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(54) **SUPPLEMENTATION OF CELLULOSE NANOFIBRILS WITH CARBOXYCELLULOSE WITH LOW DEGREE OF SUBSTITUTION**

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(57) **ABSTRACT**

The invention concerns compositions containing essentially amorphous cellulose nanofibrils, carboxyl cellulose with a degree of substitution not more than 0.95 as additive, and optionally at least one co-additive, the content in additive and optional co-additive being less than 30 wt. % relative to the weight of nanofibrils and additive and optional co-additive. The invention also concerns a method for preparing such compositions, consisting in adding to a substantially amorphous nanofibril suspension, the additive and optionally the co-additive(s), then in drying the suspension thus additivated. The resulting compositions are easily redispersable and preserve all their rheological properties.

**26 Claims, No Drawings**



**SUPPLEMENTATION OF CELLULOSE  
NANOFIBRILS WITH  
CARBOXYCELLULOSE WITH LOW  
DEGREE OF SUBSTITUTION**

This application is a 371 of PCT/FR97/01290 filed Jul. 11, 1997.

The present invention relates to compositions comprising essentially amorphous cellulose nanofibrils, carboxycellulose as additive, and optionally co-additives, as well as to a process for their preparation.

The invention relates to the suspensions obtained from such compositions.

Cellulose microfibrils and nanofibrils are well-known compounds which are used as additives for modifying the texture of media into which they are introduced. In the case of fluid media, they modify their viscosity or even their rheological profile.

However, there is a problem with cellulose microfibrils and nanofibrils, which is that they are obtained in the form of an aqueous suspension whose solids content is relatively low, from about 1 to about 5% by weight approximately. The development of these products in such a form is thus not economically viable, either in terms of storage or transportation, for example. It has thus been considered, naturally, to present them in a dry form. Unfortunately, when the cellulose microfibril or nanofibril suspensions are dried, very strong hydrogen bonds are created between the fibrils which make it necessary to use very high-shear means to redisperse these fibrils, when it is possible to resuspend them.

Attempts have been made to propose solutions to the problem of drying cellulose microfibrils. Thus, additives have been introduced during the preparation of microfibril suspensions, and more particularly at the time of the homogenization.

For example, U.S. Pat. No. 4,481,076 proposes drying the cellulose microfibrils obtained from wood pulp in the presence of additive. The contents which are most favourable for good redispersion after drying, and thus for a good viscosity level of the suspension, are from about 50 to about 100% by weight relative to the dry microfibrils. As may be observed, the amounts of additives introduced are very large. Moreover, these methods are not entirely satisfactory, even though it is possible, in principle, to redisperse these dried microfibrils, since the means used for the redispersion are still very high-shear.

International patent application WO 95/02966 describes the supplementation of microcrystalline cellulose with xanthan gum or carboxymethylcellulose, with contents of less than 33% by weight relative to the weight of microcrystalline cellulose. However, extremely high-shear conditions are used to suspend the dried cellulose, since they are carried out under the standard conditions for stirring formulations intended for applications in the food sector. The dried microfibrils can thus not be considered as easily redispersible.

The teaching provided by the prior art regarding the redispersion of microcrystalline cellulose microfibrils, and in particular those obtained from wood pulp, cannot be transposed to cellulose nanofibrils, obtained from cells with primary walls.

Firstly, the cellulose microfibrils obtained from wood are derived from secondary walls. This means that they have a greater than 70% degree of crystallinity. During the step of homogenization of the microfibrils obtained from wood, rather than observing a disentangling of the fibres, as is the

case during the step of homogenization of the cellulose nanofibrils obtained from primary walls, these fibrils are found to break. Consequently, the cellulose microfibrils obtained from secondary walls do not have the characteristics of amorphous fibrils, but, rather, have the characteristics of microcrystalline microfibrils.

Moreover, the morphologies of the microfibrils and nanofibrils are different. In point of fact, the microcrystalline microfibrils, for example obtained from cellulose with secondary walls, such as wood pulp, are conventionally in the form of aggregates from a few tens of nanometers to a few micrometers, consisting of elementary fibrils, which cannot be disentangled during the homogenization step. As regards the cellulose nanofibrils obtained from cells with primary walls, they have a diameter of not more than a few nanometers and have the appearance of filaments.

It is relatively well established that the difficulty in redispersing cellulose microfibrils or nanofibrils is associated with the existence of numerous hydrogen bonds between the fibrils, which are created during drying. Now, the number of hydrogen bonds per weight unit of cellulose is directly associated with the morphology of the said microfibrils or nanofibrils, and, more specifically, proportional to their specific surface; the greater the specific surface, the larger the number of hydrogen bonds per weight unit of cellulose. Given the particular morphology of the cellulose nanofibrils obtained from cells with primary walls, the specific surface of these nanofibrils is much higher than that of the microfibrils. A person skilled in the art would thus logically expect to encounter greater difficulties in redispersing cellulose nanofibrils.

Thus, given the state of the art presented above, it could be envisaged that larger amounts of additive than those used for the microfibrils would be necessary in order to obtain good redispersion of the dried nanofibrils.

However, the present invention has shown, against all expectations, that relatively low amounts of additive are sufficient to allow good redispersion of the dried nanofibrils, and without it being necessary to use extremely high-shear conditions. In addition, it has been found, surprisingly, that amounts of the order of those recommended in the prior art have considerable drawbacks as regards conserving the Theological properties of the nanofibrils.

This arises from the difference in behaviour between the crystalline microfibrils, for example the cellulose microfibrils obtained from secondary walls, and the nanofibrils obtained from cells with primary walls.

The reason for this is that the non-supplemented microcrystalline microfibrils are not dispersible in aqueous medium; they separate out by settling as soon as the stirring is stopped, even when very high-shear stirring means are used. Furthermore, they do not give shear-thinning Theological properties.

On the other hand, the nanofibrils obtained from primary walls are of a nature which is dispersible in aqueous medium. In addition, they give a quite specific Theological profile, of shear-thinning type, to the medium into which they are introduced.

Now, in general, drying adversely affects not only the capacity for redispersion of the dried nanofibrils and their viscosity, but also their rheological profile. Thus, large amounts of additives of the type usually used to redisperse microcrystalline microfibrils, such as those obtained from wood, i.e. as much additive as microfibrils, do not give good results as regards the shear-thinning Theological profile of the cellulose nanofibrils obtained from primary walls: the profile becomes more Newtonian, i.e. less shear-thinning.

As may be observed, the consequences of drying essentially amorphous cellulose nanofibrils in terms of the redispersion of these fibrils and their Theological properties (viscosity at low and high shear, rheological profile) cannot be solved in a satisfactory manner based on the knowledge obtained from the supplementation of microcrystalline microfibrils, for example of microfibrils obtained from cells with secondary walls.

The present invention thus provides a simple and effective solution to these problems.

These aims and others are achieved by the present invention, a first subject of which is a composition comprising essentially amorphous cellulose nanofibrils, carboxycellulose with a degree of substitution of less than or equal to 0.95 as additive, and optionally at least one co-additive, the content of additive and of optional co-additive being less than or equal to 30% by weight relative to the weight of nanofibrils and of additive and optional co-additive.

Another subject of the present invention consists of a process for preparing a composition, in which cellulose nanofibrils are prepared from cellulosic pulp by carrying out at least one extraction, optionally followed by at least one step of bleaching the pulp thus treated, after which the resulting pulp is separated out and a homogenization step is carried out in at least one cycle, the characteristic of the process being that the following steps are carried out:

at least some of the additive and optionally co-additive(s) are added to the nanofibril suspension, which has optionally undergone at least one homogenization cycle,

a step of drying the suspension thus supplemented is carried out.

A third subject of the invention relates to a suspension comprising cellulose nanofibrils, which is obtained by redispersing the composition according to the invention.

The present invention makes it possible simultaneously to propose a process for drying essentially amorphous nanofibrils in the presence of additives, as well as compositions which are dried such that they are readily redispersible, while at the same time retaining the specific rheological properties of the initial, non-dried suspensions. Thus, the suspensions according to the invention, obtained after redispersing the compositions, have a good level of viscosity at a low shear gradient, as well as a rheological profile of shear-thinning type.

In addition, the means used to redisperse the dried compositions according to the invention are considerably less shearing than those usually used to redisperse dried microfibrils obtained from wood or from other secondary walls.

Other characteristics and advantages of the present invention will emerge more clearly on reading the description and the examples which follow.

As has been mentioned previously, the subject of the present invention is the supplementation of essentially amorphous cellulose nanofibrils.

The term "essentially amorphous" is intended to refer to nanofibrils whose degree of crystallinity is less than or equal to 50%. According to a specific variant of the present invention, the degree of crystallinity is between 15% and 50%. Preferably, the degree of crystallinity is less than 50%.

The cellulose nanofibrils treated according to the present invention are obtained from cells preferably consisting of at least about 80% primary walls. Preferably, the amount of primary walls is at least 85% by weight.

Such characteristics are present in particular in parenchymal cells. Sugar beet pulp, citrus fruits such as lemons,

oranges and grapefruit, and most fruit and vegetables are examples of parenchyma.

Moreover, the nanofibrils forming part of the compositions according to the invention are, according to a particularly advantageous variant, charged at the surface with carboxylic acids and with acidic polysaccharides, alone or as a mixture.

The term "carboxylic acids" is intended to refer to simple carboxylic acids, as well as salts thereof. These acids are preferably chosen from uronic acids. More particularly, the said uronic acids are more particularly galacturonic acid and glucuronic acid.

As acidic polysaccharides, mention may be made of pectins, which are more particularly polygalacturonic acids. These acidic polysaccharides can be present as a mixture with hemicelluloses.

The cellulose nanofibrils also have a cross-section of between about 2 and about 10 nm. More particularly, the nanofibril cross-section is between about 2 and about 4 nm.

According to a particularly advantageous embodiment of the present invention, the nanofibrils forming part of the compositions according to the invention are obtained by using the treatment which will be described below.

More particularly, this treatment is carried out on the pulp of vegetables with primary walls, such as, for example, beetroot pulp, after it has undergone a preliminary step of extraction of the sucrose, according to the methods known in the art.

Thus, the process comprises the following steps:

(a) first acidic or basic extraction, after which a first solid residue is recovered,

(b) optionally, second extraction, carried out under alkaline conditions, of the first solid residue, after which a second solid residue is recovered,

(c) washing of the first or second solid residue,

(d) optionally, bleaching of the washed residue,

(e) dilution of the third solid residue obtained after step (d) so as to obtain a solids content of between 2 and 10% by weight,

(f) homogenization of the dilute suspension.

In step (a), the term "pulp" is intended to refer to wet, dehydrated pulp stored by ensilage or partially depectinized.

The extraction step (a) can be carried out in acidic medium or in basic medium.

For an acidic extraction, the pulp is suspended in an aqueous solution for a few minutes so as to homogenize the acidified suspension at a pH of between 1 and 3, preferably between 1.5 and 2.5.

This operation is carried out with a concentrated solution of an acid such as hydrochloric acid or sulphuric acid.

This step may be advantageous for removing the calcium oxalate crystals which may be present in the pulp, and which, on account of their highly abrasive nature, can cause difficulties in the homogenization step.

For a basic extraction, the pulp is added to an alkaline solution of a base, for example sodium hydroxide or potassium hydroxide, with a concentration of less than 9% by weight, more particularly less than 6% by weight. Preferably, the concentration of the base is between 1 and 2% by weight.

A small amount of a water-soluble antioxidant, such as sodium sulphite  $\text{Na}_2\text{SO}_3$ , may be added in order to limit the oxidation reactions of the cellulose.

Step (a) is generally carried out at a temperature of between about 60° C. and 100° C., preferably between about 70° C. and about 95° C.

The duration of step (a) is between about 1 hour and about 4 hours.

During step (a), partial hydrolysis takes place with release and solubilization of most of the pectins and hemicelluloses, while at the same time retaining the molecular mass of the cellulose.

The solid residue is recovered from the suspension obtained from step (a) by carrying out known methods. Thus, it is possible to separate the solid residue by centrifugation, by filtration under vacuum or under pressure, with filter gauzes or filter presses, for example, or else by evaporation.

The first solid residue obtained is optionally subjected to a second extraction step carried out under alkaline conditions.

A second extraction step, step (b), is carried out when the first step has been carried out under acidic conditions. If the first extraction has been carried out under alkaline conditions, the second step is optional.

According to the process, this second extraction is carried out with a base preferably chosen from sodium hydroxide and potassium hydroxide, whose concentration is less than about 9% by weight, preferably between about 1% and about 6% by weight.

The duration of the alkaline extraction step is between about 1 and about 4 hours. It is preferably equal to about 2 hours.

After this second extraction, if it is carried out, a second solid residue is recovered.

In step (c), the residue derived from step (a) or (b) is washed thoroughly with water in order to recover the residue of cellulosic material.

The cellulosic material from step (c) is then optionally bleached, in step (d), according to the standard methods. For example, a treatment with sodium chlorate, with sodium hypochlorite or with hydrogen peroxide in a proportion of 5–20% relative to the amount of solids treated can be carried out.

Different concentrations of bleaching agent can be used, at temperatures of between about 18° C. and about 80° C., preferably between about 50° C. and about 70° C.

The duration of this step (d) is between about 1 hour and about 4 hours, preferably between about 1 hour and about 2 hours.

A cellulosic material containing between 85 and 95% by weight of cellulose is thus obtained.

After this bleaching step, it may be preferable to wash the cellulose thoroughly with water.

The resulting suspension, which has optionally been bleached, is then rediluted in water in a proportion of 2 to 10% solids (step (e)), before undergoing a homogenization step (step (f)) comprising at least one cycle.

According to a first variant of the invention, the nanofibrils are supplemented before undergoing the homogenization step.

According to a second variant of the invention, the cellulose nanofibrils are supplemented after they have undergone at least one homogenization cycle.

The homogenization step corresponds to a mixing or blending operation or any operation of high mechanical shear, followed by one or more passages of the cell suspension through an orifice of small diameter, subjecting the suspension to a pressure drop of at least 20 mPa and to a high-speed shear action, followed by a high-speed deceleration impact.

The mixing or blending is carried out, for example, by passage(s) through the mixer or blender for a period ranging

from a few minutes to about an hour, in a machine such as a Waring Blendor fitted with a four-blade impeller or a pan mill mixer or any other type of blender, such as a colloidal mill.

The actual homogenization will advantageously be carried out in a homogenizer such as a Manton Gaulin in which the suspension is subjected to a shear action at high speed and high pressure in a narrow passage and against an impact ring. Mention may also be made of the Micro Fluidizer, which is a homogenizer mainly consisting of a compressed-air motor which creates very high pressures, an interaction chamber in which the homogenization operation takes place (elongational shear, impacts and cavitations) and a low-pressure chamber which allows depressurization of the dispersion.

The suspension is introduced into the homogenizer preferably after preheating to a temperature of between 40 and 120° C., preferably between 85 and 95° C.

The temperature of the homogenization operation is maintained between 95 and 120° C., preferably above 100° C.

The suspension is subjected to pressures of between 20 and 100 mPa and preferably above 50 mPa in the homogenizer.

Homogenization of the cellulosic suspension is obtained by a number of passages which can range between 1 and 20, preferably between 2 and 5, until a stable suspension is obtained.

The homogenization operation can advantageously be followed by a high mechanical shear operation, for example in a machine such as the Sylverson Ultra Turrax.

It should be noted that this process has been described in European patent application EP 726,356 filed on Jun. 2, 1996, and reference may thus be made thereto if necessary. Example 20 of that text in particular gives a method for preparing a suspension of essentially amorphous cellulose nanofibrils.

The additives will now be described.

The first additive in the composition according to the invention consists of carboxycellulose, in salt form or in acid form.

The cellulose used as additive is more particularly carboxymethylcellulose. Cellulose is a polymer consisting of glucose monomer units. The carboxyl group is introduced in a manner which is known per se, by reacting chloroacetic acid with cellulose.

The degree of substitution corresponds to the number of carboxymethyl groups per glucose unit. The maximum theoretical degree is 3.

According to the invention, the degree of substitution of carboxymethylcellulose is less than or equal to 0.95.

The degree of polymerization of the carboxycellulose used as nanofibril additive, in accordance with the present invention, varies within a wide range. Thus, carboxymethylcelluloses of high masses (high degree of polymerization, high viscosity) or of low masses (low degree of polymerization, low viscosity) are suitable.

In the first category, mention may be made of celluloses whose viscosity is between about 9000 mPa.s, measured in an aqueous 1% solution (Brookfield, 30 rpm), and 250 mpa.s, measured in an aqueous 6% solution (Brookfield, 60 rpm).

In the second category, mention may be made of celluloses whose viscosity is between about 250 mpa.s, measured in an aqueous 6% solution (Brookfield, 60 rpm), and 10 mpa.s, measured in an aqueous 6% solution (Brookfield, 60 rpm).

In the case of the first category, the carboxycellulose content is less than or equal to 30% by weight.

In the case of the second category, the carboxycellulose content is between 10 and 30% by weight.

The composition according to the invention can also comprise at least one co-additive chosen from:

saccharide monomers or oligomers,

compounds of formula  $(R^1R^2N)COA$ , in which formula  $R^1$  and  $R^2$ , which may be identical or different, represent hydrogen or a  $C_1-C_{10}$ , preferably  $C_1-C_5$ , alkyl radical, A represents hydrogen, a  $C_1-Cl_6$ , preferably  $C_1-C_5$ , alkyl radical or alternatively the group  $R^1R^2N$  with  $R^1$  and  $R^2$ , which may be identical or different, representing hydrogen or a  $C_1-C_{10}$ , preferably  $C_1-C_5$ , alkyl radical,

cationic or amphoteric surfactants, it being possible for these co-additives to be used alone or as a mixture.

Among the saccharide monomers or oligomers, mention may be made most particularly, and without intending to be limiting, of sorbitol, sucrose and fructose.

As regards the compounds of the type  $(R^1R^2N)COA$ , it is preferred to use compounds comprising two amide functions. Preferably, urea is used as co-additive.

Among the cationic surfactants, mention may be made of cationic quaternary ammonium derivatives such as, for example, cationic imidazoline derivatives, alkyltrimethylammonium, dialkyldimethylammonium, alkyl dimethylbenzylammonium or alkyl dimethylethylammonium halides and Quat esters.

As examples of suitable cationic compounds, mention may be made of the products sold by Rhône-Poulenc from the Rhodaquat range. It is also possible to use synthetic cationic polymers, known under the CTFA generic name of "Polyquaternium", for example the polymers Mirapol A15<sup>®</sup> or Mirapol 550<sup>®</sup> from the company Rhône-Poulenc.

The surfactants forming part of the formulation according to the invention can also be chosen from amphoteric surfactants. For example, mention may be made, without intending to be limiting, of alkylpolyamine amphoteric derivatives, alkybetaines, alkyldimethylbetaines, alkylamidopropylbetaines, alkylamidopropyl dimethylbetaines, alkyltrimethylsulphobetaines, imidazoline derivatives such as alkyl amphoacetates, alkyl amphodiacetates, alkyl amphopropionates, alkyl amphodipropionates, alkylsultaines or alkylamidopropylhydroxysultaines, and the condensation products of fatty acids and of protein hydrolysates, it being possible for these compounds to be used alone or as a mixture.

The surfactants Mirapon<sup>®</sup> Excel, Mirataine<sup>®</sup> CBS, Mirataine<sup>®</sup> CB, Mirataine H2CHA<sup>®</sup>, Ampholac 7T/X<sup>®</sup>, Ampholac 7C/X, the Miranol<sup>®</sup> range, Amphionic<sup>®</sup> SFB and Amphionic<sup>®</sup> XL may in particular be suitable for carrying out the present invention.

When the compositions according to the invention comprise one or more of the abovementioned co-additives, their content is less than 30% by weight relative to the weight of nanofibrils and of additive and of co-additive. Needless to say, the content of additive and of co-additive(s) is such that it is less than or equal to 30% relative to the weight of nanofibrils, of additive and of co-additive(s).

According to a first specific variant of the invention, the compositions comprise carboxycellulose as additive, as well as at least one co-additive chosen from saccharide monomers and oligomers or compounds of formula  $(R^1R^2N)COA$ .

In the case of this first variant, the co-additive content is between 1 and 25% by weight relative to the weight of nanofibrils and of additive and of co-additive.

According to a second specific variant of the invention, the compositions comprise carboxycellulose as additive and, as co-additive, at least one compound chosen from cationic and amphoteric surfactants.

In the case of this second variant, the co-additive content is between 1 and 10% by weight relative to the weight of nanofibrils and of additive and of co-additive.

In each of the two variants, the content of carboxycellulose additive is less than or equal to 30% by weight relative to the weight of nanofibrils and of additive and of co-additive.

In the case of redispersion additives such as carboxycellulose with a low degree of substitution (degree of substitution of less than or equal to 0.95), the higher its concentration, the more it lowers the shear-thinning nature of the cellulose nanofibrils by modifying their state of dispersion in the water. Thus, for additive concentrations of greater than 30% by weight relative to the weight of nanofibrils and of additive and of co-additive, although the nanofibrils are redispersible, their Theological profile becomes more Newtonian, i.e. less shear-thinning.

A first particularly advantageous embodiment of the present invention consists of compositions comprising nanofibrils with a content of additive and of co-additive of less than or equal to 30% by weight relative to the weight of nanofibrils and of additive and of co-additive. Preferably, the said content is between 5% and 30% relative to the same reference. It should be noted that the use of such co-additives described above makes it possible, in combination with carboxymethylcellulose, to reinforce the shear-thinning profile of the cellulose nanofibrils after redispersion.

In addition, the compositions according to the invention have a solids content of at least 40% by weight. More particularly, the solids content is at least 60% by weight and is preferably at least 70% by weight.

The particle size of the composition according to the invention can vary within a wide range. It is usually between 1 Aim and a few millimeters.

The process for preparing the compositions will now be described in greater detail.

The process according to the invention consists firstly in preparing the cellulose nanofibrils from appropriate cellulosic pulp, by carrying out a hydrolysis, optionally followed by at least one step of bleaching of the pulp thus treated. Everything which has been mentioned previously in this respect remains valid and will not be repeated here.

The process for preparing the compositions according to the invention consists, in a first step, in adding at least some of the additive and optionally co-additive(s) to the nanofibril suspension, which has optionally undergone at least one homogenization cycle. Next, in a second step, a step of drying the suspension thus supplemented is carried out.

According to a first advantageous variant of the present invention, the addition of at least some of the additive and optionally co-additive(s) is carried out after the homogenization step.

One particularly suitable embodiment of the invention consists in adding at least some of the additive and optionally co-additive(s) to the suspension after the homogenization step, after this suspension has undergone at least one concentration step.

The concentration step(s) take place by filtration, centrifugation or evaporation of some of the water from the suspension. It is possible, for example, to use filters under vacuum or under pressure, spraying towers, ovens or microwave ovens.

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