to the coating process and environmental problems.

[0019] It is therefore desirable for the operators of the field to have a thickener that allows to obtain concentrated

aqueous suspensions of inorganic pigments for paper coating having rheological and water retention characteristics that make them suitable for the application with film coaters and that do not appreciably exhibit the phenomena of misting and superficial defects.

[0020] It has now been found that these requirements are fully satisfied when a viscoelastic thickener of polysaccharidic origin, and particularly a carboxymethyl cellulose (CMC), is used as additive for the formulation of coating colour.

[0021] Although synthetic thickeners with viscoelastic behaviour are available on the market, they do not fully satisfy the water retention and thickening requirements which are necessary for this application.

[0022] In the present text, with the expression "viscoelastic thickener" we mean a thickener that exhibits an elastic modulus G' higher than the viscous module G" at least till 10% of angular amplitude (strain) in amplitude sweep test, when the amplitude sweep test is carried out at a concentration of 1% by weight in water with a Rheometer, at % strain from 0.01 to 1000, 1 Hz, 25°C and with a geometry of 60 mm 1° steel cone. The term polysaccharide is meant to include natural polysaccharides and their derivatives, such as, by way of example, alkyl-, carboxyalkyl-, hydroxyalkyl- and alkylhydroxyalkyl-polysaccharides.

Summary of invention

[0023] It is therefore a fundamental object of the present invention the use of a polysaccharide, and particularly of a carboxymethyl cellulose, that behaves as a viscoelastic thickener ("viscoelastic polysaccharide") for the preparation of aqueous suspensions of inorganic pigments useful for the coating of paper with film coaters.

[0024] It is another object of the present invention an aqueous suspension useful for the coating of paper containing: a) from 50 to 80% by weight of an inorganic pigment; b) from 0.05 to 3.0 parts by weight each 100 parts by weight of inorganic pigment of a polysaccharide, and particularly of a carboxymethyl cellulose, having an elastic modulus G' higher than the viscous module G" at least till 10% of strain in amplitude sweep test, the amplitude sweep test being carried out at a concentration of 1% by weight in water with a Rheometer, at % strain from 0.01 to 1000, 1 Hz, 25°C and with a geometry of 60 mm 1° steel cone; c) at least 15% by weight of water.

[0025] The use of the aforesaid aqueous suspension in the coating of paper with film coaters is a further object of the present invention.

Brief description of drawings

[0026] Fig. 1. Viscosimetric behavior in amplitude sweep of the Suspension (1) of the Examples formulated with a viscoelastic CMC (according to the invention).

[0027] Fig.2. Viscosimetric behavior in amplitude sweep of the Suspension (2) of the Examples formulated with non viscoelastic CMC (comparative).

[0028] Fig.3. Viscosimetric behavior in amplitude sweep of the Suspension (3) of the Examples formulated with an acrylic thickener in inverse emulsion (comparative).

[0029] Fig.4. Viscosimetric behavior in amplitude sweep of the Suspension (4) of the Examples formulated with an acrylic ASE type thickener (comparative).

Description of embodiments

[0030] The aqueous suspension for the coating of the paper according to the invention preferably contains from 0.1 to 1.5 parts by weight of polysaccharide, and particularly the carboxymethyl cellulose, each 100 parts by weight of pigment and from 20 to 40% by weight of water.

[0031] The polysaccharide, and particularly the carboxymethyl cellulose, which characterizes the coating suspensions of the present invention has Brookfield® viscosity at 1% by weight, 25°C and 60 rpm comprised between 20 and 15,000 mPa*s, preferably from 2,000 to 12,000 mPa*s.

[0032] Preferably, the thickener of the invention is a viscoelastic carboxymethyl cellulose having an elastic modulus G' higher than the viscous module G" till 50%, even more preferably till 200% of strain, measured in amplitude sweep tests in the above conditions.

[0033] Viscoelastic CMC can be prepared, by way of example, following the procedure described in DD 233 377; EP 1025130; Macromolecules, 22, 364-366, 1989 or Polymer, 39, 3155-3165, 1998.

[0034] The degree of substitution of the useful viscoelastic CMC, i.e. the average number of carboxymethyl groups for glucosidic ring, can vary between 0.4 and 1.7, preferably between 0.5 and 1.2.

[0035] It should be underlined that, although some syntheses of viscoelastic CMC are reported in the literature, the CMCs on the market normally do not have viscoelastic behavior; moreover, the few publications describing the preparation of viscoelastic CMC do not suggest its use in coating colours. Suitable inorganic pigments for the preparation of the aqueous suspensions of the present invention are those commonly used in the coating of paper, such as kaolin, calcium

carbonate, talc, titanium dioxide, barium sulfate, gypsum, and mixture thereof.

[0036] The particle dimension of the pigments is normally below 2 μm for 40 to 99% of their weight.

[0037] The aqueous suspension for the coating of paper according to the invention preferably also contains from 3 to 15 parts by weight of a polymeric binder each 100 parts by weight of pigment.

[0038] Suitable polymeric binders are those normally used in the field; we mention, by way of example, polymers of acrylic or methacrylic acid esters, co-polymer of acrylic monomers with vinyl acetate, styrene, butadiene, acrylonitrile or mixtures thereof, and the polymers based on styrene - butadiene - acrylonitrile.

[0039] The aqueous suspensions of the present invention shall have Brookfield® viscosity at 25° C and 100 rpm comprised between 100 and 3,500 mPa*s, preferably between 300 and 2,500 mPa*s; furthermore, they shall have an elastic modulus G' higher than the viscous module G" at least up to 10% of strain in amplitude sweep test, when the amplitude sweep test is carried out with a Rheometer, at % strain from 0.01 to 1000, 1 Hz, 25°C and with a geometry of 60 mm 1° steel cone.

[0040] According to the present invention, the viscoelastic polysaccharide, and particularly the viscoelastic CMC, can be used either alone or in the presence of other traditional thickeners, such as non viscoelastic CMC, starch and starch derivatives, ASE, HASE; the amount of additional traditional thickeners should obviously not be so high to modify the viscoelastic rheological behavior of the aqueous suspensions.

[0041] The Brookfield ® viscosity and the viscoelastic behavior of the aqueous suspensions are obtainable by properly dosing the thickeners, and particularly the viscoelastic carboxymethyl cellulose, taking into account its viscoelasticity and Brookfield ® viscosity, and the inorganic pigment concentration, as it will be apparent to the person skilled in the art.

[0042] The aqueous suspensions for paper coating of the invention may also contain other additives typically used in the field, such as surfactants, such as ethoxylated fatty alcohols, ethoxylated fatty acids, polyethoxysorbitan monolaurate, sodium laurylsulfate.

[0043] Lubricants, such as micronized calcium stearate, can be also be present in the compositions.

[0044] Biocides, such as the formulations based on BIT (1,2-benzoisothiazolinone), are another example of additives that can be used in the suspensions, particularly when they are to be stored for a long time.

[0045] The aqueous suspensions for paper coating of the invention are prepared by addition of the binder to the aqueous pigment suspension, followed by addition of the thickener and of the other additives and final regulation of the water amount.

[0046] It has been observed that the compositions for coating colour of the present invention are stable over the time.

[0047] Moreover, rheological analysis simulating the conditions of an industrial equipment operating with film coaters show that the coating formulations prepared according to the present invention exhibit a viscoelastic behavior (measures in amplitude sweep) that is analogous to the behavior of suspensions prepared with the inverse emulsion acrylic polymers already used in the industry.

[0048] On the contrary, products like ASE polymers or traditional non viscoelastic CMC are not suitable for the industrial use with film coaters: industrial tests showed difficulties in maintaining the correct amount of coating colour applied on the paper and release of drops of coating colour on the paper.

[0049] Examples

[0050] A base coating colour is prepared using the following products: Carbital 90 (slurry of calcium carbonate in water, 78% by weight, with a particle size 90% < 2μm, commercialized by Imeris) Dow Latex 935 (DL 935, latex SBR in aqueous emulsion, 50% by weight, commercialized by DOW) in the amounts (by weight) reported in Table 1 and by the following procedure: 1539 g of Carbital 90 are stirred at 1000 rpm by means ofa "cowless" mechanical stirrer; 240 g of Dow Latex 935 are added to the carbonate slurry; under continuous stirring 134 g of water are introduced to obtain a coating colour with solid content of about 69%.

TABLE 1. "Base coating colour" formulation

Products	Parts	Dry weight (g)	Weight (g)
CARBITAL 90	100	1200	1539
DL 935	10	120	240
WATER			134
solid content	69.00%		

[0051] The so obtained "base coating colour" is homogenized for 30 minutes at 1000 rpm with the cowless; the "base coating colour" is divided into four portions and in each portion one of the following thickener is dosed to obtain a final viscosity of 1300-1350 mPa*s (100 rpm):

- Carbocel MB2/C = low viscosity purified carboxymethyl cellulose, sodium salt, commercialized by Lamberti S.p.A.
- Viscolam VP = inverse emulsion acrylic thickener with active content 40%, commercialized by Lamberti S.p.A.
- Sipacril 2739 = direct emulsion (ASE) acrylic thickener with active content 29%, commercialized by Lamberti S.p.A.
- FUJ3017-67 = viscoelastic carboxymethyl cellulose with substitution degree 0.82, Brookfield® viscosity at 1% in water (25 °C, 60 rpm) 9,000 mPa*s, G' > G" up to 63% of strain in amplitude sweep test, at % strain from 0.01 to 1000, 1 Hz, 25°C and with a geometry of 60 mm 1° steel cone.

[0052] In order to prepare the first suspension, Suspension (1), 0.51 g of FUJ3017-67 are added to 478 g of base coating colour, under vigorous stirring. The dispersion is stirred by cowless at 1000 rpm for 30'. After 30' the pH is brought to 9 with NaOH.

[0053] The second suspension, Suspension (2) (comparative), is prepared like the Suspension (1), replacing FUJ3017-67 with a non viscoelastic CMC (Carbocel MB2C).

[0054] The third suspension, Suspension (3) (comparative), is prepared like Suspension (1), replacing FUJ3017-67 with 1.5 g of Viscolam VP.

[0055] The fourth suspension, Suspension (4) (comparative), is prepared like Suspension (1), replacing FUJ3017-67 with 2 g of Sipacril 2739.

[0056] In Table 2 the compositions, the solid content (%), the final pH and the Brookfield® viscosity at 25°C 100 rpm of the thus prepared suspensions are reported.

[0057] The rheological behavior of the Suspensions (1) - (4) has been evaluated with a AR 2000 Rheometer (TA Instruments) in amplitude sweep tests carried out at % strain from 0.01 to 1000, 1 Hz, 25°C and with a geometry of 60 mm 1° steel cone.

[0058] The relative graphs are reported in Figures 1-4; the suspension of the present invention shows an elastic behavior which is different from that of the suspensions comprising the ASE polymer and the non viscoelastic CMC and analogous to the behavior imparted by the inverse emulsion acrylic thickener.

TABLE 2 - COATING COLOUR FORMULATIONS

Ingredients/Suspensions	(1)	(2)	(3)	(4)
CARBITAL 90 (active matter)	100	100	100	100
DL 935 (active matter)	10	10	10	10
FUJ3017-67 (parts)	0.17	/	/	/
CARBOCEL MB2C (parts)	/	0.40	/	/
VISCOLAM VP (active matter)	/	/	0.20	/
SIPACRIL 2739 (active matter)	/	/	/	0.20
Solid content (%)	69.17	69.10	69.07	69.28
pH	9.03	9.06	9.00	9.03
Brookfield® viscosity100 rpm (mPa*s)	1350	1310	1300	1350

[0059] Only Suspension (1) is suitable for the application by means of film coater and during the application does not produce "misting"; only the paper obtained with Suspension (1) does not show defects (orange peel).

Claims

1. Aqueous suspension containing: a) from 50 to 80% by weight of an inorganic pigment; b) from 0.05 to 3.0 parts by weight each 100 parts by weight of inorganic pigments of a carboxymethyl cellulose having an elastic modulus G' higher than the viscous module G" at least till 10% of strain at a concentration of 1% by weight in water in amplitude sweep test performed at % strain from 0.01 to 1000, 1 Hz, 25°C and with a geometry of 60 mm 1° steel cone; c) at least 15% by weight of water.

2. Aqueous suspension according to claim 1., having Brookfield® viscosity at 25° C and 100 rpm comprised between 100 and 3,500 mPa*s and elastic modulus G' higher than the viscous module G" at least till 10% of strain in amplitude sweep test performed at % strain from 0.01 to 1000, 1 Hz, 25°C and with a geometry of 60 mm 1° steel cone.

3. Aqueous suspension for the coating of paper according to claim 1. or 2. containing from 0.1 to 1.5 parts by weight of carboxymethyl cellulose each100 parts by weight of inorganic pigment and from 20 to 40% by weight of water.

4. Aqueous suspension according to claim 3., wherein the carboxymethyl cellulose has an elastic modulus G' higher than the viscous module G" till 50% of strain in the amplitude sweep test at a concentration of 1% by weight in water.

5. Aqueous suspension according to claim 4., wherein the carboxymethyl cellulose has an elastic modulus G' higher than the viscous module G" till 200% of strain in the amplitude sweep test at a concentration of 1% by weight in water.

6. Aqueous suspension according to claim 5., wherein the carboxymethyl cellulose has Brookfield® viscosity at 1% by weight, 25°C and 60 rpm, from 20 to 15,000 mPa*s.

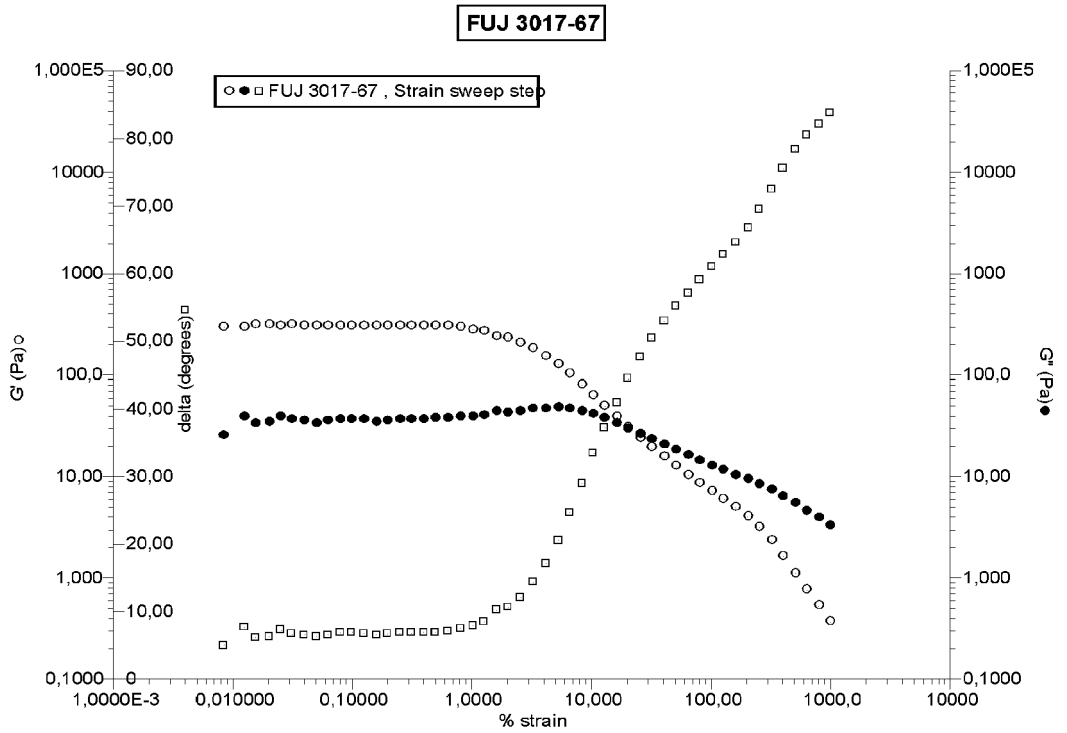
7. Aqueous suspension according to claim 5., wherein the carboxymethyl cellulose has Brookfield® viscosity at 1% by weight, 25°C and 60 rpm, from 2,000 to 12,000 mPa*s.

8. Use of an aqueous suspension according to any of the claims from 1. to 7. for the coating of paper by means of film coaters.

9. Method for the preparation of aqueous suspensions of inorganic pigments for the coating of paper comprising the use of a carboxymethyl cellulose having an elastic modulus G' higher than the viscous module G" at least up to 10% of strain in amplitude sweep test performed at % strain from 0.01 to 1000, 1 Hz, 25°C and with a geometry of 60 mm 1° steel cone at a concentration of 1% by weight in water.

10. Method for the preparation of aqueous suspensions of inorganic pigments for the coating of paper according to claim 9., wherein the carboxymethyl cellulose has an elastic modulus G' higher than the viscous module G" till 50% of strain in the amplitude sweep test at a concentration of 1% by weight in water.

11. Method for the preparation of aqueous suspensions of inorganic pigments for the coating of paper according to claim 10., wherein the carboxymethyl cellulose has an elastic modulus G' higher than the viscous module G" till 200% of strain in the amplitude sweep test at a concentration of 1% by weight in water.


FUJ 3017-67
FUJ 3017-67 , Strain sweep step
G' (Pa)
delta (degrees)
G" (Pa)
% strain
1,000E5
10000
1000
100,0
10,00
1,000
0,1000
90,00
80,00
70,00
60,00
50,00
40,00
30,00
20,00
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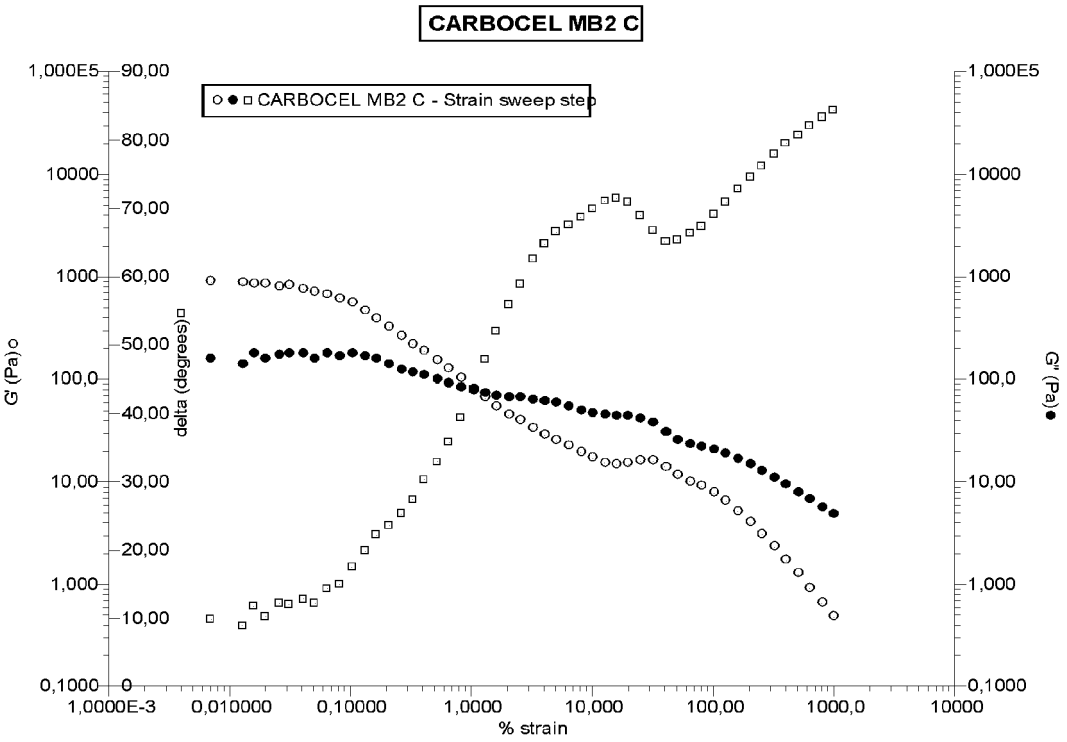
0,10000
1,0000
10,000
100,00
1000,0
10000

Fig. 1


CARBOCEL MB2 C
CARBOCEL MB2 C - Strain sweep step
G' (Pa)
delta (degrees)
G" (Pa)
% strain
1,000E5
10000
1000
100,0
10,00
1,000
0,1000
90,00
80,00
70,00
60,00
50,00
40,00
30,00
20,00
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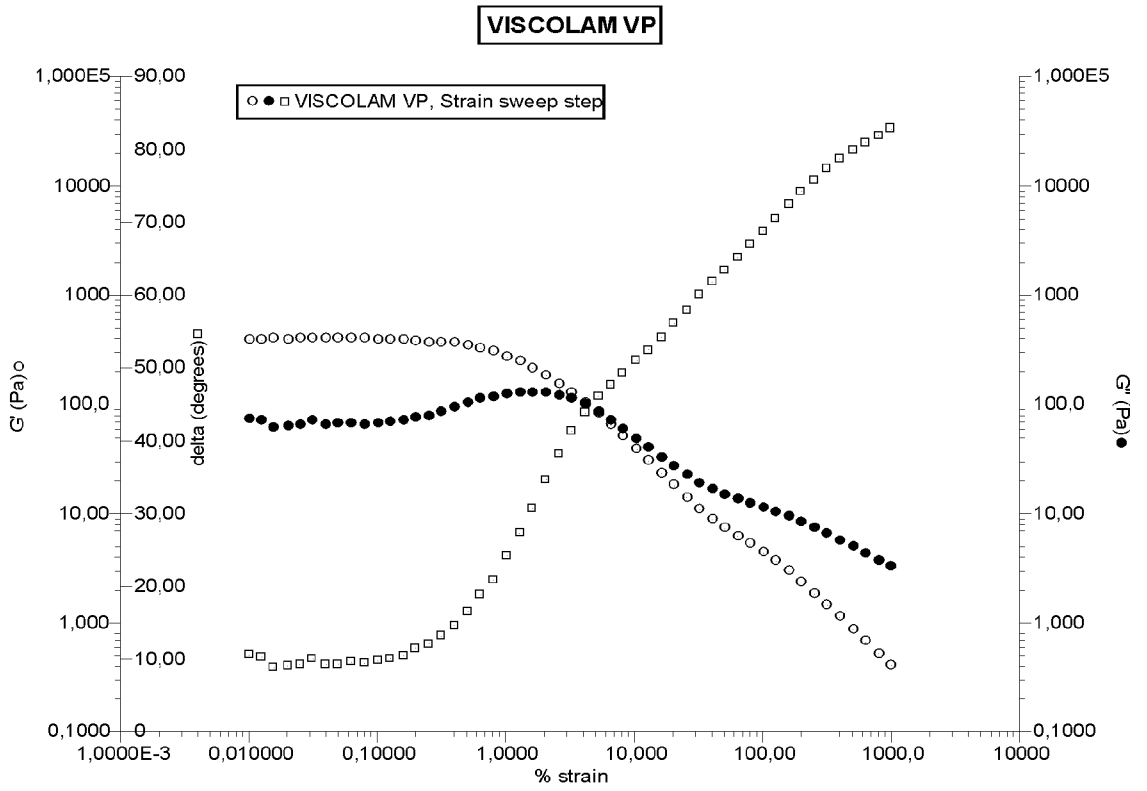
10,000
100,00
1000,0
10000

Fig. 2


VISCOLAM VP
VISCOLAM VP, Strain sweep step
G' (Pa)
delta (degrees)
G" (Pa)
% strain
1,000E5
10000
1000
100,0
10,00
1,000
0,1000
90,00
80,00
70,00
60,00
50,00
40,00
30,00
20,00
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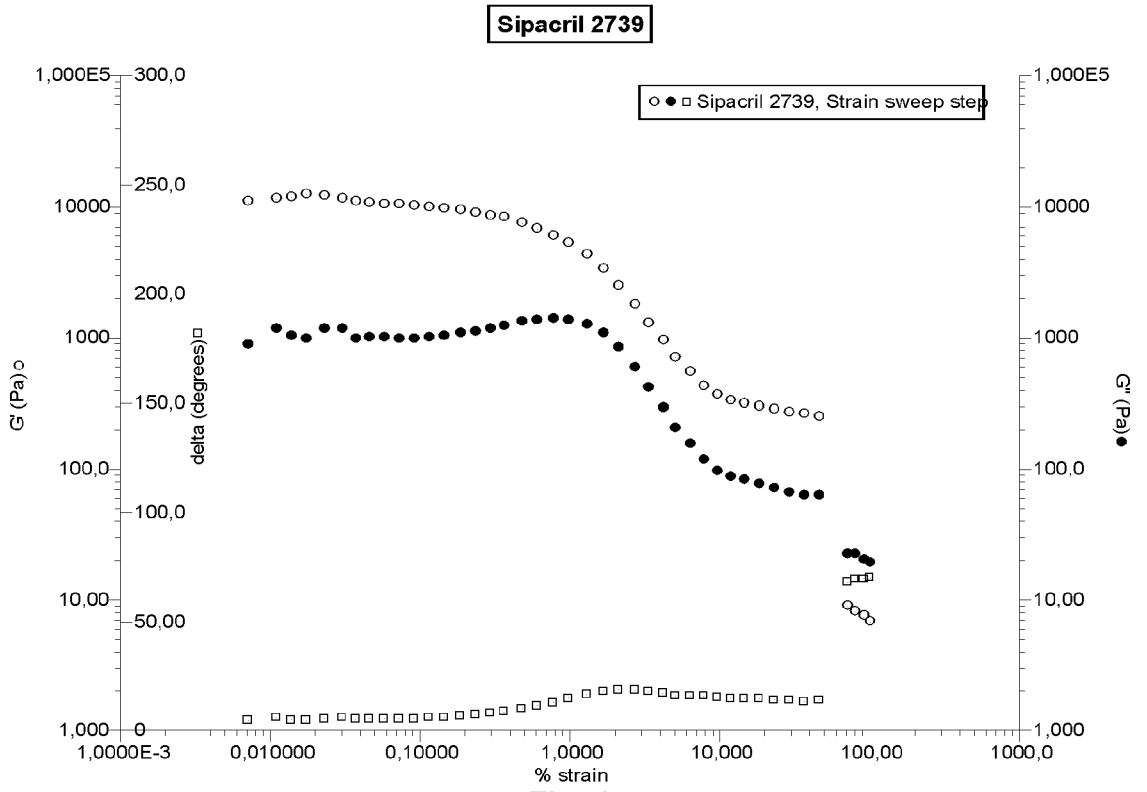
1,0000
10,000
100,00
1000,0
10000

Fig. 3


Sipacril 2739
Sipacril 2739, Strain sweep step
G' (Pa)
delta (degrees)
G" (Pa)
% strain
1,000E5
10000
1000
100,0
10,00
1,000
300,0
250,0
200,0
150,0
100,0
50,00
0
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1000,0

Fig. 4

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EUROPEAN SEARCH REPORT

Application Number
EP 10 15 3563

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (IPC)
X	US 2006/102304 A1 (NUTBEEM CHRISTOPHER [GB] ET AL) 18 May 2006 (2006-05-18) * page 1, paragraph 2 * * page 2, paragraphs 14,19 * * page 6, paragraph 88 * * page 7, paragraphs 97,101 * * page 8, paragraph 111-117 *	1-11	INV. D21H19/52 D21H19/54 D21H21/10
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A	US 5 725 648 A (BROWN MICHAEL J [US] ET AL) 10 March 1998 (1998-03-10) * column 1, line 59 - column 2, line 9 * * column 2, lines 18-25 * * column 3, line 65 - column 4, line 2 *	1-11	
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The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
Munich	3 May 2010	Beins, Ulrika

CATEGORY OF CITED DOCUMENTS

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03-05-2010

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

REFERENCES CITED IN THE DESCRIPTION

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