(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau

(43) International Publication Date
28 August 2014 (28.08.2014)

WIPO | PCT

(10) International Publication Number
WO 2014/128171 A2

(51) International Patent Classification: Not classified

(21) International Application Number:
PCT/EP2014/053248

(22) International Filing Date:
19 February 2014 (19.02.2014)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
1351548 22 February 2013 (22.02.2013) FR
61/773,198 6 March 2013 (06.03.2013) US

(71) Applicant: **L'OREAL** [FR/FR]; 14, rue Royale, F-75008 Paris (FR).

(72) Inventors: **PORTAL, Julien**; 7 Allée de la Main Ferme, F-93320 Les Pavillons sous Bois (FR). **HERNANDEZ, Franck**; 56 bis allée du centre, F-93250 Villemomble (FR). **SCHULTZE, Xavier**; 43 Allée Robillard, F-93320 Les Pavillons Sous Bois (FR).

(74) Agent: **KROMER, Christophe**; L'Oréal, D.I.P.I., 25-29 Quai Aulagnier, F-92665 Asnières-sur-Seine Cedex (FR).

(81) Designated States *(unless otherwise indicated, for every kind of national protection available)*: AE, AG, AL, AM, AO, AT, AU, AZ, BA, BB, BG, BH, BN, BR, BW, BY, BZ, CA, CH, CL, CN, CO, CR, CU, CZ, DE, DK, DM, DO, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT, HN, HR, HU, ID, IL, IN, IR, IS, JP, KE, KG, KN, KP, KR, KZ, LA, LC, LK, LR, LS, LT, LU, LY, MA, MD, ME, MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ, OM, PA, PE, PG, PH, PL, PT, QA, RO, RS, RU, RW, SA, SC, SD, SE, SG, SK, SL, SM, ST, SV, SY, TH, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, ZA, ZM, ZW.

(84) Designated States *(unless otherwise indicated, for every kind of regional protection available)*: ARIPO (BW, GH, GM, KE, LR, LS, MW, MZ, NA, RW, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, RU, TJ, TM), European (AL, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HR, HU, IE, IS, IT, LT, LU, LV, MC, MK, MT, NL, NO, PL, PT, RO, RS, SE, SI, SK, SM, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, KM, ML, MR, NE, SN, TD, TG).

Published:

— *without international search report and to be republished upon receipt of that report (Rule 48.2(g))*

WO 2014/128171 A2

(54) Title: COSMETIC COMPOSITION COMPRISING AN EXTRUDED POLYSACCHARIDE

(57) Abstract: The invention relates to a process for the preparation of a cosmetic composition obtained by kneading, at a temperature of between 100°C and 250°C, a premix comprising an ethanol-insoluble polysaccharide, an ethanol-soluble polymer and a polycarboxylic acid comprising from 4 to 8 carbon atoms, by then introducing the extrudate formed, cooled beforehand, into water or a water/ethanol mixture, and then recovering the filtrate. Application to the cosmetic treatment of the hair, in particular for the fixing or shaping of the hair.

Cosmetic composition comprising an extruded polysaccharide

The invention relates to a cosmetic composition comprising the water-soluble fraction of an extrudate comprising a polysaccharide, and also to a method for the treatment of the hair, in particular for its shaping, using such a composition

In the styling field, in particular for products intended for the shaping and/or form retention of the hairstyle, the consumer is looking for styling products exhibiting good fixing properties.

The most widespread hair compositions on the cosmetic market are compositions essentially composed of a solution, generally an alcoholic or aqueous/alcoholic solution, and of film-forming polymers (fixing components), the role of which is to form joins between the keratinous fibers.

For hair products in the form of sprays (pump-action sprays and aerosols), the quality of the spray (diffuse, broad and covering) greatly conditions the nature of the joins created between the hairs, the homogeneity of the depositions, the fixing properties and the feel.

Very few polymers of natural origin are sufficiently fixing and conveyable in water or a water/ethanol mixture to be able to be used in hair compositions for shaping the hair.
Some polysaccharides, such as starch or gum arabic, are not soluble in ethanol and, when they are conveyed in water, they form excessively viscous gels inappropriate for being packaged in the spray or aerosol lacquer form. Nevertheless, these polysaccharides exhibit advantageous hair fixing properties.

The aim of the present invention is thus to have available styling products exhibiting good hair fixing properties, based on polymer of natural origin, which are conveyed in an aqueous medium or in a water/ethanol medium and which can be dispensed in the spray form.

The applicant company has demonstrated that, by employing a specific preparation process using an ethanol-insoluble polysaccharide in the presence of an ethanol-soluble polymer and of a specific dicarboxylic acid, it is possible to obtain a hair cosmetic composition which is perfectly suitable for fixing the hair. The composition obtained is also well suited to being dispensed in the spray form and can thus be packaged as a pump-action spray or as an aerosol.

A subject matter of the present invention is thus a process for the preparation of a cosmetic composition comprising the following stages:
a) preparation of a premix comprising:
an ethanol-insoluble polysaccharide;

an ethanol-soluble polymer;
a polycarboxylic acid comprising from 4 to 8 carbon atoms or the anhydride of said acid;
and optionally a plasticizing agent;

b) said premix is kneaded under shearing, at a temperature between 100°C and 250°C, in order to form an extrudate;

c) cooling the extrudate to a temperature of less than 100°C;

d) introducing the cooled extrudate into water or into a water/ethanol mixture comprising from 0% to 75% (or 0.1% to 75%) by weight of ethanol;

e) recovering the filtrate obtained.

The composition thus obtained according to the preparation process comprises, in an aqueous or aqueous/alcoholic medium, the fraction soluble in water at ambient temperature (25°C) of the extrudate obtained according to stage b). This water-soluble fraction corresponds to the isolated filtrate obtained according to stage e) of the preparation process.

Another subject matter of the invention is a cosmetic composition capable of being obtained according to the preparation process described above.

Another subject matter of the invention is a method for the cosmetic treatment of the hair, comprising the application to the hair of a cosmetic composition as described above. The method is in particular a method for fixing and/or shaping the hair.

The premix (also known as extrusion premix) comprises an ethanol-insoluble polysaccharide.
The term “ethanol-insoluble polysaccharide” is understood to mean a polysaccharide for which the solubility in ethanol at 25°C is less than 1% by weight.

The ethanol-insoluble polysaccharide can be chosen from starch, cellulose, guar gum, gum arabic (INCI name: acacia senegal gum), alginic acid, alginate salts (in particular sodium, calcium and zinc salts) and carrageenans. Preferably, the ethanol-insoluble polysaccharide is chosen from starch or gum arabic. Preferably, the ethanol-insoluble polysaccharide is starch.

Advantageously, the ethanol-insoluble polysaccharide is a fixing polymer. The term "fixing polymer" is understood to mean a polymer capable, by application to the hair, of conferring a shape on the head of hair or of making possible the form

retention of a shape already acquired.

Preferably, the ethanol-insoluble polysaccharide has a weight-average molecular weight ranging from 10 000 to 2 000 000 g/mol.

The ethanol-insoluble polysaccharide can be present in the premix in a content ranging from 55% to 85% by weight, preferably ranging from 60% to 80% by weight, with respect to the total weight of the premix.

The premix also comprises an ethanol-soluble polymer.
The term "ethanol-soluble polymer" is understood to mean a polymer having a solubility of at least 5% by weight in ethanol at 25°C.

Advantageously, the ethanol-soluble polymer is a fixing polymer.

The ethanol-soluble polymer can be chosen from ethylcellulose, hydroxypropylcellulose, hydroxyethyl ethylcelluloses (for example Bermocoll E 481 FQ from Akzo Nobel), carboxymethylethylcelluloses and their sodium salts, carboxymethylhydroxyethylcelluloses and their sodium salts, polyvinyl alcohol, poly(C_2-C_4 hydroxyalkyl (meth)acrylate)s, such as poly(2-hydroxyethyl methacrylate) or poly(2-hydroxyethyl acrylate), polyvinylpyrrolidone or poly(2-ethyl-2-oxazoline).

Hydroxypropylcellulose is preferably used.

Preferably, the ethanol-soluble polymer has a weight-average molecular weight ranging from 10 000 to 2 000 000 g/mol.

The ethanol-soluble polymer can be present in the premix in a content ranging from 10% to 40% by weight, preferably ranging from 10% to 30% by weight, preferably ranging from 10% to 20% by weight, with respect to the total weight of the premix.

The presence of the ethanol-soluble polymer and in particular of hydroxypropylcellulose makes it possible to carry out the extrusion stage b) (kneading under shearing) at a temperature between 100°C and 250°C, as is shown in the comparative examples described below. This stage would be impossible to carry out in the absence of this ethanol-soluble polymer and would require the use of a conventional plasticizing agent, such as glycerol or sorbitol, in order to be able to extrude the polysaccharide, in particular starch, under these conditions without bringing about its deterioration.

The premix also comprises a polycarboxylic acid comprising from 4 to 8 carbon atoms or the anhydride of said acid. The polyacid can be a dicarboxylic acid or a tricarboxylic acid. Said polyacid can be hydroxylated. Preferably, the polycarboxylic acid comprises from 4 to 6 carbon atoms.

The polycarboxylic acid or its anhydride can be chosen from maleic acid, maleic anhydride, succinic acid, succinic anhydride, itaconic acid, itaconic anhydride, tartaric acid, phthalic acid, phthalic anhydride or citric acid. Preferably, the polycarboxylic acid is chosen from maleic acid, maleic anhydride or tartaric acid.

The polycarboxylic acid can be present in the premix in a content ranging from 0.1% to 30% by weight and preferably ranging from 1% to 20% by weight, with respect to the total weight of the premix.

Advantageously, the extrusion premix can comprise a plasticizing agent. Alternatively, the plasticizing agent can be added during the kneading stage.
The plasticizing agent makes it possible to lower the melting point of the polysaccharide. It thus makes possible easy processing of the polysaccharide in the process for the preparation of the cosmetic composition according to the invention Advantageously, the plasticizing agent is liquid at ambient temperature (25°C) or else is solid and, in this case, has a melting point of less than 100°C.

The plasticizing agent can be chosen from:
glycerol, ethylene glycol, propylene glycol, 1,4-propanediol, 1,2-butanediol, 1,3-butanediol, 1,4-butanediol, 1,5-pentanediol, 1,5-hexanediol, 1,6-hexanediol, 1,2,6-hexanetriol, 1,3,5-hexanetriol, neopentyl glycol, polyethylene glycols having a molecular weight ranging from 200 to 1500 g/mol, polypropylene glycols having a molecular weight ranging from 200 to 1500 g/mol, sorbitol (solid), sorbitol monoacetate, sorbitol diacetate, sorbitol monoethoxylate, sorbitol diethoxylate, sorbitol hexaethoxylate, sorbitol dipropoxylate, aminosorbitol, 2-amino-2-(hydroxymethyl)propane-1,3-diol, trimethylolpropane monoethoxylate, mannitol monoacetate, mannitol monoethoxylate, butylglucoside, glucose monoethoxylate, α-methylglucoside, sodium carboxymethylsorbitol, polyglycerol (2 to 10 glycerol units) monoethoxylate, erythritol, pentaerythritol, arabitol, adonitol, xylitol, mannitol, iditol, galactitol, allitol, glycerol esters, mono- and diglycerides, trimethylol propane, polyvinyl alcohol having from 3 to 20 repeat units, polyglycerols having from 2 to 10 repeat units, and their mixtures.

Preferably, the plasticizing agent is chosen from glycerol and sorbitol. Preferably, the plasticizing agent is glycerol.

The plasticizing agent can be present in the extrusion premix or added in the kneading stage in a content ranging from 0% (or 0.1%) to 39.9% by weight, preferably ranging from 0% (or 0.1%) to 30% by weight, preferably ranging from 1% to 20% by weight and more preferably ranging from 2% to 15% by weight, with respect to the total weight of the premix.

According to a preferred embodiment of the invention, the extrusion premix comprises:
60% to 75% by weight of starch,

15% to 20% by weight of hydroxypropylcellulose,
6% to 12% by weight of tartaric acid,
5% to 15% by weight of glycerol,
with respect to the total weight of said premix.

According to another preferred embodiment of the invention, the extrusion premix comprises:
65% to 80% by weight of starch,
15% to 20% by weight of hydroxypropylcellulose,
2% to 5% by weight of maleic anhydride,
3% to 10% by weight of glycerol,
with respect to the total weight of said premix.

According to one embodiment of the preparation process according to the invention, the process can comprise a stage of addition of the plasticizing agent during the kneading stage b). Thus, the plasticizing agent can be introduced during the kneading stage and not before the kneading stage.

The preparation process according to the invention can comprise a stage of grinding the extrudate obtained after the kneading stage. The grinding can be carried out by any known means which makes it possible to convert the extrudate into a powder.

The kneading stage b) of the preparation process described above can be carried out using a screw mixer/extruder.
It is understood that any other equivalent technical means for carrying out this kneading stage can be used.

The premix is kneaded under shearing in order to create, in the body, regions of crushing the premix.

Preferably, the kneading stage is carried out with a screw mixer/extruder. Such a kneading process is described in particular in the document EP-A-0 667 146.

The screw of the mixer/extruder can rotate at a speed ranging from 1 to 1000 revolutions/minute, preferably ranging from 1 to 250 revolutions/minute.

According to a specific form, use is made of extruders of twin-screw mixer/extruder type, these extruders being composed of the following elements:
- at least one or more barrels independently thermally regulated at a temperature ranging from 100°C to 300°C,

- two corotating shafts composed of screw elements, each element having a shape which provides the desired mixing function in the corresponding temperature zone,
- devices for metering out and introducing the various constituents of the premix,
- a variable-speed motor, which makes it possible to adjust the intensity of the shearing as a function of the rotational speed of the screws.

Mention may in particular be made, as items of equipment which make it possible to implement the invention, without limiting the invention to these devices, of the BC-21 and BC-45 models from Clextral, or the Prism Eurolab model from ThermoScientifique, or else the microextruder from DSM.

The extrudate formed is subsequently cooled to a temperature of less than 100°C. Preferably, the extrudate formed is cooled to ambient temperature (25°C).

The cosmetic composition obtained according to the preparation process described above comprises the portion of the extrudate soluble at ambient temperature (25°C) in water or in a water/ethanol mixture comprising from 0% to 75% by weight of ethanol (or comprising up to 75% by weight of ethanol, or comprising from 0.1% to 75% by weight of ethanol), subsequently known as water-soluble fraction of the extrudate. It corresponds to the filtrate isolated after introduction of the extrudate into water or the water/ethanol mixture.
Additional cosmetic ingredients as described below can be added to the isolated filtrate.

The term “cosmetic composition” is understood to mean a composition compatible with keratinous substances, which exhibits a pleasant color, odor and feel and which does not cause unacceptable discomfort (stinging, tautness or redness) liable to turn the consumer away from it.

The term "keratinous substances" is understood to mean the skin (body, face, outline of the eyes, scalp), head hair, eyelashes, eyebrows, non-head hairs, nails or lips.

Advantageously, in the cosmetic composition according to the invention, the water-soluble fraction of the extrudate can be present in a content, as dry matter, ranging from 1% to 10% by weight and preferably ranging from 2% to 7% by weight, with respect to the total weight of the composition.

The composition according to the invention can comprise additional cosmetic ingredients chosen in particular from preservatives, fragrances, sunscreens, oils, waxes, thickeners, film-forming polymers, moisturizing agents, vitamins, proteins, ceramides, surfactants, antioxidants, agents for combating free radicals, colorants, antidandruff agents, chelating agents, propellants or fillers.

Of course, a person skilled in the art will take care to choose this or these optional additional compounds and/or their amounts so that the advantageous properties of the composition according to the invention are not, or not substantially, detrimentally affected by the envisaged addition.

Advantageously, the cosmetic composition according to the invention has a viscosity ranging from 0.001 to 3 Pa.s (1 to 3000 centipoises), preferably ranging from 0.01 to 1 Pa.s (10 to 1000 centipoises) and preferably ranging from 0.03 to 0.5 Pa.s (30 to 500 centipoises), measured using a Brookfield DV-I+ viscometer, at 25°C, with the SC4-31 spindle at the rotational speed of 25 revolutions/minute. Such a composition is suited to being packaged as a spray.

The composition according to the invention can be in the form of a lacquer, spray, gel, foam, cream or lotion.
The composition can be packaged in an aerosol.
In this case, it comprises one or more propellants. The propellant(s) are chosen from non-liquefied compressed gases, such as air, nitrogen, nitrous oxide, carbon dioxide or dimethyl ether, or else from liquefied gases, such as volatile C_3-C_5 hydrocarbons, for example n-butane, isobutane, propane, pentane and halogenated hydrocarbons, such as 1,1-difluoroethane, and their mixtures. Preferably, the propellant is chosen from dimethyl ether and a propane/n-butane/isobutane mixture.

The propellant can be present in the composition according to the invention in proportions preferably ranging from 0.1% to 60% by weight, more preferably from 1.5% to 50% by weight and better still from 2% to 30% by weight, with respect to the total weight of the composition.
In a first alternative embodiment, when the propellant is a volatile C_3-C_5 hydrocarbon, it can be present in the composition in proportions preferably ranging from 0.1% to 60% by weight, more preferably from 0.5% to 30% by weight and better still from 0.5% to 8% by weight, with respect to the total weight of the composition.
In a second alternative embodiment, when the propellant is a gas partially soluble in the composition, such as dimethyl ether or 1,1-difluoroethane, it can be present in the composition in proportions preferably ranging from 10% to 50% by weight, more preferably from 30% to 45% by weight, with respect to the total weight of the composition.
The aerosol device used to package the composition of the invention can be composed of an aerosol can equipped with a dispensing valve and an actuator.
The composition can also be packaged in a pump-action spray.

The examples below illustrate the invention without, however, limiting the scope thereof.

Examples 1 to 30:

The extrusion premix was prepared by mixing all of the ingredients (one-stage process). In another alternative form of the preparation process, the plasticizing agent was added during the stage of kneading the extrusion premix (2-stage process).

Several extrudates were prepared using a twin-screw extruder (microextruder from DSM), at an extrusion speed of 80 revolutions/minute, at a temperature of 170°C, by applying an extrusion force of between 200 and 6000 newtons (force indicated by the extruding device).

The solubility in an aqueous/alcoholic medium of each extrudate prepared was determined in the following way:
10 g of extrudate was introduced into 100 g of a 70/30 (ratio by weight) water/ethanol mixture. The mixture was stirred at ambient temperature for one hour and was then left standing for one hour without stirring. If the solution is clear, the extrudate is completely soluble in the aqueous/alcoholic medium. If the solution is not clear, the extrudate is partially soluble in the aqueous/alcoholic medium: the soluble fraction of the extrudate was then determined by filtering the solution and by determining, for the filtrate, the content of extrudate solids according to conventional techniques (in particular by gravimetric measurement). The closer the solids content to the value of 10%, the more soluble the extrudate in the aqueous/alcoholic medium.

A hair lotion was subsequently prepared with each extrudate, which lotion has the following composition:

Extrudate	5%
Ethanol	28.5%
Water	q.s. for 100%

The hair solution was packaged in a pump-action spray. The quality of the spray obtained was subsequently determined as a function of the width of the dispensed spray. The quality of the spray is regarded as good when the spray is broad and well covering the sprayed surface; it is poor when the spray is narrow, forms a highly directional jet and is not very covering.

The fixing qualities of the hair lotion were also determined by applying the spray 5 times to a lock of hair. After drying, it was evaluated whether the hair was or was not well fixed.

a) Extrudates of starch, hydroxypropylcellulose and maleic anhydride:

Compositions of the extrudates prepared (contents as percentage by weight):

Extrudate	Starch (1)	Hydroxypropylcellulose (2)	Maleic anhydride	Extrusion force (N)
1	78.4	19.6	2	4600
2	76.9	19.2	3.9	5400
3	75.4	18.9	5.7	5800

(1) potato starch, sold under the name ColdSwell 1111 by KMC

(2) hydroxypropylcellulose, sold under the name Klucel EF Pharm by Ashland.

Solubilities of the extrudates:

Extrudate	Solids content at 130°C
1	8
2	8.5
3	6.6

Cosmetic properties of the hair lotions:

Extrudate	Quality of the spray	Fixing of the treated locks
1	Very good	Very good
2	Very good	Very good
3	Very good	Very good

Extrudates 1 to 3 confer good properties of spray quality and of fixing of the treated locks of hair.

b) Extrudates of starch, hydroxypropylcellulose, maleic anhydride and glycerol:

Compositions of the extrudates prepared:

Extrudate	Starch	Hydroxy-propyl-cellulose	Maleic anhydride	Glycerol	Process	Extrusion force (N)
4	73.4	18.3	3.7	4.6	1 stage	3000
5	70.2	17.5	3.5	8.8	1 stage	2800
6	67.2	16.8	3.4	12.6	1 stage	2900
7	73.4	18.3	3.7	4.6	2 stages	3200
8	70.2	17.5	3.5	8.8	2 stages	3100
9	67.2	16.8	3.4	12.6	2 stages	2800

Solubilities of the extrudates:

Extrudate	Solids content at 130°C
4	9.5
5	9.8
6	9
7	9.1
8	7.8
9	8

The results obtained show that extrudates 4 to 7 have a greater solubility in the aqueous/alcoholic medium than those of extrudates 8 and 9.

Cosmetic properties of the hair lotions:

Extrudate	Quality of the spray	Fixing of the treated locks
4	Very good	Very good
5	Very good	Very good
6	Very good	Good
7	Very good	Very good
8	Very good	Very good
9	Very good	Good

Extrudates 4 to 9 confer good properties of spray quality and of fixing of the treated locks of hair.

Comparative example: extrudate 5' without hydroxypropylcellulose (outside the invention)

On the basis of extrudate 5, the hydroxypropylcellulose was deleted and substituted by glycerol, so as to be able to extrude the starch (1-stage process).

Composition of the extrudate 5' prepared:

Extrudate	Starch	Glycerol	Maleic anhydride	Temperature (°C)	Extrusion force (N)
5’	70.2	26.3	3.5	100	1400

Extrudate 5' is not 10% soluble in the 70/30 water/ethanol mixture and forms a precipitate. It does not make it possible to prepare a sprayable hair composition.

By comparison with extrudate 5 described above, this test shows that the absence of hydroxypropylcellulose results in a starch extrudate which is insoluble in an aqueous/alcoholic medium and does not make it possible to obtain a sprayable hair composition. Thus, the presence of hydroxypropylcellulose is necessary in

order to obtain a starch extrudate which is soluble in an aqueous/alcoholic medium and in order to produce a sprayable hair composition.

c) Extrudates of starch, hydroxypropylcellulose, maleic anhydride and sorbitol:

Compositions of the extrudates prepared:

Extrudate	Starch	Hydroxypropyl-cellulose	Maleic anhydride	Sorbitol	Process	Extrusion force (N)
10	73.4	18.3	3.7	4.6	1 stage	3200
11	70.2	17.5	3.5	8.8	1 stage	3100
12	67.2	16.8	3.4	12.6	1 stage	2800
13	73.4	18.3	3.7	4.6	2 stages	3400
14	70.2	17.5	3.5	8.8	2 stages	3000
15	67.2	16.8	3.4	12.6	2 stages	2900

Solubilities of the extrudates:

Extrudate	Solids content at 130°C
10	8.9
11	10
12	9.2
13	8
14	8.5
15	7.7

The results obtained show that extrudates 10 to 12 have a greater solubility in the aqueous/alcoholic medium than those of extrudates 13 to 15.

Cosmetic properties of the hair lotions:

Extrudate	Quality of the spray	Fixing of the treated locks
10	Very good	Very good
11	Very good	Very good
12	Very good	Good
13	Very good	Very good
14	Very good	Very good
15	Very good	Good

Extrudates 10 to 15 confer good properties of spray quality and of fixing of the treated locks of hair.

d) Extrudates of starch, hydroxypropylcellulose and tartaric acid:

Compositions of the extrudates prepared:

Extrudate	Starch	Hydroxypropylcellulose	Tartaric acid	Extrusion force (N)
16	78.4	19.6	2	2700
17	76.9	19.2	3.9	3000
18	75.4	18.9	5.7	3600
19	74.1	18.5	7.4	3700
20	72.1	18	9.9	3500
21	68.4	14.5	17.1	2200

Solubilities of the extrudates:

Extrudate	Solids content at 130°C
16	6.7
17	7.3
18	9.5
19	10
20	10
21	10

The results obtained show that extrudates 18 to 21 have a greater solubility in the aqueous/alcoholic medium than those of extrudates 16 and 17, due to a higher content of tartaric acid.

The aqueous/alcoholic solutions prepared with extrudates 16, 17 and 18 were filtered in order to obtain the hair lotion.

Cosmetic properties of the hair lotions:

Extrudate	Quality of the spray	Fixing of the treated locks
16	Very good	Very good
17	Very good	Very good
18	Very good	Very good
19	Very good	Very good
20	Very good	Very good
21	Very good	Very good

Extrudates 16 to 21 confer good properties of spray quality and of fixing of the treated locks of hair.

e) Extrudates of starch, hydroxypropylcellulose, tartaric acid and glycerol:

Compositions of the extrudates prepared:

Extrudate	Starch	Hydroxy-propyl-cellulose	Tartaric acid	Glycerol	Process	Extrusion force (N)
20	72.1	18	9.9	-	1 stage	3500
22	69	17.2	9.5	4.3	1 stage	1400
23	66.1	16.5	9.1	8.3	1 stage	600
24	63.5	15.9	8.7	11.9	1 stage	250
25	69	17.2	9.5	4.3	2 stages	1900
26	66.1	16.5	9.1	8.3	2 stages	1700
27	63.5	15.9	8.7	11.9	2 stages	700

Solubilities of the extrudates:

Extrudate	Solids content at 130°C
20	10
22	9.8
23	9.7
24	9
25	10
26	9.8
27	9.6

The results obtained show that extrudates 20 to 27 have a very good solubility in the aqueous/alcoholic medium.

Cosmetic properties of the hair lotions:

Extrudate	Quality of the spray	Fixing of the treated locks
20	Very good	Very good
22	Very good	Very good
23	Very good	Very good
24	Very good	Good
25	Very good	Very good
26	Very good	Very good
27	Very good	Good

Extrudates 20 to 27 confer good properties of spray quality and of fixing of the treated locks of hair.

<u>Comparative example: extrudate 22' without hydroxypropylcellulose (outside the invention)</u>

On the basis of extrudate 22, the hydroxypropylcellulose was deleted and substituted by glycerol, so as to be able to extrude the starch (1-stage process).

Composition of the extrudate 22' prepared:

Extrudate	Starch	Glycerol	Tartaric acid	Temperature (°C)	Extrusion force (N)
22'	69	24.3	9.5	80	1500

Extrudate 22' is not 10% soluble in the 70/30 water/ethanol mixture and forms a precipitate. It does not make it possible to prepare a sprayable hair composition.

By comparison with extrudate 22 described above, this test shows that the absence of hydroxypropylcellulose results in a starch extrudate which is insoluble in an aqueous/alcoholic medium and does not make it possible to obtain a sprayable hair composition. Thus, the presence of hydroxypropylcellulose is necessary in order to obtain a starch extrudate which is soluble in an aqueous/alcoholic medium and in order to produce a sprayable hair composition.

f) Extrudates of starch, hydroxypropylcellulose and itaconic anhydride:

Compositions of the extrudates prepared:

Extrudate	Starch	Hydroxypropyl-cellulose	Itaconic anhydride	Extrusion force (N)
28	78.4	19.6	2	4500
29	76.9	19.2	3.9	5400
30	72.1	18	9.9	5900

Solubilities of the extrudates:

Extrudate	Solids content at 130°C (%)
28	6.1
29	6.3
30	6.8

The results obtained show that the solubility of extrudates 28 to 30 in an aqueous/alcoholic medium is lower than that of extrudates 1 and 16, respectively prepared with maleic anhydride or tartaric acid in place of the itaconic anhydride.

The aqueous/alcoholic solutions prepared with extrudates 28 to 30 were filtered in order to obtain the hair lotion.

Cosmetic properties of the hair lotions:

Extrudate	Quality of the spray	Fixing of the treated locks
28	Very good	Very good
29	Very good	Very good
30	Very good	Very good

Extrudates 28 to 30 confer good properties of spray quality and of fixing of the treated locks of hair.

Example 31 (outside the invention): non-extruded starch

The solubility properties of the non-extruded starch were evaluated in water and in a 70/30 water/ethanol mixture.

The 14% aqueous starch solution exists in the form of a viscous gel.
The 10% aqueous/alcoholic starch solution exists in the form of a white, unstable and non-filterable viscous solution; a significant white deposit is formed at the bottom of the flask.

The 2 solutions prepared, packaged in a pump-action spray, are not sprayable.

Example 32 (outside the invention): non-extruded starch/hydroxypropylcellulose

The solubility properties of a non-extruded mixture of starch and hydroxypropylcellulose (80/20) were evaluated in water and in a 70/30 water/ethanol mixture.

The 14% aqueous solution of the starch and hydroxypropylcellulose mixture exists in the form of a viscous gel.
The 10% aqueous/alcoholic solution of the starch and hydroxypropylcellulose mixture exists in the form of a white, unstable and non-filterable viscous solution; a significant white deposit is formed at the bottom of the flask.

The 2 solutions prepared, packaged in a pump-action spray, are not sprayable.

Example 33 (outside the invention): extruded starch/hydroxypropylcellulose

A mixture formed of 80% by weight of starch and 20% by weight of hydroxypropylcellulose was extruded at 170°C with a speed of the screws of 80 rev/min and a recorded extrusion force of 4400 N.

The solubility properties of the extrudate obtained were evaluated: the 10% aqueous/alcoholic extrudate solution exists in the form of a heterogeneous solution; a solid phase settles out at the bottom of the flask.

Example 34:

Extrudate of gum arabic, hydroxypropylcellulose and tartaric acid

Composition of the extrudate prepared:

Extrudate	Gum arabic (3)	Hydroxypropylcellulose	Tartaric acid	Extrusion force (N)
34	72.1	18	9.9	5000

(3) atomized gum arabic from Alland & Robert

The solubility of the extrudate was determined in a (50/50 weight) water/ethanol mixture:

Extrudate	Solids content at 130°C (%)
34	5.1

The aqueous/alcoholic solution prepared with extrudate 34 was filtered in order to obtain the hair lotion.

Cosmetic properties of the hair lotion:

Extrudate	Quality of the spray	Fixing of the treated locks
34	Very good	Good

Extrudate 34 confers good properties of spray quality and of fixing of the treated locks of hair.

Formulation examples 35 to 38

The contents are expressed as percentage by weight.

The aerosol compositions were packaged in a pump-action spray.

Composition 35: pump-action spray

Extrudate of example 4		5%
Ethanol		30%
Fragrance	q.s.	
Water	q.s. for	100%

Composition 36: pump-action spray

Extrudate of example 23		5%
Ethanol		30%
Fragrance	q.s.	
Water	q.s. for	100%

Composition 37: pump-action spray

Extrudate of example 5		5%
Ethanol		30%
Fragrance	q.s.	
Water	q.s. for	100%

Composition 38: pump-action spray

Extrudate of example 24		5%
Ethanol		30%
Fragrance	q.s.	
Water	q.s. for	100%

Compositions 35 to 38 are applied to dry hair at ambient temperature and without rinsing.

After drying, good form retention of the hair style (strong fixing of the hair) is obtained.

CLAIMS

1. A process for the preparation of a cosmetic composition comprising the following stages:

a) preparation of a premix comprising:
an ethanol-insoluble polysaccharide;
an ethanol-soluble polymer;
a polycarboxylic acid comprising from 4 to 8 carbon atoms or the anhydride of said acid;
and optionally a plasticizing agent;

b) said premix is kneaded under shearing, at a temperature between 100°C and 250°C, in order to form an extrudate;

c) cooling the extrudate to a temperature of less than 100°C;

d) introducing the cooled extrudate into water or into a water/ethanol mixture comprising from 0% to 75% by weight of ethanol;

e) recovering the filtrate obtained.

2. The process as claimed in the preceding claim, in which the ethanol-insoluble polysaccharide is chosen from starch, cellulose, guar gum, gum arabic, alginic acid, alginate salts and carrageenans.

3. The process as claimed in either of the preceding claims, in which the ethanol-insoluble polysaccharide is chosen from starch or gum arabic.

4. The process as claimed in the preceding claim, in which the ethanol-insoluble polysaccharide is starch.

5. The process as claimed in one of the preceding claims, in which the ethanol-insoluble polysaccharide is a fixing polymer.

6. The process as claimed in one of the preceding claims, in which the ethanol-insoluble polysaccharide is present in the premix in a content ranging from 55% to 85% by weight, preferably ranging from 60% to 80% by weight, with respect to the total weight of the premix.

7. The process as claimed in one of the preceding claims, in which the ethanol-soluble polymer is chosen from ethylcellulose, hydroxypropylcellulose, hydroxyethyl ethylcelluloses, carboxymethylethylcelluloses and their sodium salts, carboxymethylhydroxyethylcelluloses and their sodium salts, polyvinyl alcohol,

poly(C_2-C_4 hydroxyalkyl (meth)acrylate)s, polyvinylpyrrolidone or poly(2-ethyl-2-oxazoline).

8. The process as claimed in one of the preceding claims, in which the ethanol-soluble polymer is hydroxypropylcellulose.

9. The process as claimed in one of the preceding claims, in which the ethanol-soluble polymer is present in the premix in a content ranging from 10% to 40% by weight, preferably ranging from 10% to 30% by weight, preferably ranging from 10% to 20% by weight, with respect to the total weight of the premix.

10. The process as claimed in one of the preceding claims, in which the polycarboxylic acid or its anhydride is chosen from maleic acid, maleic anhydride, succinic acid, succinic anhydride, itaconic acid, itaconic anhydride, tartaric acid, phthalic acid, phthalic anhydride or citric acid.

11. The process as claimed in one of the preceding claims, in which the polycarboxylic acid is chosen from maleic acid, maleic anhydride or tartaric acid.

12. The process as claimed in one of the preceding claims, in which the polycarboxylic acid is present in the premix in a content ranging from 0.1% to 30% by weight and preferably ranging from 1% to 20% by weight, with respect to the total weight of the premix.

13. The process as claimed in one of the preceding claims, in which the plasticizing agent is chosen from glycerol, ethylene glycol, propylene glycol, 1,4-propanediol, 1,2-butanediol, 1,3-butanediol, 1,4-butanediol, 1,5-pentanediol, 1,5-hexanediol, 1,6-hexanediol, 1,2,6-hexanetriol, 1,3,5-hexanetriol, neopentyl glycol, polyethylene glycols having a molecular weight ranging from 200 to 1500 g/mol, polypropylene glycols having a molecular weight ranging from 200 to 1500 g/mol, sorbitol (solid), sorbitol monoacetate, sorbitol diacetate, sorbitol monoethoxylate, sorbitol diethoxylate, sorbitol hexaethoxylate, sorbitol dipropoxylate, aminosorbitol, 2-amino-2-(hydroxymethyl)propane-1,3-diol, trimethylolpropane monoethoxylate, mannitol monoacetate, mannitol monoethoxylate, butylglucoside, glucose monoethoxylate, α-methylglucoside, sodium carboxymethylsorbitol, polyglycerol (2 to 10 glycerol units) monoethoxylate, erythritol, pentaerythritol, arabitol, adonitol, xylitol, mannitol, iditol, galactitol, allitol, glycerol esters, mono- and diglycerides, trimethyl propane, polyvinyl alcohol having from 3 to 20 repeat units, polyglycerols having from 2 to 10 repeat units, and their mixtures.

14. The process as claimed in one of the preceding claims, in which the plasticizing agent is chosen from glycerol and sorbitol.

15. The process as claimed in one of the preceding claims, in which the plasticiz-

ing agent is present in the extrusion premix or added in the kneading stage in a content ranging from 0.1% to 39.9% by weight, preferably ranging from 0.1% to 30% by weight, preferably ranging from 1% to 20% by weight and more preferably ranging from 2% to 15% by weight, with respect to the total weight of the premix.

16. The process as claimed in one of the preceding claims, in which the kneading stage b) of the preparation process is carried out using a screw mixer/extruder.

17. A cosmetic composition capable of being obtained according to the preparation process as claimed in any one of the preceding claims.

18. A method for the cosmetic treatment of the hair, in particular for fixing and/or shaping the hair, comprising the application to the hair of a cosmetic composition as claimed in the preceding claim or obtained according to the process as claimed in any one of claims 1 to 16.