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(73) Octrooihouder(s):

Coöperatie Koninklijke Cosun U.A. te Breda

(72) Uitvinder(s):

Henri J.M. Grünbauer te Amersfoort Henricus Wilhelmus Carolina Raaijmakers te Roosendaal

**Robert Lazeroms te Sprundel** 

(74) Gemachtigde:

dr. R.C. van Duijvenbode c.s. te Den Haag

#### (54) DISPERSIONS OF PLANT-BASED SOLID PARTICLES IN LIQUID POLYOLS

- The invention provides a dispersion of a liquid continuous phase and solids dispersed therein, said dispersion consisting of:
  - (a) 50 80 wt.% of a liquid, based on the weight of the dispersion;
  - (b) 20 40 wt.% of plant-based solid particles, based on the weight of the dispersion; and
  - (c) 0 10 wt.% of one or more further ingredients other than (a) and (b), based on the total weight of the dispersion.

wherein the plant-based solid particles (b) comprise, based on dry matter of the solid particles (b), at least 70 wt.% of parenchymal cell wall material and less than 6 wt.% of lignin, wherein the plant-based solid particles (b) comprise, based on the weight of the solid particles (b), less than 7 wt.% of water, wherein the plant-based solid particles (b) have a particle size distribution characterized by a D90 of 300 µm or smaller, as measured in accordance with dry powder laser diffraction, wherein the liquid (a) consists of one or more polyol compounds, and wherein the viscosity of the dispersion at 20 °C and at a shear rate of 0.1 s<sup>-1</sup> is at least 20 Pas.

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#### DISPERSIONS OF PLANT-BASED SOLID PARTICLES IN LIQUID POLYOLS

#### FIELD OF THE INVENTION

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The invention relates to dispersions comprising liquid polyols and plant-based solid particles dispersed therein, to a process for producing said dispersions and to uses of said dispersions.

#### **BACKGROUND OF THE INVENTION**

Polyurethanes are widely used polymers and are usually obtained from reaction between polyols and (poly)isocyanates. The production of other widely used polymers such as polyesters, polyamides, polyisocyanurates, polyethers, and polycarbonates also depends on polyols as a feedstock. The feedstocks used in the methods of producing these polymers are typically petroleum-dependent. This puts pressure on the source of raw materials because of environmental concerns. The finite nature of petroleum reserves makes it imperative to search for alternative and renewable sources of raw materials to produce these polymers.

Accordingly, there has been a growing interest in replacing at least part of the petroleum-based feedstocks used in the production of these polymers by alternative and renewable sources of raw materials. Renewable sources of raw materials that are of particular interest in this regard are biomass residues, e.g. obtained in agriculture or forestry, because of their abundant availability.

Sugar beet pulp is a by-product resulting from sugar extraction of sugar beets. Sugar beet pulp has not found many industrial applications, apart from its use as or in animal feed and in the production of biogas. This residue is obtained as a wet material with about 25 wt.% dry solids. It is insoluble in many solvents and therefore has very limited reactivity.

EP0682050A1 concerns a process for the production of polyurethane foams wherein at least part of the polyol is substituted with a powder of exhaust pulp of sugar beets. The sugar beet particles are described to be more than a filler in that they act as an active agent. The water inside the sugar beet pulp particles, 6-12 wt.%, is described to help foam forming.

In a first embodiment of EP0682050A1, ground exhausted sugar beet powder is used as such. It is described in EP0682050A1 that this sugar beet powder is practically insoluble and has low reactivity. As regards this first embodiment, it is stated that it is impossible to thoroughly mix the powdered solid, *i.e.* the powder of exhaust pulp of sugar beet, with the liquid viscous polyol. As a consequence the liquid polyol is first mixed with additives, such as catalysts, fillers and surfactants; and thereafter, on said mix, a layer of powdered solid is laid

down. Example 1 of EP0682050A1 is an example of the first embodiment wherein ground spent sugar beet particles with a particle size distribution characterized by particles having sizes between 0.2 and 0.5 mm are combined with (*i.e.* 'laid down on a layer of') polyol sorbitol in weight ratios of 1:1 to 3:1. It was concluded in EP0682050A1 that the foams obtained with the sugar beet particles therein had chemical-physical characteristics which were absolutely comparable to those of commercial foams.

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EP0682050A1 discloses in a second embodiment reaction of sugar beet particles with propylene oxide to make the particles more soluble and reactive.

US2019/0119432A1 concerns the use of ground crop residues having an average particle size of less than 10 mm in polyurethane systems. The polyurethane systems comprise an isocyanate component and an isocyanate-reactive component. The average particle size of the ground crop residues (based on a number distribution model) can be less than 650 μm. Exemplary grindable crop residues include roots. The working examples disclose the production of polyurethane systems by hand-mixing of 100 parts by weight of a formulation system comprising a blend having at least a high functional polyether polyol, a low functional polyol, and additives such as a catalyst and blowing agent, 120 parts by weight of an isocyanate component and between 2.0 and 8.0 wt.%, based on the total weight of the polyurethane system, of sunflower hulls ground to an average particles size of less than 600 μm.

C. Pavier *et al.*, *European Polymer Journal*, 36, 2000, pp 1653-1658, disclose polyurethanes produced from oxypropylated sugar beet pulp and isocyanates. The polyol compound consisted of oxypropylated sugar beet pulp in poly(propylene oxide) diol.

A. Strąkowska *et al.*, *Materials*, 13, 2020, 5493, disclose rigid polyurethane foams reinforced with sugar beet pulp impregnated with aminopropylisobutyl-polyhedral oligomeric silsesquioxanes (APIB-POSS). Between 1 and 5 wt.% of impregnated sugar beet pulp was applied in the polyurethane foams. The addition was described to result in improved fire resistance. Polyurethane composite foams were synthesized by (i) alkali treatment of sugar beet pulp with a 5 wt.% solution of sodium hydroxide solution, washing with distilled water, neutralizing with a 1% acetic acid solution and drying, (ii), mixing the thus treated sugar beet pulp with aminopropylisobutyl-POSS (1:1 w/w) using a planetary ball mill, (iii) adding the mixture obtained in step (iii) to a polyol system, (iv) adding other ingredients, such as a surfactant, catalyst, and blowing agent, followed by mixed until complete dispersion of the impregnated sugar beet pulp, and (v) adding and mixing isocyanate. Steps (i) and (ii) result in impregnated sugar beet filler with very small particles sizes between about 0.5 and about 5 μm.

It can be calculated that in step (iii), between 2.8 and 14 wt.% of impregnated sugar beet filler is applied in the polyol dispersion.

Z. Zheng *et al.*, *Int. J. Food Eng.*, 12(9), 2016, pp 911–919, disclose polyurethane foams prepared from microwave liquefied sugar beet pulp and polymethylene polyphenyl isocyanate. The liquefaction procedure comprised thoroughly mixing of sugar beet pulp (milled to a particle size of 75-380 μm and dried), PEG and sulfuric acid, subjecting the resulting mixture to microwave irradiation for 25 min at 500 W during which the temperature was controlled at 160 °C, cooling the product and neutralizing the liquefaction product using magnesium oxide.

I. Domingos *et al.*, *Food and Bioproducts Processing*, 115, 2019, pp 223–229, disclose polyurethane foams from liquefied orange peel wastes. The liquefied orange peel waste was produced using a mixture of ethylene glycol and glycerol (1:1) as solvents, catalysed by sulphuric acid at 180 °C for 60 min.

There is an ongoing need for low-cost fillers based on renewable resources that can be used to replace at least part of the polyols used in the production of polymers. There is further a need for simple carrier products that make the low-cost fillers based on renewable resources readily available for application in the production of said polymers.

It is therefore an object of the invention to provide simple carrier products that make fillers based on renewable resources readily applicable in the production of polymers based on polyols. It is a further object of the invention to provide said simple and stable carrier products wherein the fillers based on renewable resources are not chemically altered materials. It is yet another object of the invention to provide simple and stable polyol systems comprising fillers based on renewable resources for use in the production of polymers.

#### **SUMMARY OF THE INVENTION**

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The inventors have unexpectedly established that one or more of the objectives can be met by dispersing plant-based solid particles, that are not chemically modified and have a specific particle size in polyols and by applying a minimum content of solids of said plant-based solid particles such that the dispersion has a specific minimum viscosity.

Accordingly, in a first aspect, the invention provides a dispersion of a liquid continuous phase and solids dispersed therein, said dispersion consisting of:

- (a) 50 80 wt.% of a liquid, based on the weight of the dispersion;
- (b) 20 40 wt.% of plant-based solid particles, based on the weight of the dispersion; and

(c) 0 - 10 wt.% of one or more further ingredients other than (a) and (b), based on the total weight of the dispersion,

wherein the plant-based solid particles (b) comprise, based on dry matter of the solid particles (b), at least 70 wt.% of parenchymal cell wall material and less than 6 wt.% of lignin,

- wherein the plant-based solid particles (b) comprise, based on the weight of the solid particles (b), less than 7 wt.% of water,
  - wherein the plant-based solid particles (b) have a particle size distribution characterized by a D90 of 300 µm or smaller, as measured in accordance with dry powder laser diffraction, wherein the liquid (a) consists of one or more polyol compounds, and
  - wherein the viscosity of the dispersion at 20 °C and at a shear rate of 0.1 s<sup>-1</sup> is at least 20 Pa·s, as measured using a C-CC27/T200/SS 45971 measuring cup and a ST24-2D/2V/2V-30-SN26943 vane rotor.

The inventors found that the dispersions according to the invention having a high content of solids exhibit shear thinning behaviour with hysteresis (thixotropy). Accordingly, at sufficiently high content of solids, the plant-based solid particles (b) are stabilized in an internal network structure that can be broken down by shearing the dispersion. At low shear rates, the viscosity of the dispersions according to the invention is sufficiently high to reduce sedimentation of the solid particles (b) to acceptable levels. At high shear rates, the viscosity of the dispersions is sufficiently low to be able to process the dispersions, for example by pumping the dispersion to a reactor vessel.

The dispersions according the invention do exhibit sedimentation over time. This means that the dispersions may need to be homogenized, for example using gentle stirring, before applying them, for example in a polyurethane system. Although, the dispersions according to the invention exhibit thixotropic behaviour, the internal network is reformed sufficiently quick after a breakdown due to stirring to stabilize the dispersion again as regards sedimentation of the plant-based solid particles (b).

In general, it was found that dispersions having a viscosity of at least 20 Pa·s at 20 °C and at a shear rate of 0.1 s<sup>-1</sup> exhibited sufficient stability against sedimentation.

In a second aspect, the invention provides a process for the production of the dispersion as defined hereinbefore, said process comprising:

(i) providing liquid (a);

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- (ii) adding the plant-based solids particles (b) and the optional one or more further ingredients (c); and
- (iii) stirring the mixture obtained in step (ii) until a homogeneous dispersion is obtained.

A third aspect of the invention concerns the use of the dispersion as defined hereinbefore as a feedstock in a process for the production of a polymer.

The inventors have established that the plant-based solid particles (b) can impart flame retardant properties to polymers. There is no need to impregnate the plant-based solid particles (b) with e.g. APIB-POSS to obtain such an effect.

A fourth aspect of the invention concerns the use of the dispersion as defined hereinbefore as a flame retardant in a polymer.

#### 15 BRIEF DESCRIPTION OF THE FIGURES

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Figures 1-3 graphically depict stability of the dispersions according to the invention and of comparative dispersions at room temperature (~20 °C) as a function of time.

Figures 4-9 graphically depict viscosity vs shear rate profiles of the dispersions according to the invention and of comparative dispersions at 20 °C.

Figure 10 graphically depicts thixotropic behaviour of a few dispersions according to the invention.

#### DETAILED DESCRIPTION

In a first aspect, the invention concerns a dispersion of a liquid continuous phase and solids dispersed therein, said dispersion consisting of:

- (a) 50 80 wt.% of a liquid, based on the weight of the dispersion;
- (b) 20 40 wt.% of plant-based solid particles, based on the weight of the dispersion; and
- (c) 0 10 wt.% of one or more further ingredients other than (a) and (b), based on the total weight of the dispersion,
- wherein the plant-based solid particles (b) comprise, based on dry matter of the solid particles (b), at least 70 wt.% of parenchymal cell wall material and less than 6 wt.% of lignin, wherein the plant-based solid particles (b) comprise, based on the weight of the solid particles (b), less than 7 wt.% of water,

wherein the plant-based solid particles (b) have a particle size distribution characterized by a D90 of 300  $\mu$ m or smaller, as measured in accordance with dry powder laser diffraction, wherein the liquid (a) consists of one or more polyol compounds, and wherein the viscosity of the dispersion at 20 °C and at a shear rate of 0.1 s<sup>-1</sup> is at least 20 Pa·s, as measured using a C-CC27/T200/SS 45971 measuring cup and a ST24-2D/2V/2V-30-SN26943 vane rotor.

Unless specified otherwise, all viscosities as defined herein are measured using a C-CC27/T200/SS 45971 measuring cup and a ST24-2D/2V/2V-30-SN26943 vane rotor.

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As described hereinbefore, the dispersion is a dispersion of a liquid continuous phase with solid particles dispersed therein. This dispersion consists of a liquid (a), plant-based solid particles (b) and one more further ingredients (c) that are different form the liquid (a) and the plant-based solid particles (b).

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As will be appreciated by the skilled person, the mere fact that the one more further ingredients (c) are different from the liquid (a) and the plant-based solid particles (b) does not mean that the one more further ingredients (c) cannot be liquid-like or cannot be solid particles.

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As will be appreciated by those skilled in the art, the term 'plant-based solid particles' as used herein refers to solid particles that consist of natural ingredients of the plant from which the particles are obtained. This means that the plant-based solid particles do not comprise plant ingredients that have been chemically modified. Accordingly, the plant-based solid particles do for example not comprise oxypropylated or liquefied plant pulp particles.

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#### Liquid (a)

The dispersion comprises liquid (a) consisting of one or more polyol compounds. The term 'polyol compounds' is also referred to in the art as 'polyhydric compounds'. The term polyol compounds as used herein refers to organic compound wherein the individual molecules contain two or more hydroxyl groups. As will be appreciated by those skilled in the art, polyol compounds are obtained by chemical synthesis which typically is a statistical process. Consequently, polyol compounds typically have an average hydroxy functionality that is not exactly two or exactly three, but they have for example a real value in between. Accordingly the average hydroxy functionality of the polyol compounds in liquid (a) need not be an integer

but typically is a real number. The average hydroxy functionality of the polyol compounds is preferably a real number between 1.9 and 7, such as a real number between 1.9 and 4. The average hydroxy functionality of the liquid (a) consisting of one or more polyol compounds is preferably a real number between 1.9 and 3.

The one or more polyol compounds constituting liquid (a) preferably have an hydroxyl number from 50 mg KOH/g to 550 mg KOH/g, more preferably 100 to 550 mg KOH/g, as measured in accordance with ASTM D4274 - 16. In an embodiment, liquid (a) consisting of one or more polyol compounds has an hydroxyl number from 50 mg KOH/g to 550 mg KOH/g, more preferably 100 to 550 mg KOH/g, as measured in accordance with ASTM D4274 – 16.

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In an embodiment, liquid (a) consists of one or more polyol compounds having an average hydroxy functionality of 4 or lower and one or more polyol compounds having an average hydroxy functionality of higher than 4.

In an embodiment, liquid (a) consists of one or more polyol compounds having an average hydroxy functionality of between 1.9 and 4 and one or more polyol compounds having an average hydroxy functionality of between higher than 4 and 7.

The term 'liquid' in the context of 'liquid (a) consisting of one or more polyol compounds' means that the one or more polyol compounds are liquid at temperatures above 5 °C.

Preferred examples of the one or more polyol compounds constituting liquid (a) are chosen from the group consisting of pentaerythritol, trimethylolpropane, glycols, (hydroxy alkylated) sugar alcohols, glycerine, (modified) polyether polyols (for example such as sold under the trade name Voranol<sup>TM</sup> by Dow Chemical Company, US), (modified) aliphatic and aromatic polyester polyols (for example such as sold under the trade name Stepanpol<sup>®</sup> by Stepan Company, US), polycarbonate polyols and polyols derived from plant-based natural oils.

Non-limiting examples of glycols are ethylene glycol, diethylene glycol, triethylene glycol, tetraethylene glycol, polyethylene glycol, propylene glycol, dipropylene glycol, tripropylene glycol, polypropylene glycol, neopentyl glycol, 1,3-propanediol, 1,3-butanediol, 1,4-butanediol, 1,6-hexanediol and 4-cyclohexanedimethanol.

Preferred examples of polyol compounds include diethylene glycol-phthalic anhydride-based polyester polyol (Stepanpol® PS-3152, average hydroxy functionality of 2.0), sucrose/glycerine initiated polyether polyol (Voranol<sup>TM</sup> RH 360, average hydroxy functionality of 4.5), polypropylene glycol (Voranol<sup>TM</sup> 1010 L, average hydroxy functionality of 2).

In a preferred embodiment, the liquid (a) consisting of the one or more polyol compounds has a viscosity of between 0.1 and 15 Pa·s at 20 °C and at a shear rate of 0.1 s<sup>-1</sup>, as measured using a C-CC27/T200/SS 45971 measuring cup and a ST24-2D/2V/2V-30-SN26943 vane rotor. In a preferred embodiment, the liquid (a) consisting of the one or more polyol compounds has a viscosity of between 0.2 and 10 Pa·s, as measured at 20 °C and at a shear rate of 0.1 s<sup>-1</sup>, as measured using a C-CC27/T200/SS 45971 measuring cup and a ST24-2D/2V/2V-30-SN26943 vane rotor.

In another embodiment, the liquid (a) consisting of the one or more polyol compounds has a viscosity of between 2 and 8 Pa·s, as measured at 20 °C and at a shear rate of 0.1 s<sup>-1</sup>, as measured using a C-CC27/T200/SS 45971 measuring cup and a ST24-2D/2V/2V-30-SN26943 vane rotor. In yet another embodiment, the liquid (a) consisting of the one or more polyol compounds has a viscosity of between 0.1 and 3 Pa·s, as measured at 20 °C and at a shear rate of 0.1 s<sup>-1</sup>, as measured using a C-CC27/T200/SS 45971 measuring cup and a ST24-2D/2V/2V-30-SN26943 vane rotor.

#### Solid particles (b)

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The plant-based solid particles (b) comprise, based on dry matter of the solid particles (b), at least 70 wt.% of parenchymal cell wall material and less than 6 wt.% of lignin. The term 'parenchymal cell wall material' is well known in the art. Parenchymal cell walls, which may also be denoted 'primary cell walls', refer to the soft or succulent tissue, which is the most abundant cell wall type in edible plants. For instance, in sugar beets, the parenchymal cells are the most abundant tissue that surrounds the secondary vascular tissues (xylem and phloem). In this respect, reference is made to P.W. van der Poel et al., Sugar Technology, Verlag Dr Albert Bartens KG, Berlin 1998, page 211. Parenchymal cell walls contain relatively thin cell walls compared to secondary cell walls and are tied together by pectin. In secondary cell walls (xylem and phloem tissues), the cell walls are much thicker than parenchymal cells and are linked together with lignin. The plant-based solid particles (b) can thus be distinguished from for example wood pulp in their low lignin content.

Polysaccharides typically make up 90% or more of the primary plant cell walls, cellulose, hemicelluloses and pectins being the main constituents. The precise morphology and (chemical) make-up of parenchymal cell walls may vary from species to species. The parenchymal cell wall material in the plant-based solid particles (b) can be obtained from a variety of plant sources containing parenchymal cell walls.

In an embodiment, the parenchymal cell wall material is obtained from wet spent sugar beet pulp, wet pulp from citrus fruits, wet pulp from tomatoes, wet spent pulp from chicory, wet pulp from potatoes, wet pulp from pineapple, wet pulp from apple, wet pulp from cranberries, wet pulp from grapes, and/or wet pulp from carrots (exclusive of the stems and leaves), preferably obtained from wet spent sugar beet pulp and/or wet spent chicory pulp, more preferably obtained from wet spent sugar beet pulp, e.g. as a by-product of sucrose production.

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In a preferred embodiment, the plant-based solid particles (b) are obtained from wet spent sugar beet pulp, wet pulp from citrus fruits, wet pulp from tomatoes, wet spent pulp from chicory, wet pulp from potatoes, wet pulp from pineapple, wet pulp from apple, wet pulp from cranberries, wet pulp from grapes, and/or wet pulp from carrots (exclusive of the stems and leaves), more preferably obtained from wet spent sugar beet pulp and/or wet spent chicory pulp, even more preferably obtained from wet spent sugar beet pulp, e.g. as a by-product of sucrose production.

In a very preferred embodiment, the plant-based solid particles (b) as defined hereinbefore are obtained by or obtainable by a process consisting of:

- (A) drying wet spent sugar beet pulp, wet pulp from citrus fruits, wet pulp from tomatoes, wet spent pulp from chicory, wet pulp from potatoes, wet pulp from pineapple, wet pulp from apple, wet pulp from cranberries, wet pulp from grapes, and/or wet pulp from carrots (exclusive of the stems and leaves), more preferably obtained from wet spent sugar beet pulp and/or wet spent chicory pulp, even more preferably obtained from wet spent sugar beet pulp, during which the temperature of the pulp preferably does not exceed 95 °C;
- (B) milling or comminuting the dried pulp obtained in step (A); and
- (C) optionally subjecting the milled or comminuted dried pulp obtained in step (B) to sieving.

Since the plant-based solid particles (b) as defined hereinbefore are obtained by or obtainable by this process, step (ii) results in the water content as defined hereinbefore and step (ii) or combined steps (ii) and (iii) result in the particle size distribution as defined hereinbefore.

Preferably, the plant-based solid particles (b) comprise, based on the weight of the solid particles (b), less than 6.5 wt.% of water, more preferably less than 5.5 wt.%, even more preferably less than 5 wt.%, still more preferably less than 4.5 wt.%.

Since process steps (i) to (iii) are presumed not to substantially affect the dry matter composition of the parenchymal cell wall containing plant material, it is believed that the plant-based solid particles (b) will have a dry matter composition largely corresponding to that of the untreated material or the pulp, having cellulose, pectin and hemicellulose as the main constituents.

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Preferably the plant-based solid particles (b) comprise, based on dry matter of the solid particles (b), at least 15 wt.%, preferably 15-40 wt.%, more preferably 15-35 wt.%, most preferably 15-30 wt.% of cellulose.

Preferably the plant-based solid particles (b) comprise, based on dry matter of the solid particles (b), at least 15 wt.%, preferably 15-40 wt.%, more preferably 15-35 wt.%, most preferably 15-30 wt.% of pectin.

Preferably the plant-based solid particles (b) comprise, based on dry matter of the solid particles (b), at least 15 wt.%, preferably 15-40 wt.%, more preferably 15-35 wt.%, most preferably 20-35 wt.% of hemicellulose.

Other typical, non- or sparingly soluble, parenchymal cell (wall) constituents that may be present in the plant-based solid particles (b) include protein, lignin, residual sugars, fat and ash.

Hence, in an embodiment, the plant-based solid particles (b) comprise, based on dry matter of the solid particles (b), 1-25 wt.%, preferably 3-15 wt.%, of protein.

In an embodiment, the plant-based solid particles (b) comprise, based on dry matter of the solid particles (b), 0-5 wt.%, preferably 0-4 wt.%, more preferably 0-3 wt.%. of lignin.

In an embodiment, the plant-based solid particles (b) comprise, based on dry matter of the solid particles (b), less than 10 wt.%, preferably less than 6 wt.%, more preferably less than 3 wt.% of sugars.

In an embodiment, the plant-based solid particles (b) comprise, based on dry matter of the solid particles (b), less than 10 wt.%, preferably less than 6 wt.%, more preferably less than 3 wt.% of sugars.

In an embodiment, the plant-based solid particles (b) comprise, based on dry matter of the solid particles (b), less than 2 wt.%, preferably less than 1 wt.%, more preferably less than 0.5 wt.% of fat.

In an embodiment, the plant-based solid particles (b) comprise, based on dry matter of the solid particles (b), less than 6 wt.%, preferably less than 5 wt.%, more preferably less than 4 wt.% of ash.

In a preferred embodiment of the invention, the constituents of the plant-based solid particles (b) are similar to that of common spent sugar beet pulp, comprising, based on dry matter of the solid particles (b), 15-35 wt.%, preferably 15-30 wt.%, more preferably 18-26 wt.% cellulose, 15-40 wt.%, preferably 20-38 wt.%, more preferably 22-35 wt.% hemicellulose, 15-35 wt.%, preferably 20-30 wt.%, more preferably 21-27 wt.% pectin, 5-15 wt.% protein, less than 5 wt.% lignin, less than 5 wt.% sugars, less than 6 wt.% of ash and less than 1 wt.% fat.

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In a preferred embodiment, the plant-based solid particles (b) essentially consist of fragments comprising clusters of collapsed/ruptured parenchymal cells, presumably with the cell wall structures largely or completely intact, *i.e.* at the primary, secondary and tertiary level.

As described hereinbefore, the particle size distribution of the plant-based solid particles (b) is determined by dry powder laser diffraction. Accordingly, the particle diameter characteristics D10, D50 and D90 are based on a volume distribution and the median particle diameter D50 is a volume median. The median particle diameter D50 is the diameter where half of the population of the plant-based solid particles (b) lies below. This volume median particle diameter is often referred to in the art as Dv50 or Dv0.5. The diameters D10 and D90 are often referred to in the art as Dv10 or Dv0.1 and Dv90 or Dv0.9, respectively. The D10 diameter is the diameter where 10% of the population of the plant-based solid particles (b) lies below. Similarly, the D90 diameter is the diameter where 90% of the population of the plant-based particles (b) lies below.

As described hereinbefore, the plant-based solid particles (b) have a particle size distribution characterized by a D90 of 300  $\mu$ m or smaller, as measured with dry powder laser diffraction. As will be appreciated by the skilled person the wording 'of xx  $\mu$ m or smaller' is identical to 'equal to or less than xx  $\mu$ m'.

In a preferred embodiment, the plant-based solid particles (b) have a particle size distribution characterized by a D90 of 300  $\mu$ m or smaller, a D50 of 150  $\mu$ m or smaller and a D10 of 50  $\mu$ m or smaller, such as a particle size distribution characterized by a D90 of between 155 and 300  $\mu$ m, a D50 of between 55 and 150  $\mu$ m and a D10 of between 5 and 50  $\mu$ m, as measured with dry powder laser diffraction.

In another preferred embodiment, the plant-based solid particles (b) have a particle size distribution characterized by a D90 of 300  $\mu$ m or smaller, a D[3,2] between 25 and 65  $\mu$ m and a D[4,3] between 80 and 160  $\mu$ m, as measured with dry powder laser diffraction.

In a more preferred embodiment, the plant-based solid particles (b) have a particle size distribution characterized by a D90 of 280  $\mu m$  or smaller, a D50 of 130  $\mu m$  or smaller and a D10 of 30  $\mu m$  or smaller, such as a particle size distribution characterized by a D90 of between 135 and 280  $\mu m$ , a D50 of between 35 and 130  $\mu m$  and a D10 of between 5 and 30  $\mu m$ , as measured with dry powder laser diffraction.

In another more preferred embodiment, the plant-based solid particles (b) have a particle size distribution characterized by a D90 of 280  $\mu$ m or smaller, a D[3,2] between 30 and 60  $\mu$ m and a D[4,3] between 90 and 140  $\mu$ m, as measured with dry powder laser diffraction.

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In an even more preferred embodiment, the plant-based solid particles (b) have a particle size distribution characterized by a D90 of 265  $\mu$ m or smaller, a D50 of 110  $\mu$ m or smaller and a D10 of 25  $\mu$ m or smaller, such as a particle size distribution characterized by a D90 of between 115 and 280  $\mu$ m, a D50 of between 30 and 110  $\mu$ m and a D10 of between 5 and 25  $\mu$ m.

In another even more preferred embodiment, the plant-based solid particles (b) have a particle size distribution characterized by a D90 of 265  $\mu$ m or smaller, a D[3,2] between 35 and 55  $\mu$ m and a D[4,3] between 105 and 135  $\mu$ m, as measured with dry powder laser diffraction.

In embodiments, the amount of plant-based solid particles (b) is 20 - 39 wt.%, 20 - 38 wt.%, 20 - 37 wt.%, 20 - 36 wt.% or 20 - 35 wt.%, based on the total weight of the dispersion.

In other embodiments, the amount of plant-based solid particles (b) is 22 - 40 wt.%, 25 - 40 wt.%, 26 - 40 wt.%, 27 - 40 wt.%, 28 - 50 wt.%, 29 - 40 wt.%, or 30 - 40 wt.%, based on the total weight of the dispersion.

#### Further ingredients

As described hereinbefore, the dispersion comprises 0 - 10 wt.% of one or more further ingredients other than liquid (a) and plant-based solid particles (b), based on the total weight of the dispersion.

As will be appreciated by the skilled person, the mere fact that the one or more further ingredients (c) are different from the liquid (a) and the plant-based solid particles (b) does not mean that the one or more further ingredients (c) cannot be liquid-like or cannot be solid particles.

Preferred examples of the one more further ingredients (c) are selected from the group consisting of dyes, pigments, dispersants, surfactants, catalysts, porogenic agents, fillers, water-binding agents, water, anti-foaming agents, flame retardants, UV stabilizers, antioxidants, adhesion promoters, plasticizers, thickeners and rheology modifiers. More preferred examples of the one more further ingredients (c) are selected from the group consisting of dyes, pigments, flame retardants, catalysts, dispersants and surfactants. Even more preferred examples of the one more further ingredients (c) are selected from the group consisting of catalysts, dispersants and surfactants.

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Pigments are examples of the one more further ingredients that take the form of solid particles. Surfactants and dispersants are examples of the one more further ingredients that may, at least partly, dissolve in the liquid (a) to constitute together with liquid (a) the liquid continuous phase of the dispersion. Surfactants and dispersants may however also, at least partly, physically attach to or adsorb onto the surface of solid particles. Dyes, anti-foaming agents, flame retardants, thickeners and rheology modifiers are examples of the one more further ingredients that preferably dissolve in the liquid (a) to constitute together with liquid (a) the liquid continuous phase of the dispersion.

In a very preferred embodiment the one or more further ingredients do not include tris(2-chloroisopropyl)phosphate (TCPP) and/or aminopropylisobutyl-polyhedral oligomeric silsesquioxanes (APIB-POSS).

If the one or more further ingredients comprise solid particles, these solid particles preferably have a particle size distribution characterized by a D90 of less than 300  $\mu$ m, more preferably less than 250  $\mu$ m, even more preferably less than 200  $\mu$ m, still more preferably less than 175  $\mu$ m, as measured with laser diffraction.

In embodiments, the amount of the one or more further ingredients (c) is 0 - 9 wt.%, 0 - 8 wt.%, 0 - 7 wt.%, 0 - 6 wt.%, 0 - 5 wt.%, 0 - 4 wt.%, 0 - 4 wt.%, 0 - 3.5 wt.% or 0 - 3 wt.%, based on the total weight of dispersion.

In other embodiments, the amount of the one or more further ingredients (c) is 0.05 - 10 wt.%, 0.1 - 10 wt.%, 0.2 - 10 wt.%, 0.5 - 10 wt.%, 1 - 10 wt.% or 1.5 - 10 wt.%, based on the total weight of the dispersion.

In still other embodiments, the amount of the one or more further ingredients (c) is 0.05 - 9 wt.%, 0.1 - 8 wt.%, 0.2 - 7 wt.%, 0.3 - 6 wt.%, 0.4 - 5 wt.% or 0.5 - 4.5 wt.%, based on the total weight of the dispersion.

# 5 Liquid continuous phase

The liquid continuous phase of the dispersion is formed by the liquid (a), by any liquid further ingredient (c) that is added to it and further by any further ingredient that dissolves in liquid (a).

#### 10 Dispersed phase

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The dispersed phase of the dispersion is formed by solid particles (b), by any further ingredient (c) in particulate form and further by any further ingredient (c) that physically adsorbs onto the solid particles (b) and/or onto any further ingredient (c) in particulate form.

#### 15 Rheological behaviour and stability of the dispersion

Fluid materials, by definition, are systems which flow when subjected to stress. How they respond to an input stress is the heart of rheological testing. In general, when a material has a uniform phase, such as a solution or pure substance, it is referred to as a simple fluid. Materials which contain more than one phase, such as solid particles dispersed in a liquid, are considered structured fluids since their rheological behaviour is in general dominated by the interactions of the constituents.

Many factors may affect the stability of structured fluids. The viscosity of the liquid phase in dispersions usually plays an important role on the flow properties of the material. Dispersions have wide variations in performance depending on particle size, shape, concentration, and any interaction of the dispersed particles with the continuous phase in which they are suspended.

Nearly all structured fluids have a viscosity that drops at higher shear rates. This is the phenomenon of shear thinning which becomes more pronounced as the volume concentration of solid particles increases.

For many fluid materials, viscosity is mostly independent of time, and is only a function of the shear rate and temperature. For concentrated dispersions, however, the viscosity often does not reach a steady value for some time upon application of shear rate. This steady state is dependent on the stabilization of internal network structures that can be broken down by shearing, and require time to rebuild. Upon ceasing the shear rate which caused the breakdown, the material reforms its internal network, and the viscosity recovers. The ability of a fluid

material to exhibit shear thinning with increased shear rate and time-dependent recovery with decreasing shear rate is called 'thixotropy'. Thus, thixotropic materials lose structure during shear, and rebuild it on standing.

Therefore, viscosity of dispersions, or structured fluids in general, should be measured not only as a function of increasing shear rate while the structure is broken down but also as a function of decreasing shear rate while the system recovers its structure. If the recovery is fast, the plot of viscosity as a function of decreasing shear rate would be superimposed on the plot of viscosity as a function of increasing shear rate. However, in thixotropic systems it takes time for the fluid to restore its structure and therefore the 'down curve' lies below the 'up curve'. This phenomenon is known as the 'hysteresis effect'.

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As described hereinbefore, the dispersions as described herein need to be sufficiently stable as regards sedimentation of the plant-based solid particles (b). The dispersion should be homogeneous for a sufficiently long time to be able to homogeneously mix it with further ingredients that are required for the intended application. For example, the dispersion should be homogeneous for a sufficiently long time to be able to homogeneously mix it with isocyanates to provide a homogeneous distribution of the plant-based solid particles (b) in the reaction mixture and in the final polyurethane.

The inventors found that the dispersions according to the invention having a high content of solids exhibit shear thinning behaviour with hysteresis (thixotropy). Accordingly, at sufficiently high content of solids, the plant-based solid particles (b) are stabilized in an internal network structure that can be broken down by shearing the dispersion. At low shear rates, the viscosity of the dispersions according to the invention is sufficiently high to reduce sedimentation of the solid particles (b) to acceptable levels. At high shear rates, the viscosity of the dispersions is sufficiently low to be able to process the dispersions, for example by pumping the dispersion to a reactor vessel. In this respect, reference is made to the appended examples.

The dispersions according the invention do exhibit sedimentation over time. This means that the dispersions may need to be homogenized, for example using gentle stirring, before applying them, for example in a polyurethane system. Although, the dispersions according to the invention exhibit thixotropic behaviour, the internal network is reformed sufficiently quick after a breakdown due to stirring to stabilize the dispersion again as regards sedimentation of the plant-based solid particles (b). In this respect, reference is made to the appended examples.

It was, however, found that the degree of shear thinning and hysteresis strongly depends on the content of the plant-based solid particles (b) in the dispersion and on the viscosity of the liquid (a). Higher loads of plant-based solid particles (b) resulted in more stable dispersions. The appended examples show that for a liquid (a) having a viscosity of about 0.2 Pa·s, more than 30 wt.% of plant-based solid particles (b) are needed to provide sufficient dispersion stability as regards sedimentation. For a liquid (a) having a viscosity of about 5 Pa·s, more than 20 wt.% of solid particles (b) were needed to provide sufficient dispersion stability as regards sedimentation.

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In general, it was found that dispersions having a viscosity of at least 20 Pa·s at 20 °C and at a shear rate of 0.1 s<sup>-1</sup> exhibited sufficient stability against sedimentation.

In preferred embodiments, the dispersion as defined hereinbefore has a viscosity at 20 °C and at a shear rate of 0.1 s<sup>-1</sup> of at least 25 Pa·s, at least 30 Pa·s, at least 40 Pa·s, at least 50 Pa·s, at least 75 Pa·s, or at least 100 Pa·s.

In other preferred embodiments, the dispersion as defined hereinbefore has a viscosity at 20 °C and at a shear rate of 0.1 s<sup>-1</sup> of between 20 and 1500 Pa·s, of between 25 and 1500 Pa·s, between 30 and 1500 Pa·s, between 40 and 1500 Pa·s, between 50 and 1500 Pa·s, between 75 and 1500 Pa·s, or between 100 and 1500 Pa·s.

In still other preferred embodiments, the dispersion as defined hereinbefore has a viscosity at 20 °C and at a shear rate of 0.1 s<sup>-1</sup> of between 25 and 1400 Pa·s, between 25 and 1200 Pa·s, between 25 and 1000 Pa·s, between 25 and 800 Pa·s, between 25 and 600 Pa·s, or between 25 and 400 Pa·s.

In preferred embodiments, the dispersion as defined hereinbefore has a viscosity at 20 °C of: (i) at least 20 Pa·s at a shear rate of 0.1 s<sup>-1</sup> and between 0.5 and 17 Pa·s at a shear rate of 1000 s<sup>-1</sup>; (ii) at least 25 Pa·s at a shear rate of 0.1 s<sup>-1</sup> and between 1 and 20 Pa·s at a shear rate of 1000 s<sup>-1</sup>; (iii) at least 30 Pa·s at a shear rate of 0.1 s<sup>-1</sup> and between 1.5 and 25 Pa·s at a shear rate of 1000 s<sup>-1</sup>; (iv) at least 40 Pa·s at a shear rate of 0.1 s<sup>-1</sup> and between 2 and 30 Pa·s at a shear rate of 1000 s<sup>-1</sup>; (v) at least 50 Pa·s at a shear rate of 0.1 s<sup>-1</sup> and between 2 and 35 Pa·s at a shear rate of 1000 s<sup>-1</sup>; (vi) at least 75 Pa·s at a shear rate of 0.1 s<sup>-1</sup> and between 2 and 40 Pa·s at a shear rate of 1000 s<sup>-1</sup>; (vii) at least 100 Pa·s at a shear rate of 0.1 s<sup>-1</sup> and between 2 and 45 Pa·s at a shear rate of 1000 s<sup>-1</sup>; (viii) between 20 and 1500 Pa·s at a shear rate of 0.1 s<sup>-1</sup> and between 0.5 and 100 Pa·s at a shear rate of 1000 s<sup>-1</sup>; (viii) between 20 and 1500 Pa·s at a shear rate of 0.1 s<sup>-1</sup> and

rate of 0.1 s<sup>-1</sup> and between 0.5 and 95 Pa·s at a shear rate of 1000 s<sup>-1</sup>; (x) between 20 and 1200 Pa·s at a shear rate of 0.1 s<sup>-1</sup> and between 0.5 and 90 Pa·s at a shear rate of 1000 s<sup>-1</sup>; (xi) between 20 and 1000 Pa·s at a shear rate of 0.1 s<sup>-1</sup> and between 0.5 and 85 Pa·s at a shear rate of 1000 s<sup>-1</sup>; (xii) between 20 and 800 Pa·s at a shear rate of 0.1 s<sup>-1</sup> and between 0.5 and 80 Pa·s at a shear rate of 1000 s<sup>-1</sup>; (xiii) between 20 and 600 Pa·s at a shear rate of 0.1 s<sup>-1</sup> and between 0.5 and 75 Pa·s at a shear rate of 1000 s<sup>-1</sup>; or (xiv) between 20 and 400 Pa·s at a shear rate of 0.1 s<sup>-1</sup> and between 0.5 and 70 Pa·s at a shear rate of 1000 s<sup>-1</sup>.

#### Process for the preparation of the dispersion

A second aspect of the invention concerns a process for the production of the dispersion as defined hereinbefore, said process comprising:

- (i) providing liquid (a);
- (ii) adding the plant-based solids particles (b) and the optional one or more further ingredients (c); and
- (iii) stirring the mixture obtained in step (ii) until a homogeneous dispersion is obtained.

This process is preferably performed at a temperature between 5 and 50  $^{\circ}$ C, more preferably between 15 and 35  $^{\circ}$ C.

The one or more further ingredients (c) can be added to liquid (a) before or after addition of the plant-based solids particles (b) to liquid (a). Since addition of the plant-based solids particles (b) to liquid (a) increases the viscosity, addition of the one or more further ingredients (c) to liquid (a) prior to addition of the plant-based solids particles (b) is preferred.

#### <u>Uses</u>

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A third aspect of the invention concerns the use of the dispersion as defined hereinbefore as a feedstock in a process for the production of a polymer.

This third aspect of the invention can also be worded as a process for the production of a polymer using the dispersion as defined hereinbefore as a feedstock.

In a preferred embodiment, this aspect concerns the use of the dispersion as a flame retardant in a process for the production of a polymer.

A fourth aspect of the invention concerns the use of the dispersion as defined hereinbefore as a flame retardant in a polymer.

In a preferred embodiment, the polymer is chosen from the group consisting of polyurethanes, polyisocyanurates, polyesters, polyethers, polycarbonates and polyamides.

In another preferred embodiment, the polyurethanes are polyurethane foams or polyurethane adhesives.

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Thus, the invention has been described by reference to certain embodiments discussed above. It will be recognized that these embodiments are susceptible to various modifications and alternative forms well known to those of skill in the art.

Furthermore, for a proper understanding of this document and its claims, it is to be understood that the verb 'to comprise' and its conjugations are used in its non-limiting sense to mean that items following the word are included, but items not specifically mentioned are not excluded. In addition, reference to an element by the indefinite article 'a' or 'an' does not exclude the possibility that more than one of the element is present, unless the context clearly requires that there be one and only one of the elements. The indefinite article 'a' or 'an' thus usually means 'at least one'.

#### **EXAMPLES**

#### Measurement protocols

The following analytical protocols were applied in the examples.

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#### Measurement of the viscosity and hysteresis

The dynamic viscosity and hysteresis of samples was measured at 20 °C using a cup and vane rotor relative geometry at a shear rate of between 0.1 s<sup>-1</sup> and 1000 s<sup>-1</sup>.

More particularly, a Physica MCR301 rheometer (Anton Paar, Germany) with a stainless steel C-CC27/T200/SS 45971 measuring cup (diameter 28.918 mm) and a ST24-2D/2V/2V-30-SN26943 vane rotor was used.

The dynamic viscosity of the different polyols at 20 °C was determined by incrementally increasing the shear rate from 0.1 s<sup>-1</sup> to 1000 s<sup>-1</sup> in 22 steps that are equidistant on a log scale, to obtain the dynamic viscosity at 23 different shear rates.

The dynamic viscosity and hysteresis of samples at 20 °C was measured by the following subsequent steps:

- (i) stirring the sample to get a good dispersion;
- 20 (ii) filling the cup with the dispersion;
  - (iii) waiting 25 seconds before starting the measurements;
  - (iv) measuring the viscosity by incrementally increasing the shear rate from 0.1 s<sup>-1</sup> to 1000 s<sup>-1</sup> in 22 steps that are equidistant on a log scale to obtain the dynamic viscosity at 23 different shear rates; and
- 25 (v) measuring the viscosity again by incrementally decreasing the shear rate from 1000 s<sup>-1</sup> to 0.1 s<sup>-1</sup> in 22 steps that are equidistant on a log scale to obtain the dynamic viscosity at 23 different shear rates.

#### Measurement of thixotropic behaviour

Thixotropic behaviour was tested by subsequently applying three shear rate regimes and by measuring the viscosity at 20 °C as a function of time using a Physica MCR301 rheometer (Anton Paar, Germany) with a stainless steel C-CC27/T200/SS 45971 measuring cup (diameter 28.918 mm) and a ST24-2D/2V/2V-30-SN26943 vane rotor.

The detailed protocol comprises the following steps:

- (i) stirring the sample to get a good dispersion;
- (ii) filling the cup with the dispersion;

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- (iii) pre-shear the sample at a shear rate of 1 s<sup>-1</sup> for 30 seconds;
- (iv) measuring, in a first regime, the viscosity at a shear rate of 1 s<sup>-1</sup> for 2 minutes at a sampling interval of 1 second;
- (v) measuring, in a second regime, the viscosity at a shear rate of 200 s<sup>-1</sup> for 1 minute at a sampling interval of 1 second; and
- (vi) measuring, in a third regime, the viscosity again at a shear rate of 1 s<sup>-1</sup> for 10 minutes at a sampling interval of 1 second.

#### Measurement of dispersion stability

Stability of the dispersions was tested by measuring turbidity at room temperature (~20 °C) as a function of time in a Turbiscan® LAB<sup>TM</sup> using Turbisoft 2.0, Formulaction, France. The stability of the dispersions was measured by the following subsequent steps:

- 15 (i) gently shake the dispersion to be analysed;
  - (ii) take a new measuring cell with a cap (both disposable parts of the Turbiscan® LAB<sup>TM</sup>);
  - (iii) place the measuring cell in the provider holder of the Turbiscan® LAB<sup>TM</sup>;
  - (iv) fill the measuring cell with about 20 ml of the dispersion obtained in step (i);
  - (v) close the measuring cell with the cap;
- 20 (vi) measure the transmission and backscattering of the measuring cell as a function of time and height in the measuring cell and compute the Turbiscan® Stability Index (TSI) in the top part of the measuring cell from these data using Turbisoft 2.0 software.

The TSI [-] is calculated from transmission and backscattering signals. It sums up all the variations in the sample, to give as a result a unique number reflecting the destabilization of a given sample. The higher the TSI, the stronger is the destabilization in the sample.

#### Measurement of particle size

The particle size distribution was measured with dry powder laser diffraction, more particularly with a Malvern Mastersizer 3000 equipped with an Aero S automated dry powder dispersion unit. The specific settings were: air pressure 3.5, feed rate 90, refractive index 1.53, particle density 1 and absorption index 0.1.

Using dry powder laser diffraction, D10, D50, D90, D[3,2] and [D4,3] values were obtained.

#### Measurement of moisture content

Moisture (water) content of the solid particles was determined by measuring the loss of mass on drying using the following protocol:

- 5 (i) dry a container for at least 30 minutes at 105 °C;
  - (ii) place the container in a desiccator and cool the container during 30 minutes, wherein the valve of the desiccator is open during the first 5 minutes and is closed during the following 25 minutes;
  - (iii) Measure the weight of the empty container (accuracy 0.1 mg, weight=G1);
- 10 (iv) Fill the container with about 5 gram of the sample and measure the weight of the filled container (accuracy 0.1 mg, weight=G2);
  - (v) Subject the filled container to 3 hours of drying in a drying chamber at 105 °C;
  - (vi) Cool the filled container during 30 minutes in a desiccator, wherein the valve of the desiccator is open during the first 5 minutes and is closed during the following 25 minutes;
- 15 (vii) Measure the weight of the filled container after drying (accuracy 0.1 mg, weight=G3);
  - (viii) Perform duplo measurement; and
  - (ix) Calculate the moisture content (wt.%) of the solid particles from: (G2 G3) / (G2 G1) \* 100%.

#### 20 Materials

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The following materials were applied in the examples.

#### Plant-based solid particles (b)

- Milled dry wood chips from pinewood. The milled wood chips from pine wood were sieved using a sieve with 100 μm sieve openings (Retsch, Germany). The fraction passing the sieve (<100 μm) was used in the examples.
- BetaBind-A<sup>®</sup>, batch no 120432, obtained from Cosun Beet Company, The Netherlands. The particle size distribution of BetaBind-A<sup>®</sup> was measured in accordance with the protocol as defined hereinbefore. Characteristics are presented in Table 1. BetaBind-A<sup>®</sup> is obtained by drying and milling spent sugar beet pulp. No additives or chemicals are used during its preparation from spent sugar beet pulp. BetaBind-A<sup>®</sup> had a water content of 4.06 wt.%, as measured in accordance with the protocol as defined hereinbefore. Betabind-A<sup>®</sup> mainly comprises parenchymal cell wall materials. More in particular, Betabind-A<sup>®</sup> comprises, based on dry matter, 15-35 wt.% cellulose, 22-35 wt.%,

hemicellulose, 20-30 wt.%, pectin, less than 5 wt.% lignin, 5-15 wt.% protein, less than 5 wt.% sugars, less than 1 wt.% fat and less than 6 wt.% ash.

- BetaBind-A<sup>®</sup> Small fraction. This small particles fraction was obtained using sieving of BetaBind-A<sup>®</sup>. The small particles fraction was the fraction passing a 212 μm sieve (Retsch, Germany). The particle size distribution of 'BetaBind-A<sup>®</sup> Small fraction' was measured in accordance with the protocol as defined hereinbefore. Characteristics are presented in Table 1. 'BetaBind-A<sup>®</sup> Small fraction' had a water content of 2.93 wt.%, as measured in accordance with the protocol as defined hereinbefore.
- BetaBind-A® Large fraction. This large particles fraction was obtained using sieving of BetaBind-A®. The large particles fraction was the fraction passing a 355 μm sieve (Retsch, Germany) and being left on a 212 μm sieve (Retsch, Germany). The particle size distribution of 'BetaBind-A® Large fraction' was measured in accordance with the protocol as defined hereinbefore. Characteristics are presented in Table 1. 'BetaBind-A® Large fraction' had a water content of 3.46 wt.%, as measured in accordance with the protocol as defined hereinbefore.

Table 1:

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Sample	D10	D50	D90	D[3,2]	D[4,3]
	[µm]	[µm]	[µm]	[µm]	[µm]
BetaBind-A®	26.8	330	890	59.5	395
'BetaBind-A® Small fraction'	22.7	105	253	43.1	123
'BetaBind-A® Large fraction'	202	330	524	203	348

### Liquid (a): polyols

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• Polyol A: Stepanpol® PS-3152, obtained from Stepan Company, US.

This is a diethylene glycol-phthalic anhydride-based polyester polyol, having an average molecular weight of 174 g/mol and an average hydroxy functionality of 2.0. Polyol A has a hydroxyl number of 300-330 mg KOH/g and a viscosity at 25 °C (as measured by Stepan Company) of 2.677 Pa·s. The viscosity of polyol A at 20 °C, as measured with the protocol as defined hereinbefore, showed Newtonian behaviour and was about 4.9 Pa·s.

• Polyol B: Voranol<sup>TM</sup> RH 360, obtained from Dow Chemical Company, US.

This is a sucrose/glycerine initiated polyether polyol, having an average hydroxy functionality of 4.5. Polyol B has a hydroxyl number of 345-375 mg KOH/g as measured using ASTM

D4274-94d and a viscosity at 25 °C (as measured by Dow Chemical Company) of between 2.5 and 3.5 Pa·s, as measured using ASTM D445-94. The viscosity of polyol B at 20 °C, as measured with the protocol as defined hereinbefore, showed Newtonian behaviour and was about 5.2 Pa·s.

• Polyol C: Voranol<sup>TM</sup> 1010 L, obtained from Dow Chemical Company, US.

This polyol is a polypropylene glycol (propyleneglycol initiated, 1000 g/mol, homopolymer diol), having an average hydroxy functionality of 2. Polyol C has a hydroxyl number of 106-114 mg KOH/g as measured using ASTM D4274-94d and a viscosity at 25 °C (as measured by Dow Chemical Company) of 140 mPa·s, as measured using ASTM D445-94. The viscosity of polyol C at 20 °C, as measured with the protocol as defined hereinbefore, showed Newtonian behaviour and was about 0.21 Pa·s.

#### Example 1

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The following dispersions were made (see Table 2) with the ingredients described *supra*. The dispersions were produced by weighing 18 gram of plant-based solid particles (b) in a Duran bottle of 100 ml and by adding 72 gram of liquid (a) to prepare a 20 wt.% mixture of plant-based solid particles (b), based on the weight of the dispersion. Mixtures with other weight percentages of plant-based solid particles (b) were prepared in a similar way. The mixtures were stirred to prepare dispersions. For polyols A and B, a mixer was needed to prepare a dispersion. It was observed that preparing a dispersion was more difficult for smaller particles (BetaBind-A® Small fraction) than for larger particles (BetaBind-A® Large fraction). The samples with BetaBind-A® Large fraction are not according to the invention.

Table 2

Dispersion	Liquid (a)	Solid particles (b)	Content Solid particles (b) [wt.%]
A-5wt.% Small	Polyol A	BetaBind-A® Small fraction	5
A-10wt.% Small	Polyol A	BetaBind-A® Small fraction	10
A-20wt.% Small	Polyol A	BetaBind-A® Small fraction	20
A-30wt.% Small	Polyol A	BetaBind-A® Small fraction	30
A-20wt.% Large	Polyol A	BetaBind-A® Large fraction	20
B-5wt.% Small	Polyol B	BetaBind-A® Small fraction	5
B-10wt.% Small	Polyol B	BetaBind-A® Small fraction	10

B-20wt.% Small	Polyol B	BetaBind-A® Small fraction	20
B-25wt.% Small	Polyol B	BetaBind-A® Small fraction	25
B-10wt.% Large	Polyol B	BetaBind-A® Large fraction	10
B-25wt.% Large	Polyol B	BetaBind-A® Large fraction	25
C-15wt.% Small	Polyol C	BetaBind-A® Small fraction	15
C-25wt.% Small	Polyol C	BetaBind-A® Small fraction	25
C-28wt.% Small	Polyol C	BetaBind-A® Small fraction	28
C-30wt.% Small	Polyol C	BetaBind-A® Small fraction	30
C-33wt.% Small	Polyol C	BetaBind-A® Small fraction	33
C-25wt.% Large	Polyol C	BetaBind-A® Large fraction	25
C-33wt.% Large	Polyol C	BetaBind-A® Large fraction	33

Stability of the dispersions at room temperature (~20 °C) was measured as a function of time using the protocol as described hereinbefore. Results are graphically presented in Figure 1 (dispersions with Polyol A), Figure 2 (dispersions with Polyol B) and Figure 3 (dispersions with Polyol C).

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The viscosity and hysteresis of the dispersions was measured at 20 °C using the protocol as described hereinbefore. Results are graphically presented in Figures 4-5 (dispersions with Polyol A), Figures 6-7 (dispersions with Polyol B) and Figures 8-9 (dispersions with Polyol C).

It can be concluded from Figures 5, 7 and 9 that dispersions with the same weight percentage of plant-based solid particles (b) and only differing in particle size show rather similar shear thinning and hysteresis profiles. However, it follows from Figures 1-3 that dispersions with the same weight percentage of plant-based solid particles (b) and only differing in particle size show different stability as regards sedimentation. The dispersions with BetaBind-A® Large fraction (not according to the invention) are far less stable than dispersions according to the invention comprising BetaBind-A® Small fraction.

It can further be concluded from Figures 1-3 that for Polyol A, B and C, the stability of the dispersions according to the invention improves with increasing weight percentage of BetaBind-A<sup>®</sup> Small fraction.

It follows from Figure 4 that dispersions comprising BetaBind-A® small fraction and Polyol A only start to show substantial shear thinning behaviour with increased viscosity at low shear rates when 20 wt.% or more of the BetaBind-A® Small fraction is used. The same

observation holds true for dispersions comprising BetaBind-A® Small fraction and Polyol B (see Figure 6). Polyol A and B have similar viscosities. Polyol C has a much lower viscosity. It follows from Figure 8 that dispersions comprising BetaBind-A® Small fraction and Polyol C only start to show substantial shear thinning behaviour with increased viscosity at low shear rates when 30 wt.% or more of the BetaBind-A® Small fraction is used. It can be concluded from combined Figures 1-4, 6 and 8 that only the dispersions comprising BetaBind-A® Small fraction that show substantial shear thinning behaviour with increased viscosity at low shear rates exhibit sufficient stability as regards sedimentation. These sufficiently stable dispersions have in common that their viscosity at low shear rates (0.1 s<sup>-1</sup>) is above 20 Pa·s, as measured with a C-CC27/T200/SS 45971 measuring cup and a ST24-2D/2V/2V-30-SN26943 vane rotor.

Thixotropic behaviour was measured at 20 °C using the protocol as described hereinbefore for samples A-20wt.% Small, B-20wt.% Small, B-25wt.% Small and C-30wt.% Small. Results are graphically represented in Figure 10. As can be inferred from Figure 10, the dispersions all recover at least 50% of the viscosity measured in the first regime within a few seconds from the start of the third regime. Moreover, the dispersions are able to regain about 90% of the value of the viscosity of the first regime within 10 seconds. Consequently, the internal network is reformed sufficiently quick after a breakdown due to stirring to stabilize the dispersion again as regards sedimentation of the plant-based solid particles (b).

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#### **Comparative Example**

It was tried to make a dispersion of 20 wt.% of the milled dry wood chips as defined hereinbefore in Polyol A. No dispersions were, however, obtained, not even with intense stirring. Without wishing to be bound by any theory, it is hypothesized that Polyol A does not substantially wet the rather apolar lignin-rich dry wood chips.

#### Example 3

Two polyurethane foams were produced using dispersions according to the invention (20 wt.% and 30 wt.% BetaBind-A® small fraction, based on the weight of the dispersion, in Polyol A). The 'further ingredients' in the dispersion were DMCHA as catalyst (N,N-dimethylcyclohexylamine), K-octoate (AKDRY K Octoate %15, Akpa), traces of water and surfactant Tegostab® B 8491 (Evonik). The total amount of further ingredients in the dispersion was about 4 wt.%, based on the total weight of the dispersion. To 126 parts of the dispersion, 16.5 parts of cyclopentane as a blowing agent and 194 parts of polymeric 4,4 -diphenylmethane

diisocyanate (pMDI, Voranate<sup>TM</sup> M 647, Dow) were added. The ingredients were hand mixed and allowed to react. The tack free time was between 60 and 75 seconds.

A comparative polyurethane foam was produced that was similar to the polyurethane foams produced using a dispersion according to the invention. The only difference was that the 20 wt.% BetaBind-A<sup>®</sup> small fraction, based on the weight of the dispersion, was replaced with 20 wt.% of the flame retardant tris(2-chloroisopropyl)phosphate (TCPP, obtained from Supelco), based on the weight of the dispersion. TCPP is a toxic flame retardant commonly used in polyurethane foam in consumer products, in home insulation, and in electronics. It is used as an additive to polyurethane foam, it is not chemically bound and it escapes from products into the indoor environment. The tack free time was about 75 seconds.

The two polyurethane foams produced using dispersions according to the invention and the comparative polyurethane foam were subjected to B2 flame retardancy tests according to DIN 4102. It was concluded that both the polyurethane foams produced using dispersions according to the invention and the comparative polyurethane foam met the B2 flame retardancy rating of DIN 4102. Accordingly, the B2 flame retardancy rating of DIN 4102 can be realized by replacing the toxic flame retardant TCPP in polyurethane foams with the plant-based solid particles as defined herein.

#### **CONCLUSIES**

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- 1. Dispersie van een vloeibare continue fase met vaste stof daarin gedispergeerd, waarbij de dispersie bestaat uit:
  - (a) 50 80 gew.% van een vloeistof, gebaseerd op het gewicht van de dispersie;
  - (b) 20 40 gew.% van plantaardige vaste deeltjes, gebaseerd op het gewicht van de dispersie; en
  - (c) 0 10 gew.% van één of meerdere verdere ingrediënten anders dan (a) en (b), gebaseerd op het totaalgewicht van de dispersie,
- waarbij de plantaardige vaste deeltjes (b), gebaseerd op droog stof van de vaste deeltjes (b), ten minste 70 gew.% parenchymaal celwandmateriaal omvatten en minder dan 6 gew.% lignine,

waarbij de plantaardige vaste deeltjes (b), gebaseerd op het gewicht van de vaste deeltjes (b), minder dan 7 gew.% water omvatten,

waarbij de plantaardige vaste deeltjes (b) een deeltjesgrootteverdeling hebben die wordt gekarakteriseerd door een D90 van 300 µm of kleiner, zoals gemeten met droog poeder laserdiffractie,

waarbij de vloeistof (a) bestaat uit één of meer polyolverbindingen, en waarbij de viscositeit van de dispersie bij 20 °C en bij een afschuifsnelheid van 0.1 s<sup>-1</sup> ten minste 20 Pa·s bedraagt, zoals gemeten met een C-CC27/T200/SS 45971 measuring cup en een ST24-2D/2V/2V-30-SN26943 vane rotor.

- 2. Dispersie volgens conclusie 1, waarin de één of meer polyolverbindingen die vloeistof (a) vormen een hydroxylgetal van 50 mg KOH/g tot 550 mg KOH/g hebben, bij voorkeur 100 tot 550 mg KOH/g, zoals gemeten in overeenstemming met ASTM D4274 16.
- 3. Dispersie volgens conclusie 1 or 2, waarin de vloeistof (a) die bestaat uit de één of meer polyolverbindingen een viscositeit heeft tussen 0.1 en 15 Pa·s, bij voorkeur tussen 0.2 en 10 Pa·s, bij 20 °C en bij een afschuifsnelheid van 0.1 s<sup>-1</sup>, zoals gemeten met een C-CC27/T200/SS 45971 measuring cup en een ST24-2D/2V/2V-30-SN26943 vane rotor.
- 4. Dispersie volgens één van de conclusies 1 tot 3, waarin de één of meer polyolverbindingen die vloeistof (a) vormen worden gekozen uit de groep bestaande uit pentaerytritol, trimethylolpropaan, glycolen, (hydroxy gealkyleerde) suikeralcoholen, glycerine, (gemodificeerde) polyetherpolyolen, (gemodificeerde) alifatische en aromatische

polyesterpolyolen, polycarbonaatpolyolen en polyolen afgeleid van plantaardige natuurlijke oliën.

- 5. Dispersie volgens één van de conclusies 1 tot 4, welke een viscositeit bij 20 °C en bij een afschuifsnelheid van 0.1 s<sup>-1</sup> van ten minste 25 Pa·s heeft, ten minste 30 Pa·s, ten minste 40 Pa·s, ten minste 50 Pa·s, ten minste 75 Pa·s, of ten minste 100 Pa·s, zoals gemeten met een C-CC27/T200/SS 45971 measuring cup en een ST24-2D/2V/2V-30-SN26943 vane rotor.
- 6. Dispersie volgens één van de conclusies 1 tot 5, welke een viscositeit bij 20 °C en bij een afschuifsnelheid van 0.1 s<sup>-1</sup> tussen 25 en 1400 Pa·s heeft, tussen 25 en 1200 Pa·s, tussen 25 en 1000 Pa·s, tussen 25 en 800 Pa·s, tussen 25 en 600 Pa·s, of tussen 25 en 400 Pa·s, zoals gemeten met een C-CC27/T200/SS 45971 measuring cup en een ST24-2D/2V/2V-30-SN26943 vane rotor.
- 7. Dispersie volgens één van de conclusies 1 tot 6, waarin de plantaardige vaste deeltjes (b) een deeltjesgrootteverdeling hebben die wordt gekarakteriseerd door een D90 van 300 μm of kleiner, een D50 van 150 μm of kleiner en een D10 van 50 μm of kleiner.
- Dispersie volgens één van de conclusies 1 tot 7, waarin de plantaardige vaste deeltjes (b) een deeltjesgrootteverdeling hebben die wordt gekarakteriseerd door een D90 tussen 155 en 300 μm, een D50 tussen 55 en 150 μm en een D10 tussen 5 en 50 μm, zoals gemeten met droog poeder laserdiffractie.
- Dispersie volgens één van de conclusies 1 tot 7, waarin de plantaardige vaste deeltjes (b) een
   deeltjesgrootteverdeling hebben die wordt gekarakteriseerd door een D90 van 265 μm of kleiner, een D50 van 110 μm of kleiner en een D10 van 25 μm of kleiner.
  - 10. Dispersie volgens één van de conclusies 1 tot 9, waarin de één of meerdere verdere ingrediënten (c) worden gekozen uit de groep bestaande uit kleurstoffen, pigmenten, dispersanten, oppervlakte-actieve stoffen, katalysatoren, porogene middelen, vulstoffen, water-bindende middelen, water, antischuimmiddelen, vlamvertragers, UV stabilisatoren, antioxidanten, adhesiepromotoren, weekmakers, verdikkers en reologiemodificatoren.

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11. Dispersie volgens één van de conclusies 1 tot 10, waarin het gehalte van de één of meerdere verdere ingrediënten (c) 0 – 9 gew.% bedraagt, 0 - 8 gew.%, 0 – 7 gew.%, 0 - 6 gew.%, 0 –

5 gew.%, 0 - 4.5 gew.%, 0 - 4 gew.%, 0 - 3.5 gew.% of 0 - 3 gew.%, gebaseerd op het totaalgewicht van de dispersie.

- 12. Dispersie volgens één van de conclusies 1 tot 11, waarin de plantaardige vaste deeltjes (b) worden verkregen door of verkrijgbaar zijn door een werkwijze bestaande uit:
  - (A) het drogen van natte gebruikte suikerbietenpulp, natte pulp van citrusvruchten, natte pulp van tomaten, natte gebruikte pulp van cichorei, natte pulp van aardappelen, natte pulp van ananas, natte pulp van appel, natte pulp van veenbessen, natte pulp van druiven, en/of natte pulp van wortelen (exclusief de stengel en de bladeren), met meer voorkeur verkregen uit natte gebruikte suikerbietenpulp en/of natte gebruikte pulp van cichorei, met nog meer voorkeur verkregen uit natte gebruikte suikerbietenpulp, gedurende welke stap de temperatuur van de pulp bij voorkeur niet boven 95 °C uitkomt;
  - (B) het malen of verpulveren van de gedroogde pulp verkregen in stap (A); en
  - (C) het optioneel onderwerpen van de gemalen of verpulverde gedroogde pulp verkregen in stap (B) aan zeven.
- 13. Dispersie volgens één van de conclusies 1 tot 12, waarin de plantaardige vaste deeltjes (b), gebaseerd op droge stof van de vaste deeltjes (b), 15-35 gew.%, bij voorkeur 15-30 gew.%, met meer voorkeur 18-26 gew.% cellulose omvatten, 15-40 gew.%, bij voorkeur 20-38 gew.%, met meer voorkeur 22-35 gew.% hemicellulose, 15-35 gew.%, bij voorkeur 20-30 gew.%, met meer voorkeur 21-27 gew.% pectine, 5-15 gew.% eiwit, minder dan 5 gew.% lignine, minder dan 5 gew.% suikers en minder dan 1 gew.% vet
- 25 14. Werkwijze voor de vervaardiging van een dispersie zoals gedefinieerd in één van de conclusies 1 tot 13, waarbij de werkwijze de volgende stappen omvat:
  - (i) het verschaffen van vloeistof (a);
  - (ii) het toevoegen van de plantaardige vaste deeltjes (b) en de optionele één of meerdere verdere ingrediënten (c); en
- 30 (iii) her roeren van het mengsel verkregen in stap (ii) tot een homogene dispersie wordt verkregen.
  - 15. Toepassing van de dispersie zoals gedefinieerd in één van de conclusies 1 tot 13 als een grondstof in een werkwijze voor de vervaardiging van een polymeer.

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- 16. Toepassing van de dispersie zoals gedefinieerd in één van de conclusies 1 tot 13 als een vlamvertrager in een polymeer.
- Toepassing volgens conclusie 15 of 16, waarbij het polymeer wordt gekozen uit de groep
   bestaande uit polyurethanen, polyisocyanuraten, polyesters, polyethers, polycarbonaten en polyamides.

Fig. 1

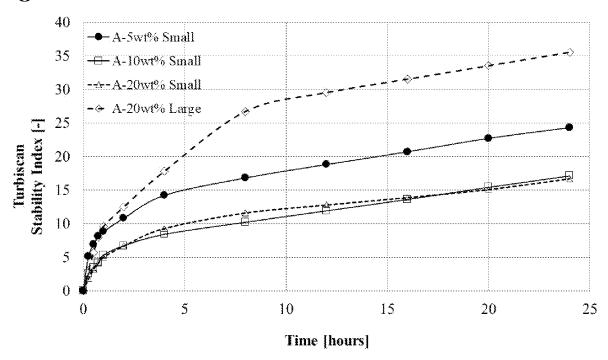
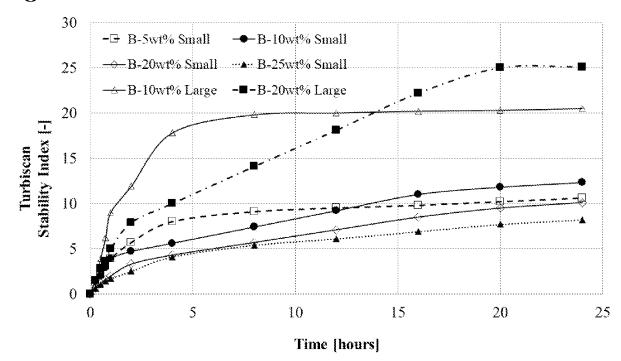
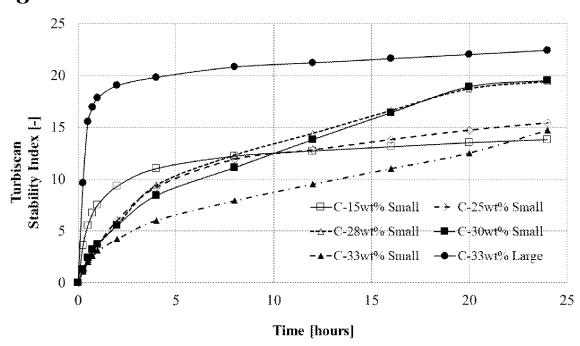


Fig. 2



*Fig.* 3





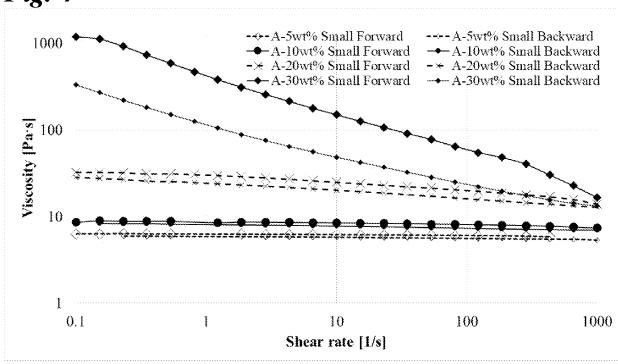


Fig. 5

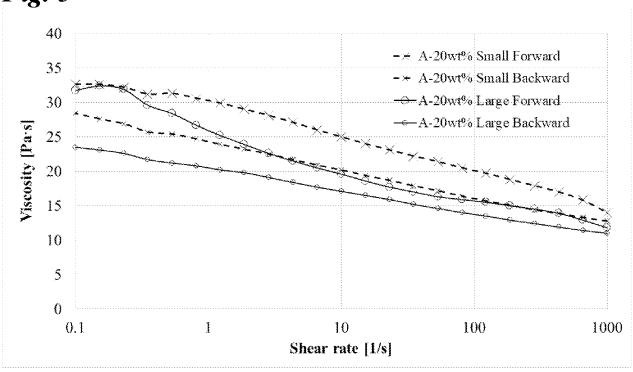
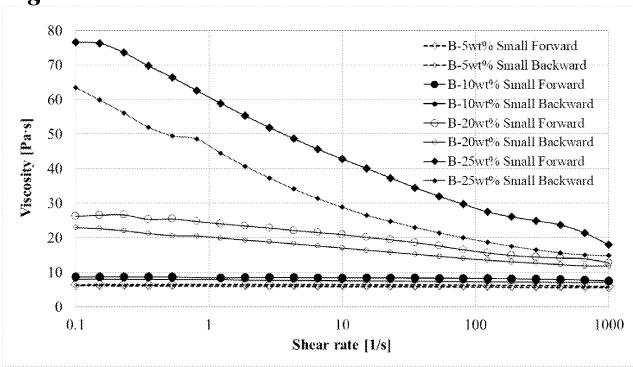


Fig. 6





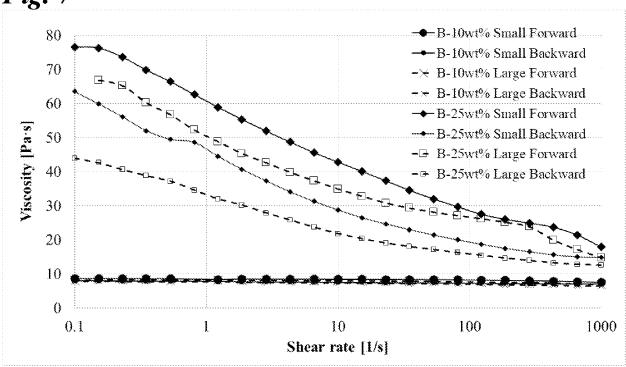


Fig. 8

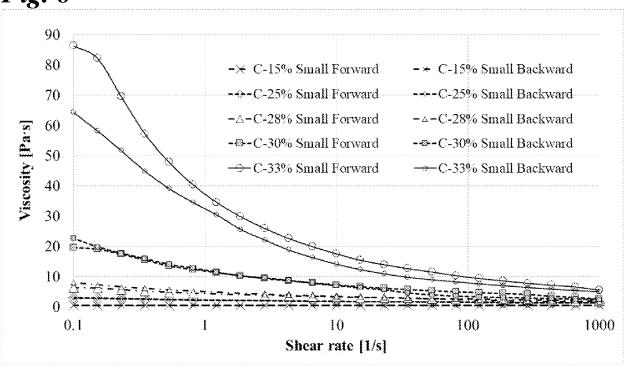


Fig. 9

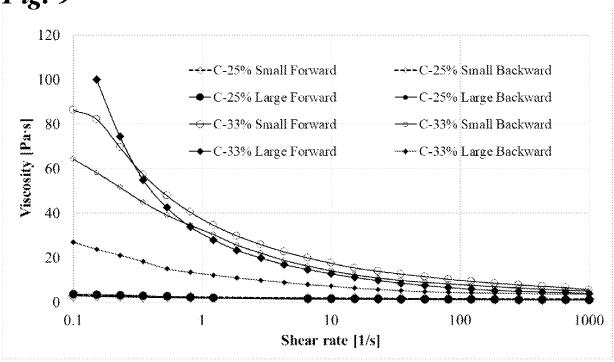
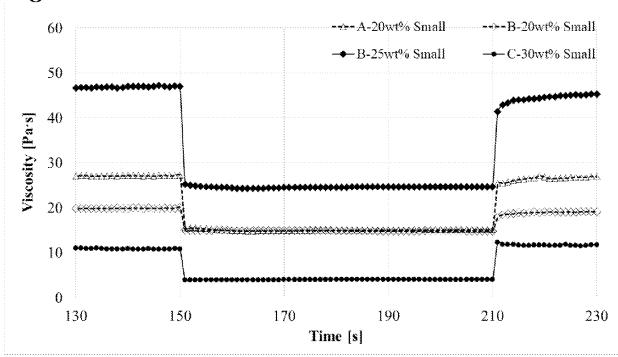


Fig. 10



# **SAMENWERKINGSVERDRAG (PCT)**

#### RAPPORT BETREFFENDE NIEUWHEIDSONDERZOEK VAN INTERNATIONAAL TYPE

IDENT	IFICATIE VAN DE N	NATIONALE AANVRAGE	KENMERK VAN DE AA	NVRAGER OF VAN DE GEMACHTIGDE	
Nederl	ands aanvraag nr.		Indieningsdatum		
	2027850			29-03-2021	
			Ingeroepen voorrangsda	atum	
Aanyra	ger (Naam)				
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	Coöperatie K	oninklijke Cosun U.A.			
Datum	van het verzoek voo	or een onderzoek van	Door de Instantie voor Ir	nternationaal Onderzoek aan	
interna	tionaal type		het verzoek voor een on	derzoek van internationaal type	
			toegekend nr.		
05-06-2021				SN78908	
I. CLA	SSIFICATIE VAN H	ET ONDERWERP (bij toepassir	I ng van verschillende classific	aties, alle classificatiesymbolen opgeven)	
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II. ONL	PERZOCHTE GEBIE	EDEN VAN DE TECHNIEK Onderzochte minimumdocume	entatie		
Classif	catiesysteem	Classificatiesymbolen	Sittatio		
	IPC	Zie onderzoeksrapport			
Onderzochte andere documentatie dan de minimum documentatie, voor zover dergelijke documenten in de onderzochte gebieden					
zijn opgenomen					
III. GEEN ONDERZOEK MOGELIJK VOOR BEPAALDE CONCLUSIES (opmerkingen op aanvullingsblad)				(opmerkingen op aanvullingsblad)	
IV.		NHEID VAN UITVINDING		(opmerkingen op aanvullingsblad)	

Form PCT/ISA 201 A (11/2000)

#### ONDERZOEKSRAPPORT BETREFFENDE HET RESULTAAT VAN HET ONDERZOEK NAAR DE STAND VAN DE TECHNIEK VAN HET INTERNATIONALE TYPE

Nummer van het verzoek om een onderzoek naar de stand van de techniek

NL 2027850

A. CLASSIFICATIE VAN HET ONDERWERP

INV. C08G18/64

ADD.

C08H8/00

C08L1/00

Volgens de Internationale Classificatie van octrooien (IPC) of zowel volgens de nationale classificatie als volgens de IPC.

C09K21/14

B. ONDERZOCHTE GEBIEDEN VAN DE TECHNIEK

Onderzochte miminum documentatie (classificatie gevolgd door classificatiesymbolen)

C08G C09J G02F C08H C08L C09K C09D

Onderzochte andere documentatie dan de mimimum documentatie, voor dergelijke documenten, voor zover dergelijke documenten in de onderzochte gebieden zijn opgenomen

Tijdens het onderzoek geraadpleegde elektronische gegevensbestanden (naam van de gegevensbestanden en, waar uitvoerbaar, gebruikte trefwoorden)

EPO-Internal, WPI Data

A US 4 481 076 A (HERRICK FRANKLIN W [US]) 6 november 1984 (1984-11-06) * sample 2; tabel 2 *  A,D EP 0 682 050 Al (ERIDANIA [IT]) 15 november 1995 (1995-11-15) in de aanvraag genoemd * tabel 2 *  A WO 2004/011518 A2 (UNIV MINNESOTA [US]; 1-17 RUAN R ROGER [US] ET AL.) 5 februari 2004 (2004-02-05) * tabel 14 *  A WO 2018/002445 A2 (BETULIUM OY [FI]) 4 januari 2018 (2018-01-04) * bladzijden 16-17; tabellen 1,2 *  -/   Verdere documenten worden vermeld in het vervolg van vak C.  * Speciale categorieën van aangehaalde documenten "A" niet tot de categorie X of Y behorende literatuur die de stand van de techniek beschrijt "D' ind e octroolaanvraage, gesubilozeerd op of na de indieningsdatum, waarin dezelfde uitwriffing wordt beschreven "C" niet-schriftelijke stand van de techniek "D" und echtoolaanvraage, gesubilozeerd op of na de indieningsdatum, waarin dezelfde uitwriffing wordt beschreven "C" niet-schriftelijke stand van de techniek "D" und echtoolaanvraage de voorrangsdatum en de indieningsdatum, waarin dezelfde uitwriffing wordt beschreven "D" und echtoolaanvraage vermeld "C" eedree octooriojaanvraage, gesubilozeerd op of na de indieningsdatum, waarin dezelfde uitwriffing wordt beschreven "C" inde-schriftelijke stand van de techniek "D" und echtoolaanvraage de voorrangsdatum en de indieningsdatum, waarin dezelfde categorie, waarbij de combinatie voor dezelfde categorie, waarbij de combinatie voor de vaard als niet mentief beschouwd ten opzichte van deze literatuur de de combinatie van dezelfde categorie, waarbij de combinatie voor de vaard als niet mentief beschouwd ten opzichte van dezelfde categorie, waarbij de combinatie voor de vaard als niet nietuw of niet inventief beschouwd ten opzichte van dezelfde categorie, waarbij de combinatie voor dezelfde categorie, waarbij de combinatie voor de vaard als niet nietuw of niet inventief beschouwd ten opzichte van dezelfde categorie, waarbij de combinatie voor de vaard als niet nietuw of niet inventief beschouwd ten opzichte van dezelfde categorie, waarbij de combi	C. VAN BEL	ANG GEACHTE DOCUMENTEN		
6 november 1984 (1984–11–06)  * sample 2; tabel 2 *  A,D EP 0 682 050 A1 (ERIDANIA [IT]) 15 november 1995 (1995–11–15) in de aanvraag genoemd  * tabel 2 *  A WO 2004/011518 A2 (UNIV MINNESOTA [US]; RUAN R ROGER [US] ET AL.) 5 februari 2004 (2004–02–05)  * tabel 14 *  A WO 2018/002445 A2 (BETULIUM OY [FI]) 4 januari 2018 (2018–01–04)  * bladzijden 16–17; tabellen 1,2 *  ——/—   Verdere documenten worden vermeld in het vervolg van vak C.  * Speciale categorieën van aangehaalde documenten  "A" niet tot de categorie X of Y behorende literatuur die de stand van de techniek beschrijf  "D' in de octrooiaanvrage, gepubliceerd op of na de indieningsdatum, waarn dezefde uitvinding wordt beschreven  "L" om andere redenen vermeldd literatuur  "T" om andere redenen vermeld wordt peschreven  "L" om andere redenen vermelde literatuur  "T" tussen de voorvangsdatum en de indieningsdatum, waarn de verdenen vermelde literatuur  "T" tussen de voorvangsdatum en de indieningsdatum gepubliceerde literatuur  "Ve de conclusie wordt als niet nieuw of niet inventiet beschouwd ten opzichte van de combinatie voor de valman voor de haind liggend wordt geacht literatuur andezelfde categorie, waarolj de combinatie voor de valman voor de haind liggend wordt geacht literatuur andezelfde categorie, waarolj de combinatie voor de valman voor de haind liggend wordt geacht literatuur literatuur  "Verzendatum van het rapport van het onderzoek naar de standere handere van dezelfde categorie, waarolj de combinatie voor de valman voor de haind liggend wordt geacht literatuur literatuur  "Verzendatum van het rapport van het onderzoek naar de standere hadres gevierdelde octroo	Categorie °	Geciteerde documenten, eventueel met aanduiding van speciaal	van belang zijnde passages	Van belang voor conclusie nr.
15 november 1995 (1995–11–15) in de aanvraag genoemd  * tabel 2 *   A	A	6 november 1984 (1984-11-06) * sample 2;	IW [US])	1–17
RUAN R ROGER [US] ET AL.) 5 februari 2004 (2004–02–05) * tabel 14 *  A WO 2018/002445 A2 (BETULIUM OY [FI]) 4 januari 2018 (2018–01–04) * bladzijden 16–17; tabellen 1,2 *  ———————————————————————————————————	A,D	15 november 1995 (1995-11-15) in de aanvraag genoemd		1–17
4 januari 2018 (2018–01–04)  * bladzijden 16–17; tabellen 1,2 *  ———————————————————————————————————	A	RUAN R ROGER [US] ET AL.) 5 februari 2004 (2004-02-05)	TA [US];	1–17
Speciale categorieën van aangehaalde documenten  "A" niet tot de categorie X of Y behorende literatuur die de stand van de techniek beschrijft  "D" in de octrooiaanvrage vermeld  "E" eerdere octrooi(aanvrage), gepubliceerd op of na de indieningsdatum, waarin dezelfde uitvinding wordt beschreven  "L" om andere redenen vermelde literatuur  "O" niet-schriftelijke stand van de techniek  "P" tussen de voorrangsdatum en de indieningsdatum gepubliceerde literatuur  Datum waarop het onderzoek naar de stand van de techniek van internationaal type werd voltooid  15 december 2021  Naam en adres van de instantie  European Patent Office, P.B. 5818 Patentlaan 2	A	4 januari 2018 (2018-01-04)	<b>:</b> *	1–17
"A" niet tot de categorie X of Y behorende literatuur die de stand van de techniek beschrijft "D" in de octrooiaanvrage vermeld "E" eerdere octrooi(aanvrage), gepubliceerd op of na de indieningsdatum, waarin dezelfde uitvinding wordt beschreven "L" om andere redenen vermelde literatuur "O" niet-schriftelijke stand van de techniek "P" tussen de voorrangsdatum en de indieningsdatum gepubliceerde literatuur "P" tussen de voorrangsdatum en de indieningsdatum gepubliceerde literatuur  Datum waarop het onderzoek naar de stand van de techniek van internationaal type werd voltooid  Naam en adres van de instantie European Patent Office, P.B. 5818 Patentlaan 2	X Verd	lere documenten worden vermeld in het vervolg van vak C.	Leden van dezelfde octrooifamilie	zijn vermeld in een bijlage
"L" om andere redenen vermelde literatuur  "O" niet-schriftelijke stand van de techniek  "P" tussen de voorrangsdatum en de indieningsdatum gepubliceerde literatuur  "Au de combinatie van dezelfde categorie, waarbij de combinatie voor de vakman voor de hand liggend wordt geacht  "B" tussen de voorrangsdatum en de indieningsdatum gepubliceerde literatuur  "Au lid van dezelfde octrooifamilie of overeenkomstige octrooipubliceerde literatuur  "Au lid van dezelfde octrooifamilie of overeenkomstige octrooipubliceerde literatuur  "Au lid van dezelfde octrooifamilie of overeenkomstige octrooipubliceerde literatuur  "Au lid van dezelfde octrooifamilie of overeenkomstige octrooipubliceerde literatuur  Verzenddatum van het rapport van het onderzoek naar de stand et echniek van internationaal type  15 december 2021  Naam en adres van de instantie  European Patent Office, P.B. 5818 Patentlaan 2	"A" niet tot technie "D" in de oo	de categorie X of Y behorende literatuur die de stand van de ek beschrijft ctrooiaanvrage vermeld	literatuur die niet bezwarend is voor o maar wordt vermeld ter verheldering het principe dat ten grondslag ligt aan "X" de conclusie wordt als niet nieuw of nie	le octrooiaanvrage, van de theorie of n de uitvinding
internationaal type werd voltooid de techniek van internationaal type  15 december 2021  Naam en adres van de instantie European Patent Office, P.B. 5818 Patentlaan 2  De bevoegde ambtenaar	"L" om andere redenen vermelde literatuur "O" niet-schriftelijke stand van de techniek		van de combinatie van deze literatuur met andere gec literatuur van dezelfde categorie, waarbij de combinati de vakman voor de hand liggend wordt geacht	
Naam en adres van de instantie  European Patent Office, P.B. 5818 Patentlaan 2  De bevoegde ambtenaar				t onderzoek naar de stand va
European Patent Office, P.B. 5818 Patentlaan 2	1	5 december 2021		
NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Fay: (-31-70) 340-3016  Pötzsch, Robert	Naam en ac	European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk		

Fax: (+31-70) 340-3016

Pötzsch, Robert

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Nummer van het verzoek om een onderzoek naar de stand van de techniek

NL 2027850

	VAN BELANG GEACHTE DOCUMENTEN	Van helene weer
Categorie °	Geciteerde documenten, eventueel met aanduiding van speciaal van belang zijnde passages	Van belang voor conclusie nr.
A	PAVIER C ET AL: "Urethanes and polyurethanes from oxypropylated sugar beet pulp - I. Kinetic study in solution", EUROPEAN POLYMER JOURNAL, PERGAMON PRESS LTD OXFORD, GB, deel 36, nr. 8, 1 augustus 2000 (2000-08-01), bladzijden 1653-1658, XP004195763, ISSN: 0014-3057, DOI: 10.1016/S0014-3057(99)00245-1 * samenvatting *	1–17
A	EP 3 447 113 A1 (PROCTER & GAMBLE [US]) 27 februari 2019 (2019-02-27) * tabel 1 *	1-17

#### ONDERZOEKSRAPPORT BETREFFENDE HET RESULTAAT VAN HET ONDERZOEK NAAR DE STAND VAN DE TECHNIEK VAN HET INTERNATIONALE TYPE

Informatie over leden van dezelfde octrooifamilie

Nummer van het verzoek om een onderzoek naar de stand van de techniek

NL 2027850

In het genoemd o	rapport ctrooigeschrift		Datum van oublicatie	O۱	rereenkomend(e) geschrift(en)		Datum van publicatie
US	4481076	A	06-11-19	84 AT	64420	т	15-06-199
				CA	1208631	A	29-07-198
				EP	0120471	<b>A2</b>	03-10-198
				FI	841220	A	29-09-198
				IN	160347	В	04-07-198
				JP	S591891 <b>4</b> 1	A	26-10-198
				MX	161160	A	09-08-199
				NO	165932	В	21-01-199
				US	4481076	A	06-11-198
EP	0682050	A1	15-11-19	95 GE:	 En		
WO	2004011518	A2	05-02-20	04 AU	2003261191	A1	16-02-200
				WO	2004011518	A2	05-02-200
WO	2018002445	A2	04-01-20	 18 EP	3478724	A2	08-05-201
				WO	2018002445		04-01-201
EP	3447113	A1	27-02-20				15-01-201
				CN	105339481	A	17-02-201
				EP	2824170	A1	14-01-201
				EP	3447113	A1	27-02-201
				JP	6250804	в2	20-12-201
				JP	2016525591	A	25-08-201
				US	2015159120	A1	11-06-201
				US	2017191003	A1	06-07-201
				WO	2015006634	<b>A</b> 1	15-01-201

# WRITTEN OPINION

File No. SN78908	Filing date (day/month/year) 29.03.2021	Priority date (day/month/year)	Application No. NL2027850				
International Patent Classification (IPC) INV. C08G18/64 C08H8/00 C08L1/00 C09K21/14							
Applicant Coöperatie Koninklijl	Applicant Coöperatie Koninklijke Cosun U.A.						
,							
This opinion co	ntains indications relating to the	following items:					
⊠ Box No. I	Basis of the opinion						
☐ Box No. II	Priority						
☐ Box No. III	Non-establishment of opinion with	regard to novelty, inventive step a	nd industrial applicability				
☐ Box No. IV	Lack of unity of invention						
⊠ Box No. V	Reasoned statement with regard to applicability; citations and explanat	novelty, inventive step or industri ions supporting such statement	al				
☐ Box No. VI	Certain documents cited						
☐ Box No. VII	Certain defects in the application						
☐ Box No. VIII	☐ Box No. VIII Certain observations on the application						
		Examiner					
		Pötzsch, Robert					

# **WRITTEN OPINION**

	Box No. I Basis of this opinion							
1.	1. This opinion has been established on the basis of the latest set of claims filed before the s	start of the search.						
2.	With regard to any <b>nucleotide and/or amino acid sequence</b> disclosed in the application and necessary to the claimed invention, this opinion has been established on the basis of:							
8	a. type of material:							
	☐ a sequence listing							
	☐ table(s) related to the sequence listing							
	b. format of material:							
	☐ on paper							
	☐ in electronic form							
	c. time of filing/furnishing:							
	☐ contained in the application as filed.							
	☐ filed together with the application in electronic form.							
	$\square$ furnished subsequently for the purposes of search.							
3.	3.  In addition, in the case that more than one version or copy of a sequence listing and/or has been filed or furnished, the required statements that the information in the subsection copies is identical to that in the application as filed or does not go beyond the application appropriate, were furnished.	quent or additional						
4.	4. Additional comments:							
	Box No. V Reasoned statement with regard to novelty, inventive step or industria citations and explanations supporting such statement	l applicability;						
1.	1. Statement							
	Novelty Yes: Claims 1-17 No: Claims							
	Inventive step Yes: Claims 1-17 No: Claims							
I	Industrial applicability  Yes: Claims  1-17  No: Claims							

2. Citations and explanations

see separate sheet

#### Re Item V

# Reasoned statement with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

- 1 Reference is made to the following document:
  - D1 US 4 481 076 A (HERRICK FRANKLIN W [US]) 6 november 1984 (1984-11-06)
- D1 is regarded as being the prior art closest to the subject-matter of claim 1, and discloses (table 2 the sample 2) a dispersion prior to addition of water comprising:
  - 28.5 wt.% microfibrillated cellulose (MFC) (i.e. a plant based particle) and
  - 71.5 wt.% glycerin (i.e. a polyol)

Example 1 states that MFC pulp fibers are cut to a length of 0.7 mm. The size of MFC should therefore be expected to be above a D90 of 300 microns.

D1 states that the MFC is prepared from southern pine which is known to have a lignin content of about 30 wt.%.

The subject-matter of claim 1 therefore differs from this known dispersion at least in that:

- the lignin content should be less than 6 wt.% and is therefore new.
- the size dictribution in terms of D90 of teh solid particles is less than 300 microns.

No technical effect has been shown for this difference. The problem to be solved by the present invention may be regarded as to provide an alternative dispersion.

The solution to this problem proposed in claim 1 of the present application is considered as involving an inventive step for the following reasons: There is no hint in D1 nor in any other prior art to be found that would have motivated the skilled person to modify the dispersion in this manner, i.e. lower the lignin content and reducing the size of the solid particles.

Claim 1 is therefore inventive.

3 Claims 14-16 refer to a method of preparing the dispersion and to uses thereof.

These claims are therefore novel and inventive as well.

4 Claims 2-13 and 17 are dependent on claims 1 or 15-16, respectively, and as such also meet the requirements of novelty and inventive step.