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(54) **A METHOD FOR MANUFACTURING PULP PRECURSOR MATERIAL FOR COMPOUNDING APPLICATIONS AND PRODUCTS THEREOF**

VERFAHREN ZUR HERSTELLUNG VON PULPEVORLÄUFERMATERIAL FÜR
MISCHUNGSANWENDUNGEN UND PRODUKTE DARAUS

PROCÉDÉ DE FABRICATION D'UN MATÉRIAU PRÉCURSEUR DE PÂTE À PAPIER POUR
MÉLANGER DES APPLICATIONS ET PRODUITS ASSOCIÉS

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Description

[0001] A method for manufacturing pulp precursor material from chemical pulp for compounding applications and products thereof

Field of the Invention

[0002] The invention relates to a method and a system for manufacturing pulp precursor material having high chemical pulp content and product thereof, which pulp precursor material is suitable for further use in polymer compounding applications. The invention further relates to the use of adsorption agents that are suitable for surface interactions with the cellulose fibres in such manufacturing method.

Background

[0003] Wood-based materials have in many applications emerged as a sustainable and renewable source to replace the previously used non-renewable fibre types in fibre-based polymer composites. Cellulose fibre-based polymer composites are manufactured by compounding together polymer and cellulose fibre-based material, in order to achieve a homogenous blend of the two different raw materials, which two different raw materials remain separate and distinct within the finished structure. The polymer binds the composite materials together, while the cellulose fibre-based material typically reinforces the composite. In composite compounding, the used polymer type and amount, as well as the cellulose fibre-based material, are selected based on the required properties of the manufactured composite product. Compounding involves that the polymer obtains a molten state, whereas the cellulose fibre-based material remains solid. As the components are dosed automatically via feeders or hoppers, the mixing, as well as temperature control, are important factors in the composite compounding.

[0004] In the past, compounding has been in general been made by simply adding the components together in a mixer/compounder, whereby the material are mixed and processed into a composite, typically via direct extrusion or moulding. Commonly used polymer matrix materials in cellulose fibre-reinforced polymer composites have included thermoplastic polyolefins, such as polyethylene and polypropylene. Wood flour and sawdust, denoting wood that has simply been ground into smaller particles, have for a long time been the most widely available cellulose fibre-based materials. The tensile and tear strength of a polymer composite comprising wood flour or sawdust is, however, relatively weak and many aromatic groups contained in the wood can be highly odorous. Thus such cellulose fibre-based materials are no longer a preferred source material for high quality composites.

[0005] More recently, a variety of wood pulp-based constituents, mechanical and chemical pulp in particular, have emerged as potential sources of fibre-based materials, which may be used together with thermoplastic polymers in composite products. In comparison to ground wood particles, wood pulp in general is much more processed form of cellulosic material that is obtained from the secondary xylem (i.e., heartwood and sapwood) of trees, whereby, depending of the pulping method, the obtained cellulose fibres contain varying amounts of the three main components of wood: cellulose, hemicelluloses, and lignin.

[0006] The properties of mechanical pulp, however, are still inferior compared to chemical pulp. Mechanical pulping is not designed to remove lignin from the wood, whereby lignin remains to a large extent in mechanical pulp. Lignin can begin to participate in condensation reactions already at a temperature as low as 90°C. The condensation reactions are accelerated considerably when the temperature reaches 130°C, whereas many polymers require much higher compounding temperatures than this. Thus, compounding easily leads to heat-induced lignin condensation reactions, which may darken the formed product and further produce water that remains entrapped into the composite melt. Upon elevated pressure and temperature the entrapped moisture may evaporate and expand into gaseous phase, which is why the forming moisture would need to be removed. This may be problematic, as upon compounding, the melting polymer encapsulates the pulp-based constituents. The use of more processed cellulosic material, wherein lignin has been removed to a large extent, with polymers, however, poses a compatibility problem in the compounding stage. Chemical pulping in particular, provides typically pulp containing cellulose fibres that have a different chemical nature with respect to many of the common thermoplastics that are used for compounding. Chemical pulping, such as Kraft process, decreases the amounts of hemicelluloses and lignin in the fibres, thereby rendering the hydroxyl groups of the cellulose fibres more accessible on the fibre surface. Such highly processed cellulose fibres are thus very hydrophilic and have a strong affinity towards water. Bleached cellulose fibres even become slightly anionic in aqueous solution, due to the ionization of functional carboxyl groups in the oxidized cellulose. The chemical nature of processed cellulose fibres thus causes a compatibility problem, when such material is sought to combine with a thermoplastic polymer, such as polyethylene or polypropylene, which are extremely hydrophobic. Due to the absence of attraction towards such hydrophobic materials, the processed cellulose fibres do not easily become evenly distributed throughout the polymer matrix. The immiscibility of the two components may therefore lead to fibre agglomerate formation within the polymer matrix. The

lack of affinity towards each other may further hinder the interaction and adhesion between the processed cellulose fibres and polymer matrix upon compounding, which can be reflected in reduced reinforcing effect of the fibres in a formed polymer composite. As a result, the polymer matrix in the formed composite material may not transfer focused loads effectively onto the fibres.

[0007] One way to address the immiscibility of the two components has been to use a compounding polymer that contains a traditional coupling agent, such as maleic anhydride grafted polypropylene, wherein the grafted group can be used e.g. to reduce the surface tension and interfacial energy between bleached cellulose fibres and polypropylene and thereby promote covalent or hydrogen bonding of the two components. However, even with traditional coupling agents, obtaining a composite having a homogeneous fibre dispersion may be challenging. In addition to choosing an effective coupling agent, particular care has to be given to the compounding temperature and shear forces involved in the mixing. Poor dispersion of the cellulose fibres may further cause premature solidification of the mixture and formation of isolating layers, which can lead to excessive accumulation of heat inside the machinery. Cellulose fibres can start to degrade when the temperature rises above 220°C (see Analytical pyrolysis of natural organic polymer by S.C.Moldoveanu, Vol 20, Elsevier, 1998). To avoid charring, the majority of compounding processes are performed in a temperature less than 200°C, such as in the range between 160 and 190°C, as long as the temperature is high enough for the matrix polymer to melt. Extensive shearing forces, on the other hand, may induce fibre failures, such as fibrillation, de-lamination, fracturing and tearing, which decrease the strength of the composite.

Summary of the Invention

[0008] The invention addresses the problems disclosed above. The compounding of polymer and chemical pulp containing cellulose fibres may be improved by modifying the characteristics of the chemical pulp before compounding. In particular, by a combination of fibre pre-processing and treatment of the cellulose fibre surfaces with an adsorption agent, the hydrogen bonding of the cellulose fibres towards each other may be reduced and the compatibility of the cellulose fibre surface towards a subsequently added compounding polymer may be improved. In addition, the adsorption agent may be arranged to provide functionality to the cellulose fibre surfaces. A subsequent addition of a minute amount of thermoplastic compatibilizer can be used to improve the miscibility of the cellulose fibres with hydrophobic polymers, such that pulp precursor material having improved dispersion and distribution characteristics may be obtained. This leads to pulp precursor material which can be provided at a high chemical pulp content and improved compounding properties, such as dispersion and mechanical strength, in addition with improved moisture repellence. The improved characteristics of the pulp precursor material are thereby available for the compounder, who can benefit of the enhanced compounding properties of the pulp precursor material upon compounding. The pulp precursor material may thus be configured to act as a 'master batch', which in itself promotes easy dispersion of the chemical pulp upon compounding. This is advantageous, as the composite manufacturer can reach a desired chemical pulp content level with better homogeneity and less compounding, which is reflected in better thermomechanical properties of the composite. Furthermore, the composite manufacturer has better capability to choose the chemical pulp content level of the formed polymer composite, which has been difficult in the past, in particular due to the agglomeration tendency of bleached chemical pulp within the polymer matrix.

[0009] In the context of this disclosure, chemical pulp refers to material originating from wooden material, which has undergone a chemical pulping process and which thereby has been processed into fibrous form, the chemical pulp thereby containing cellulose fibres. The expression 'pulp content' is used to specify the weight percentage of material derived from the chemical pulping process, with respect to the total weight of the material, unless otherwise specified.

[0010] Chemical pulping disintegrates the structure of the wood with strong chemicals in a cooking process, thereby producing fibrous material with a very high cellulose fibre content. The purpose of chemical pulping is to degrade and dissolve the lignin in wood so that the cellulose fibres can be separated without mechanical treatment. Compared to mechanical pulping, the chemical cooking also preserves better the original cellulose fibre length. However, this delignification process also degrades considerable amounts of hemicelluloses and in lesser amounts cellulose fibres. For this reason, the delignification process is stopped before significant losses of cellulose and hemicelluloses, while leaving less than 10 % of the original lignin in the pulp. The delignification process used to break down lignin is referred to as a chemical cooking. A chemical pulping process thus removes nearly all of the lignin and at least part of the hemicelluloses, while preserving the fibre structure and length better than semi-chemical or mechanical methods. A chemical pulping process therefore provides more processed cellulose fibres that has excellent physical properties, such as stiffness and rigidity, which is not obtained with mechanical or semi-mechanical pulping processes. A chemical pulping process further provides cellulose fibres containing pores. Examples of chemical pulping processes are, for example, the sulphite pulping process or the Kraft pulping process. The Kraft pulping process uses sodium sulphide and alkali to separate cellulose fibres from other compounds in the wood material. Non-limiting examples of chemical pulp are Kraft pulp, sulphite pulp and dissolving pulp.

[0011] The remaining lignin in the chemical pulp can be further removed through bleaching processes, thereby providing

bleached chemical pulp. Bleached chemical pulp typically contains lignin in an amount of less than 1 wt.% of the bleached chemical pulp. Bleaching is typically performed in multi-stage sequence of steps, which utilize different bleaching chemicals. Typical bleaching chemical include chlorine dioxide (ClO_2), hypochlorite (NaClO), oxygen (O_2), hydrogen peroxide (H_2O_2), and ozone (O_3). All of these bleaching chemicals are oxidative, and therefore the bleaching reactions can be considered as oxidation reactions. The first bleaching steps are further delignification stages, whereas the later steps are brightening stages, in which the brown-colour inducing chromophores are removed, thereby increasing the pulp whiteness and brightness. Brightness may be advantageous in fibre-based polymer composite objects wherein lighter colours or paintability is preferred.

[0012] Bleached chemical pulp presents superior properties when compared to conventional wooden material in fibre-based polymer composites. The Kraft process, in particular, decreases considerably the amounts of hemicelluloses, lignin, wood extractives and inorganics in the pulp material such that only residual traces of these compounds remain; thereby the bleached chemical pulp may be denoted as essentially 'lignin free'. This has three main effects on the properties of the bleached chemical pulp containing cellulose fibres. First, the bleached chemical pulp is stiff and strong, because the flexible lignin and hemicellulose components are mostly removed. Thus, the highly ordered, rigid cellulose fibres may be used to provide a reinforcing effect on a fibre-based polymer composite. Second, cellulose fibrils and hydroxyl groups become more accessible on the surface of the cellulose fibres. This enables surface interactions of the cellulose fibres with adsorption agents. Third, the removal of lignin and hemicelluloses from the fibres creates pores into the cellulose fibre structure. The pores may improve the effect of the adsorption agents with the cellulose fibres. The removal of lignin also removes most of the aromatic groups from the pulp, which thereby results to a raw material that is less odorous. The chemical pulping method may thus be used to provide a range of highly processed fibrous raw material, which may be further tailored to contain specific properties, which may be transferred to the fibre-based polymer composite.

[0013] Adsorption agents have been observed to address the immiscibility of the cellulose fibres with compounding polymers by reducing hydrogen bonding of the cellulose fibres towards each other. Furthermore, the adsorption agent may be arranged to contain functional groups, such as amine groups of vinyl groups or cationic sites. Amine group ($-\text{NH}_2$), for example, is reactive with maleic anhydride. Maleic anhydride may be present in a thermoplastic compatibilizer that is added to the dried bleached chemical pulp containing cellulose fibres, when manufacturing pulp precursor material. Advantageously, the adding of the adsorption agent is performed in water suspension when refining the pulp, or either before or after dewatering the refined pulp. The adding of the adsorption agent may be performed in a separate reactor unit. The adding of the adsorption agent may also be performed in a fluffing unit, when decreasing the bulk density of dewatered pulp, such that the adsorption agent interacts with the cellulose fibre surfaces, thereby providing cellulose fibres that contain adsorption agent on the fibre surface. Experimental data suggests that silanes may be particularly suitable adsorption agents for surface interactions with the highly processed cellulose fibres present in bleached chemical pulp. Vinyl silane (vinyltriethoxysilane) and aminosilane (aminopropyltriethoxysilane) in particular have showed nearly linear adsorption behaviour in dosage amounts of up to 3 wt.% per dry bleached chemical pulp. Experimental data further suggests that refining of the bleached chemical pulp may be used to increase the adsorbed fraction of the adsorption agent, in particular when silanes are used. A moderate refining of up to 100 kWh/t has been observed to improve the impact strength of a fibre-based polymer composite containing bleached chemical pulp with non-treated cellulose fibres. When adding the adsorption agent in small amounts of less than 0.3 wt.% of the bleached chemical pulp, a positive effect was observed on the tensile and impact strength of a fibre-based polymer composite.

[0014] In addition to silanes, also other adsorption agents, such as polyethyleneimines, polyallyl amines or cationic polyelectrolytes may be suitable. In particular, the slightly anionic charge of the oxidized cellulose fibre surface due to the ionization of functional groups, mainly carboxyl groups, may be used to amplify the adsorption of cationic surfactants and polyelectrolytes onto the surface of the cellulose fibre through electrostatic interactions.

[0015] The adsorption agent may thus be provided on the surface of the cellulose fibres of refined pulp, either before or after dewatering the refined pulp. The adsorption agent may thus also be provided on the surface of the cellulose fibres after dewatering the refined pulp, in a fluffing unit, in which the fibre material of the bleached chemical pulp is separated in a manner so as to increase the available specific surface of the cellulose fibre material so that the accessibility for the educts, whether an adsorption agent or a thermoplastic compatibilizer, to the cellulose fibre material surface is optimized. Prior to mixing with the thermoplastic compatibilizer, the dewatered pulp is advantageously dried in a flash drying unit, such that pulp precursor material which has a chemical pulp content of equal to or higher than 80 wt.% is obtained.

[0016] A small amount of thermoplastic compatibilizer, which can be a polymer, may be mixed with the dried bleached chemical pulp containing cellulose fibres, without concomitant melting of the thermoplastic compatibilizer upon mixing. The addition of the thermoplastic compatibilizer in an amount of less than 12 wt.% of the weight of the pulp precursor material has been observed to improve the dispersion of the bleached chemical pulp upon later compounding with polymers that are commonly used in fibre-based polymer composites, such as polyolefins, in particular in combination with bleached chemical pulp that has been treated with an adsorption agent as described above.

[0017] The cellulose fibres can therefore be provided with predictable quality and characteristics, which improve the affinity of the fibres towards the polymer upon compounding and promote the interaction between the cellulose fibres and the polymer upon compounding, such that an improved reinforcing effect of the fibres may be obtained to a formed polymer composite. Thus, bleached chemical pulp may be used to provide pulp precursor material, which has a pulp content of equal to or higher than 80 wt.%, for compounding with a further polymer, wherein the bleached chemical pulp may be arranged to contain adsorption agent on the surface of the cellulose fibres. The processing of the bleached chemical pulp may be arranged to provide a more reactive surface towards adsorption agents. Such bleached chemical pulp, in combination with a suitable adsorption agent and a thermoplastic compatibilizer, has the advantage that a pulp precursor material may be obtained, wherein the pulp precursor material has improved dispersion characteristics in further compounding with a polymer, and a composite product with improved mechanical properties may be obtained.

[0018] According to an aspect of the invention, there is provided a pulp precursor material for compounding with a polymer, the pulp precursor material comprising

- dried bleached chemical pulp containing cellulose fibres,
- adsorption agent on the surface of the cellulose fibres, , said adsorption agent being for reducing hydrogen bonding of the cellulose fibres towards each other, and
- less than 12 wt.% of thermoplastic compatibilizer which has been mixed with the dried bleached chemical pulp, said thermoplastic compatibilizer being for improving miscibility of the cellulose fibers with hydrophobic polymers;

wherein the pulp content of the pulp precursor material is equal to or higher than 80 wt.% of the pulp precursor material and the moisture content of the precursor material is less than 10 wt.% of the pulp precursor material.

[0019] According to another aspect of the invention, there is provided a method for manufacturing pulp precursor material for compounding with a polymer, the method comprising:

- adding adsorption agent that is suitable for surface interactions with cellulose fibres to bleached chemical pulp, said adsorption agent being for reducing hydrogen bonding of the cellulose fibres towards each other,
- dewatering the pulp by pressing or filtering such that a pulp content in the range of 40 to 50 wt.% is obtained,
- evaporating moisture from the pulp in a flash drying unit, thereby obtaining dried pulp having a moisture content of less than 10 wt.%, and
- mixing less than 12 wt.% of thermoplastic compatibilizer into the dried pulp, thereby obtaining pulp precursor material which has a pulp content of equal to or higher than 80 wt.% and which is suitable for compounding with a polymer, said thermoplastic compatibilizer being for improving miscibility of the cellulose fibers with hydrophobic polymers.

[0020] Advantageously, the thermoplastic compatibilizer and the dried bleached chemical pulp have been mixed with each other as a dry-blend.

[0021] Further, the method may comprise

- compacting the dried pulp, such that the bulk density of the dried pulp is higher than 100 kg/m³, preferably in the range of 150 to 300 kg/m³, prior to mixing the thermoplastic compatibilizer into the dried pulp and, when necessary,
- compressing the pulp precursor material into granulates or pellets in a compacting unit, wherein the compacting unit is configured to provide granulates or pellets having a bulk density of higher than 300 kg/m³ and a moisture content of less than 0.5 wt.%.

[0022] According to a further aspect of the invention, there is provided a system for manufacturing pulp precursor material which has a pulp content of equal to or higher than 80 wt.% for compounding with a polymer, wherein the system may comprise:

- an inlet configured to receive bleached chemical pulp containing cellulose fibres from a pulping process,
- a refining unit configured to increase the surface area of the cellulose fibres in the bleached chemical pulp,
- adding means for adding adsorption agent to the surface of cellulose fibres,
- a dewatering unit configured to decrease the water content of the pulp such that pulp having a pulp content in the range of 40 to 50 wt.% is obtainable,
- a fluffing unit configured to decrease the bulk density of the pulp, such that fluffed pulp having a moisture content of less than 50 wt.% is obtainable,
- a flash drying unit configured to evaporate moisture from the pulp such that dried pulp having a pulp content of equal to or higher than 80 wt.% and a moisture content of less than 10 wt.% is obtainable, and
- a mixing unit configured to admix thermoplastic compatibilizer into the dried pulp, such that pulp precursor material which has a pulp content of equal to or higher than 80 wt.% and is suitable for compounding with a polymer is

obtainable, and

- a compacting unit configured to provide granulates or pellets having a bulk density of higher than 300 kg/m³, such as a bulk density between 300 and 600 kg/m³ and a moisture content of less than 0.5 wt.%.

[0023] The invention is further described in the independent and dependent claims.

Description of the Drawings

[0024]

Figures 1a and b show, by way of an examples, variations of a method for manufacturing pulp precursor material from bleached chemical pulp containing cellulose fibres,

Figure 2 shows examples of chemical compounds that may act as adsorption agents with bleached chemical pulp containing cellulose fibres,

Figure 3 shows scanning electron microscope images of bleached primary chemical pulp fibres treated with 0.2 wt.% addition of aminosilane,

Figure 4 shows scanning electron microscope images of bleached primary chemical pulp fibres treated with 0.8 wt.% addition of aminosilane,

Figure 5 shows scanning electron microscope images of bleached primary chemical pulp fibres treated with 0.2 wt.% addition of vinylsilane,

Figure 6 shows scanning electron microscope images of bleached primary chemical pulp fibres treated with 0.8 wt.% addition of vinylsilane,

Figure 7 shows isotherms (1 h) of vinyl silane and aminosilane adsorption to bleached primary chemical pulp fibres (g/100g), measured as a function of the chemical dosage per dry pulp (wt.%),

Figure 8 shows isotherms (1h) of the adsorbed fraction (%) of vinyl silane and aminosilane to bleached primary chemical pulp fibres, measured as a function of the chemical dosage per dry pulp (wt.%),

Figure 9 is a diagram of silane adsorption rate with 2 wt.% dosage of vinyl silane or aminosilane, respectively, to bleached primary chemical pulp fibres, measured as a function of the reaction time (minutes),

Figure 10 is a diagram of silane adsorption amount with 2 wt.% dosage of vinyl silane or aminosilane, respectively, to bleached primary chemical pulp fibres, measured as a function of pulp refining (kWh/t),

Figure 11 shows flash dried pulp having a pulp content of equal to or higher than 80 wt.% treated with positively charged adsorption agent,

Figure 12 shows pulp precursor material pellets that have been compressed in a pelletizer to a bulk density of 600 kg/m³ and a moisture content of less than 0.5 wt.%,

Figure 13 is experimental data demonstrating the mechanical properties of fibre-based polymer composite samples manufactured of polypropylene and pulp precursor material that contains bleached primary chemical pulp fibres treated with 0.2 wt% addition of a cationic surfactant, and

Figure 14 is experimental data demonstrating the effect of fibre refining of to the mechanical properties of fibre-based polymer composite samples manufactured of polypropylene and pulp precursor material that contains bleached primary chemical pulp fibres that have been refined and then treated with 0.8 wt% addition of cationic surfactants.

Detailed Description of the Invention

A method for manufacturing pulp precursor material having high pulp content from bleached chemical pulp

[0025] Reference is made to Figures 1a and 1b, which illustrate, by way of examples, variations of a method for manufacturing pulp precursor material FIB1 from bleached chemical pulp PLP1 containing cellulose fibres and the subsequent compounding of the pulp precursor material PRE1 with a polymer POL2.

[0026] The following symbols will be used below, with reference to Figures 1a and 1b:

A = washing unit

B = refining unit

C = reactor unit

D = dewatering unit

E = fluffing unit

F = flash drying unit

G = mixing unit

H = compacting unit

I = compounding unit

IN1 = inlet pulp suspension to washing unit A

OUT1 = outlet pulp suspension from washing unit A

PLP1 = pulp suspension from the last bleaching stage of the chemical pulping

RF1 = refined pulp suspension

RC1 = pulp suspension from reactor

DEW1 = dewatered pulp

FLF1 = fluffed pulp

DRY1 = flash-dried pulp

FIB1 = pulp precursor material

GR1 = granulate

CMP1 = fibre-based polymer composite

CHEM1 = adsorption agent, such as APTES, VTES, PEI, CAT1 or CAT2

POL1 = thermoplastic compatibilizer

POL2 = compounding polymer

RES1 = filtrate from dewatering unit D

RES2 = moisture from fluffing unit E

RES3 = evaporated moisture from flash drying unit F

[0027] Figure 1a is a simplified version of a method for manufacturing pulp precursor material FIB1 from bleached chemical pulp PLP1 containing cellulose fibres. The unit operations with dashed lines in Figure 1b represent alternative steps in the method, which can be performed instead of the simplified version that has been illustrated in both Figures 1a and 1b. The alternative unit operations with dashed lines can be performed independently or in combination with other alternative steps, when necessary, for example to improve the fibre quality, defibrillation and available fibre surface area (step B), to improve the effect of the adsorption agent chemicals during the cellulose fibre surface treatment (steps C and E), to separate cellulose fibres from each other, thereby decreasing the bulk density (step E) or to compress and reduce the moisture content of the pulp precursor material (step H). The compounding (step I) of the obtained pulp precursor material with a further polymer is typically performed afterwards separately by the compounder. In particular, the addition of the adsorption agent CHEM1, while added only in small amounts, may be performed in one or multiple steps according to process requirements, in conjunction with unit operations B, C, D and/or E. The unit operations B, C, E, H, and I can therefore be considered optional and each performed independently, when necessary. For example, the refined pulp suspension RF1 from the refining unit B may be directed to the dewatering unit D, without passing through a reactor unit C. Further, if the pulp suspension PLP1 is refined at the refining unit B or if the adsorption agent CHEM1 is added into a reactor unit C, the pulp suspension PLP1 from the washing unit A does not need to pass directly to the dewatering unit D, but may be directed either to the refining unit B or to the reactor unit C. Likewise, when a fluffing unit E is used to separate cellulose fibres from each other, the dewatered pulp DEW1 may be directed to the fluffing unit E and does not need to pass directly to the flash drying unit F.

Bleached chemical pulp

[0028] Chemical pulp comprises fractions, which can be classified into primary chemical pulp and reject fractions.

Primary chemical pulp defines the fraction of the chemical pulp which is considered to be of sufficient quality for further use, for example in paper manufacturing. Reject defines the fraction of the chemical pulp which does not reach a predefined quality standard and is therefore considered to be of insufficient quality for further use in paper manufacturing. Reject material that is separated from the primary material is either discarded or, when possible, recycled back into the pulping process. Primary chemical pulp, which is bleached, is referred to as bleached primary chemical pulp.

[0029] When manufacturing high quality material for fibre-polymer composite applications, the starting material may be obtained from a chemical pulping process as a suspension PLP1 of never-dried bleached chemical pulp that typically contains lignin in an amount of less than 3%, more preferably less than 1 wt.%, most preferably less than 0.5 wt.% of the bleached chemical pulp. The amount of carbohydrates present in the bleached chemical pulp may be determined by sulfuric acid hydrolysis according to Tappi standard T250 cm-85 (see "Analysis of the organic material dissolved during totally chlorine-free bleaching" Doctoral thesis, University of Jyväskylä, 1999, p, 19-20).

[0030] Bleaching is typically performed in multi-stage sequence of steps, wherein the first bleaching steps are further delignification stages, whereas the later steps are brightening stages. At the last bleaching stage of the chemical pulping process, the bleached chemical pulp is typically washed in a washing unit A. The washing unit A may be, for example, a low consistency drum displacer washer in a fibreline, which can accommodate multiple washing stages in a single unit. The inlet pulp IN1 which enters the forming zone of the washing unit A typically has a consistency in the range of 4 to 6 wt.%. The outlet pulp OUT1 which exits the forming zone of the washing unit A typically has at a consistency in the range of 10 to 12 wt.%. When the outlet pulp OUT1 has passed the final wash zone of the washing unit A, the pulp consistency may be raised higher, up to 15 wt.%, with a vacuum pump. When manufacturing pulp precursor material FIB1 of bleached chemical pulp containing cellulose fibres, preferably of bleached primary chemical pulp, the pulp suspension PLP1 may therefore be obtained as a suspension PLP1 after washing and acidification, prior to the last high consistency bleaching tower of the pulping process, such that the pulp content of the suspension PLP1 is in the range of 3 to 15 wt.%, preferably in the range of 4 to 6 wt.% or in the range of 10 to 13 wt.%, most preferably in the range of 10 to 12 wt.%. The selection of the pulp content of the suspension PLP1 may be performed based on the pulp type and the type of the washing unit A.

[0031] Experimental results have demonstrated that the properties of bleached chemical pulp made of hardwood pulp differs from bleached chemical pulp made of softwood pulp. Table 1, below, represents exemplary results obtained from chemical pulp mill fibreline, comparing the distribution of cellulose fibre length in millimetres (mm) in hardwood (birch) and softwood (pine). The fibre length distribution is dependent of the pulping process and wood material used. Thus, variations in the fibre length distributions are possible. Of notice is, however, that at the washing unit, prior to the drying machine, bleached chemical pulp made of softwood has a large fraction of cellulose fibre lengths in the range of 2.0 to 3.2 mm, whereas hardwood had the majority of the cellulose fibre lengths typically in the range of 0.6 to 1.2 mm. The average cellulose fibre length of bleached chemical pulp made of softwood is much larger than the average cellulose fibre length of bleached chemical pulp made of hardwood.

Table 1. An example of fibre furnish analysis performed with Metso Fibre Image Analyzer (Metso FS5), showing a distribution of average fibre length fractions in bleached chemical pulp made of softwood and hardwood at a pulp mill.

cellulose fibre length (mm)	hardwood (%)	softwood (%)
0 - 0.2	1.6	3.2
0.2 - 0.6	8.1	4.5
0.6 - 1.2	64.3	11.1
1.2 - 2.0	23.6	21.8
2.0 - 3.2	1.9	39.9
3.2 - 7.6	0.5	19.6

[0032] In addition to the average cellulose fibre length, also the proportion of hemicelluloses varies in the bleached chemical pulp made of softwood and hardwood. The two most common hemicelluloses, xylans and glucomannans, are present in different amounts in different wood species. Xylans are more prevalent in hardwoods, such as birch, while glucomannans are the dominant hemicelluloses in softwoods, such as pine and spruce. Bleached chemical pulp made of hardwood have been observed to better resist degradation of the material caused by high temperatures, which may occur upon compounding, wherein the polymer processing temperature may be well over 200°C.

Fibre furnish analysis

[0033] Fibre furnish analysis according to ISO standards ISO 9184-1 and 9184-4:1990 may be used in identification of papermaking fibres from pulp material. The analysis may be used, for example, to distinguish cellulose fibres produced by chemical, semi-chemical, such as chemithermomechanical, or mechanical method from each other. The analysis may further be used, for example, in differentiation of cellulose fibres produced by kraft or sulphite process in hardwood pulps and in differentiation of cellulose fibres from softwood and hardwood from each other. Metso Fibre Image Analyzer (Metso FS5) is an example of a device, which can be used according to the manufacturer's instructions to perform the fibre furnish analysis. For example, a high resolution camera may be used to acquire a greyscale image of a sample, of which image the properties of the fibres in the sample may be determined. The greyscale image may be acquired from a sample placed in a transparent sample holder, such as a cuvette, using a 0.5 millimetre depth of focus according to ISO 16505-2 standard. The wood species used in a pulp material may be distinguished by comparison method, wherein a sample fibre is compared against a known reference fibre. Fibre length may be determined according to ISO 16065-N.

Determination of moisture content and pulp content

[0034] The moisture content and pulp content of a chemical pulp suspension may be determined by a thermogravimetric method, which uses a balance unit and a heating unit for determining the weight loss of a sample due to drying.

[0035] The weight loss in a known amount of chemical pulp suspension due to drying is directly proportional to the moisture content of the chemical pulp suspension. When determining the pulp content of a pulp precursor material, a modified thermogravimetric method containing two consecutive steps is used, wherein first the moisture content of the pulp precursor material is determined, followed by determination of the pulp content, as disclosed below. The modified thermogravimetric method may, if necessary, be further used for determination of the pulp content of a compounded material, such as a fibre-based polymer composite formed of the pulp precursor material.

[0036] An example of a thermogravimetric method for determining the moisture content of a sample is oven drying, wherein the sample is placed in an aluminium container and the initial weight of the sample is determined with 0,001 g accuracy. The sample is then oven dried under laboratory conditions at a temperature of 120°C for 24 hours, followed by cooling the sample down to room temperature in an excicator. The dry weight of the sample is then determined with 0,001 g accuracy, thereby obtaining the weight loss of the sample due to oven drying, indicating the moisture content of the sample. The sample may be chemical pulp suspension or pulp precursor material.

[0037] Alternatively, the moisture content may be determined by infrared drying method. Infrared drying has the advantage of being a fast and precise method, when compared to oven drying method. In infrared drying, the sample is placed on a balance unit and heated by an infrared heat source, until the balance unit no longer detects weight loss due to drying. The moisture content of the sample is the total loss in weight due to drying. An example of infrared moisture analyser suitable for moisture content determination is Sartorius MA100, which may be used according to the manufacturer's instructions. The infrared heat source, such as a halogen lamp, a CQR quartz glass heater or a ceramic heating element, may be selected based on the material to be analyzed.

[0038] Once the moisture content of the sample is known, the pulp content of the pulp precursor material may be determined. The pulp content determination is a solvent-based analysis, wherein the thermoplastic compatibilizer (typically a polyolefin based polymer) is dissolved and extracted out of the pulp precursor material with decalin, the remaining pulp is dried and the weight of the dried pulp is determined. Decalin, which refers to decahydronaphthalene and has the formula $C_{10}H_{18}$ (CAS Registry Number 91-17-8), is an industrial solvent which can dissolve many types of resins, but which is insoluble to water. Therefore, the dry sample from the above-described moisture content determination may be used for the pulp content determination. Alternatively, a fresh sample may be first dried with infrared moisture analyser or oven dried as described above (120°C, 24 hours) to remove water and determine the moisture content of the sample. Subsequently, an amount in the range of 0.5 to 1 g of the dried material is weighed and added into 80 ml of decalin, thereby forming a mixture. The mixture is allowed to rest for 12 hours, followed by boiling the mixture for 8 hours, to ensure that all of the thermoplastic compatibilizer is dissolved into the decalin. After boiling, the mixture is filtered through a filter paper and the filtrate containing the decalin and the dissolved thermoplastic compatibilizer is discarded. The non-dissolved material remaining on the filter paper is oven dried at a temperature of 102°C for 24 hours, followed by cooling the dried material down to room temperature in an excicator. The obtained dried material is the amount of chemical pulp in the sample, which is weighed to calculate the pulp content of the pulp precursor material.

[0039] The pulp content, expressed in weight percentages (wt.%), therefore is the weight of the fibre-based components in the pulp precursor material, including cellulose fibres and hemicelluloses. The loss of weight that occurs upon oven drying or during infrared drying is the moisture content of the pulp precursor material. The rest, up to 100 wt.%, is the amount of thermoplastic compatibilizer and adsorption agent in the pulp precursor material, which have been added in weight percentages (wt.%) of the flash dried pulp.

[0040] Advantageously, the pulp content and the moisture content are determined as average values from a series of at least five samples of pulp precursor material, each sample having an initial weight in the range of 5 to 50 g, to improve the representativeness of the material under determination.

5 *Refining of the fibres*

[0041] Refining denotes a process by which cellulose fibres are mechanically treated to alter their intrinsic properties. As the purpose of chemical pulping is to degrade and dissolve the lignin in wood so that the cellulose fibres can be separated without mechanical treatment, the cellulose fibres typically remain well preserved. Refining may be used to increase the surface area of the cellulose fibres in the bleached chemical pulp suspension PLP1, when necessary. The refining may be carried out in a refining unit B configured to increase the surface area of the cellulose fibres in the bleached chemical pulp via fibrillation. The bleached chemical pulp suspension PLP1 may be refined at a pulp content up to 12 wt.% with a high consistency refiner, such as in the range of 4 to 12 wt.%. However, to avoid fibre curl, the refining of the bleached chemical pulp suspension PLP1 is preferably carried out at a consistency, wherein the pulp content is equal to or less than 6 wt.%. Examples of refining unit B suitable for the method for manufacturing pulp precursor material FIB1 are, for example, conical disc, flat disc and double disc refiners.

[0042] Refining of the bleached chemical pulp suspension PLP1 may have a positive effect for a fibre-based polymer composite CMP1 performance. The fibrillation may increase the available bonding surface of the cellulose fibres with adsorption agents CHEM1. However, excessive refining may also cause delamination, which decreases the ultimate strength of the fibres, thereby providing a negative effect for the composite CMP1 performance. Conversely, the reduction of stiff lamellar structure in the fibre cell wall may increase the flexibility of the fibres. These effects can be observed in the performance of a fibre-based polymer composite CMP1 containing refined fibres; when the refining enhances the fibre dispersion and the amount of coupled surface, it can lead to increased impact strength. However, the reduced strength of the fibres may result in a decrease of the tensile strength of the composite CMP1.

[0043] Refining changes the distribution of average fibre length fractions of the bleached chemical pulp. When the specific refining energy input (kWh/t) is increased, the amount of long fibre fraction in the bleached chemical pulp decreases while the amount of fine material increases. Thus, the refining decreases the average cellulose fibre length. In addition, refining may be used to decrease the pulp freeness, which denotes how fast or slow water drains through a fibre mat. Pulp freeness therefore is a measure of water intake into the fibres, and relates to the absorbency behaviour of the cellulose fibres.

[0044] Therefore, the specific refining energy of the refining may be based on the type bleached chemical pulp or the adsorption agent used in the method. For example, it has been observed, that the amount of adsorbed amount of a silane-based adsorption agent may be increased by increasing the specific refining energy of the bleached chemical pulp. Preferably, the specific refining energy of the refining is equal to or less than 100 kWh/tonne, more preferably equal to or less than 80 kWh/tonne, most preferably equal to or less than 40 kWh/tonne.

Surface treatment of the cellulose fibres with an adsorption agent

[0045] The chemical pulping process decreases the amounts of hemicelluloses and lignin in the fibres, thereby rendering the hydroxyl groups of the cellulose fibres more accessible on the fibre surface. This promotes the hydrogen bonding of the cellulose fibres towards each other, which is likely to cause agglomeration of the fibres later in the fibre-based polymer composite manufacturing process, in particular upon compounding the cellulose fibres together with a compounding polymer, such as a polyolefin.

[0046] An adsorption agent CHEM1 as disclosed herein denotes a small molecule or a compound that is suitable for surface interactions with the cellulose fibres in water suspension and may be arranged to reduce hydrogen bonding of the cellulose fibres towards each other. Adsorption agent CHEM1 thereby addresses the immiscibility of the cellulose fibres with common compounding polymers. Addition of an adsorption agent CHEM1 into a bleached chemical pulp suspension thus may be arranged to provide chemically surface-treated bleached chemical pulp fibres, with improved dispersion capability upon polymer compounding. The adsorption agent CHEM1 may be added into the bleached chemical pulp before mixing the bleached chemical pulp with a compounding polymer at a mixing unit G. Advantageously, the adsorption agent CHEM1 may be added into the pulp suspension PLP1 at a refining unit B or into refined pulp suspension RF1 in a reactor unit C, as the relatively high water content of the pulp suspension and the heat generated at the refining unit B may be arranged to improve the efficiency of the adsorption.

[0047] Alternatively, or in addition, the adsorption agent CHEM1 may be added at a dewatering unit D or when fluffing dewatered pulp DEW1 at a fluffing unit E.

[0048] Reference is made to Figure 2, which shows examples of chemical compounds that may act as adsorption agent CHEM1 with bleached chemical pulp containing cellulose fibres. A surfactant, in general, is a molecule that contains both a hydrophobic and hydrophilic end group. Cationic surfactants are examples of a molecule which can be arranged

to adsorb onto a surface of cellulose fibres, thereby lowering the free energy of the interphase between cellulose fibre surface and a polymer. Usually, the hydrophobic group consists of one or several hydrocarbon chains, while the hydrophilic group is an ionic or highly polar group. Cationic surfactant adsorption onto cellulose fibre surfaces of bleached chemical pulp inhibits the generation of fibre-networks. The addition of cationic surfactants may thus reduce agglomeration and enhance the dispersion of cellulose fibres into polymer containing composites. A cationic surfactant that may act as adsorption agent CHEM1 can be, for example, a polyallylamine or a strong cationic surfactant containing one long hydrocarbon chain CAT1 or two hydrocarbon chains CAT2. Besides cationic surfactants, also cationic polyelectrolytes may act as adsorption agent CHEM1 with bleached chemical pulp containing cellulose fibres. Cationic polyelectrolytes can be adsorbed onto the surface of the cellulosic fibres through electrostatic interactions, thereby saturating the fibre surface and inducing a charge reversal. However, the adsorption phenomenon varies depending on the properties of polyelectrolyte. An example of a polyelectrolyte that may act as adsorption agent can be, for example, a cationic, branched polyethyleneimine PEI containing terminal amine groups ($-NH_2$), which may be protonated into amino groups ($-NH_3^+$) in acidic conditions. Of particular interest are silane based compounds that may act as adsorption agents CHEM1 with bleached chemical pulp containing cellulose fibres. In particular, organosilanes containing amine or vinyl functional groups, especially aminosilanes and vinylsilanes such as aminopropyltriethoxysilane APTES and vinyltriethoxysilane VTES, have been observed to show nearly linear adsorption behaviour onto the surface of the cellulosic fibres, as evidenced by Figure 7. This has been surprising, since in aqueous solutions silane compounds have a polymerization tendency, which decreases the rate of adsorption. When the adsorption agent CHEM1 contains functional groups, such as amine groups of vinyl groups or cationic sites, the agent may be arranged to improve the affinity towards the thermoplastic compatibilizer POL1 upon compounding. Amine group ($-NH_2$), for example, is reactive with maleic anhydride. Maleic anhydride may be present in a thermoplastic compatibilizer POL1 that is added to the flash dried pulp DRY1 containing cellulose fibres, when manufacturing pulp precursor material FIB1. Of notice is, that in addition to improved affinity towards the thermoplastic compatibilizer POL1 in the method, cellulose fibre surfaces containing silane compounds have furthermore shown a fibre debonding effect, such that formation of cellulose fibre agglomerates prior to mixing the thermoplastic compatibilizer POL1 may be reduced.

[0049] The adsorption agent CHEM1 may be added in an amount up to 3 wt.%, calculated of the amount of flash dried pulp DRY1. Vinyl silane (vinyltriethoxysilane) and aminosilane (3-aminopropyltriethoxysilane) in particular have experimentally shown nearly linear adsorption behaviour in dosage amounts of up to 3 wt.%, as evidenced by Fig 7. Experimental data further suggests that adsorption agent CHEM1 which is added in an amount of equal to or less than 0.8 wt.% of the flash dried pulp DRY1, preferably in the range of 0.05 to 0.8 wt.% of the flash dried pulp DRY1, most preferably in the range of 0.1 to 0.3 wt.% of the flash dried pulp DRY1 already enhances the dispersion of the fibres and provides improved affinity towards the thermoplastic compatibilizer POL1. Addition of the adsorption agent CHEM1 in amount of 0.2 wt.% per dry pulp DRY1 already display excellent coverage of the cellulose fibre by the adsorption agent CHEM1, as can be evidenced from Figs 3 and 5. Addition of the adsorption agent CHEM1 in amount up to 0.2 wt.% per dry pulp DRY1 may in fact improve the mechanical properties of a fibre-based polymer composite manufactured from the pulp precursor material FIB1, such as tensile strength and/or impact strength, as evidenced by Figure 13. The adsorption agent CHEM1 may be added in larger amounts, such as up to 3 wt.% of the flash dried pulp DRY1. However, when the pulp precursor material FIB1 contains more than 0.3 wt.% of the adsorption agent CHEM1, the mechanical properties of a fibre-based polymer composite manufactured from the pulp precursor material FIB1, such as tensile strength and/or impact strength, may suffer, as evidenced by Figure 13.

[0050] With reference to Figure 1b, adding of the adsorption agent CHEM1 may be performed in water suspension to bleached chemical pulp suspension PLP1 in one or multiple occasions. Advantageously, the adding of the adsorption agent CHEM1 is performed in water suspension to refined pulp RF1, before or after dewatering the refined pulp by means of a dewatering unit D, such as a filter or a press. The adding of the adsorption agent CHEM1 may be performed, for example, in conjunction with the refining, at the refining unit B, or in a separate reactor unit C. A separate reactor unit C, such as a bleaching reactor, is advantageous, as this allows a precise control of the adsorption treatment. The use of a bleaching reactor furthermore may facilitate recycling or re-use the unabsorbed portion of the adsorption agent CHEM1, which has been added into the reactor unit C. However, the adding of the adsorption agent CHEM1 may also be performed while dewatering refined pulp suspension RC1 at a dewatering unit D, wherein the unabsorbed portion of the adsorption agent CHEM1 may then be collected from the filtrate RES1, which has been separated from the dewatered pulp DEW1. It is also possible to add the adsorption agent CHEM1 into a fluffing unit E, such that the adsorption agent CHEM1 interacts with the cellulose fibre surfaces when decreasing the bulk density of dewatered pulp DEW1, thereby providing fluffed pulp FLF1 containing cellulose fibres that contain adsorption agent CHEM1 on the fibre surface. Depending of the chemical properties of the adsorption agent CHEM1, some of the adsorption agent CHEM1 may end up to the moisture RES2, which leaves from the fluffing unit E upon fluffing the dewatered pulp DEW1.

[0051] The adding of the adsorption agent CHEM1 that is suitable for surface interactions with the cellulose fibres in water suspension may be performed before or after dewatering the pulp suspension PLP1 or the refined pulp suspension RF1 by pressing or filtering. When adding the adsorption agent CHEM1 into a separate reactor unit C, the pulp suspension

PLP1 or the refined pulp suspension RF1 may be diluted with water to have a pulp content in the range of 3 to 4 wt.%. The adsorption reaction may be performed in a room temperature or higher, such as in a temperature in the range of 20 to 70°C, in atmospheric pressure. The reaction time may be varied, typical reaction times being in the range of 5 to 120 minutes, the reaction time and temperature depending of the desired surface coverage of the adsorption agent CHEM1. With aminosilanes and vinylsilanes, shorter adsorption times in the range of 5 to 60 minutes, when reacted in room temperature in the range of 20 to 30°C, have displayed higher adsorption kinetics, the highest adsorption rate being achieved ca. 30 minutes after addition of the adsorption agent CHEM1, as evidenced by Figure 9. The adsorbed fraction of aminosilanes and vinylsilanes furthermore increases as a function of the specific refining energy, as evidenced by Figure 10, thereby evidencing the effect of refining to the adsorbed amounts of an adsorption agent in the pulp precursory material. A moderate refining of up to 100 kWh/t, such as equal to or less than 100 kWh/t, more preferably equal to or less than 80 kWh/t, most preferably equal to or less than 40 kWh/t, has further been observed to improve the impact strength of a fibre-based polymer composite, as evidenced by Figure 14. The abbreviation kWh/t is used to denote the units of specific refining energy used, i.e. the amount of energy transferred from the refiner's motor to the fibre, which can be calculated according to equation 1 (below)

$$\text{Equation 1: } SRE = \frac{WL - W0}{Tph}$$

, wherein SRE denotes the specific refining energy (kWh/tonne), WL denotes the motor load (kW), W0 denotes the 'no load energy' required to spin the rotor in a pulp slurry (kW) and Tph denotes tons of pulp slurry per hour. The pulp slurry refers to the consistency of the pulp suspension, that is, the pulp content, which in the context of bleached chemical pulp suspension PLP1 is equal to or less than 6 wt.%.

Drying of the cellulose fibres

[0052] When manufacturing pulp precursor material FIB1, the bleached chemical pulp suspension PLP1 needs to be dried sufficiently, before adding of thermoplastic compatibilizer POL1. To further avoid cellulose fibre agglomeration upon mixing with the thermoplastic compatibilizer POL1, the cellulose fibres that have been treated with the adsorption agent CHEM1 may further be separated from each other by fluffing, such that the dried pulp is provided with a high pulp content but sufficiently low bulk density, wherein the thermoplastic compatibilizer POL1 may be evenly distributed throughout the thereby formed pulp precursor material FIB1. The pulp content of the bleached chemical pulp suspension PLP1 or the refined pulp suspension RF1 or the pulp suspension from reactor RC1 may be increased from an inlet consistency in the range of 3 to 15 wt.%, preferably in the range of 4 to 6 wt.% or in the range of 10 to 13 wt.%, by dewatering the pulp, thereby providing dewatered pulp DEW1 having an outlet consistency higher than 30 wt.%. The dewatering unit D configured to decrease the water content of the pulp may be, for example, a pressure filter, a roll press or a screw press, which are conventionally used to thicken bleached chemical pulp at a temperature in the range of 50 to 70°C. The dewatering unit D may, when necessary, be arranged to comprise an inlet for adding adsorption agent CHEM1. Chemicals, such as the adsorption agent CHEM1, can be added in the method before the dewatering step, as long as the addition of the chemicals which may end up to the filtrate RES1 does not cause difficulties into the water circulation of the fibre line, which is a likely target of the filtrate RES1 from the filter/press D. An advantage of a pressure filter is, that it enables chemical modification with a higher pressure. The pressure filter may be configured to operate in multiple stages, thereby first removing water and/or exchanging solvent, if needed, and then introducing a modification chemical, such as an adsorption agent CHEM1. The pressure filter may comprise a further washing stage, whereby the chemical remaining residues are removed before further drying. A pressure filter therefore enables adsorption reactions, which would not otherwise be feasible in a water-based environment. The pressure filter may be used, for example, to improve the adsorption of silane based adsorption agents CHEM1. Ideally, the dewatering unit D is configured to provide a predictable thickening effect, such that dewatered pulp DEW1 having a stable outlet consistency in the range of 35 to 50 wt.% is obtained.

[0053] The dewatered pulp DEW1 may be further processed in a fluffing unit E, when necessary, to separate the cellulose fibres from each other, thereby decreasing the bulk density of the dewatered pulp DEW1 and obtaining fluffed pulp FLF1. An example of a fluffing unit E is a pulp fluffer, which may be used to improve the quality of the dewatered pulp DEW1 and the heat economy of the pulp drying in the method for manufacturing pulp precursor material FIB1. The fluffing unit E functions by disintegrating the cellulose fibres from each other, thereby decreasing the bulk density of the dewatered pulp DEW1. Typical fluffed pulp FLF1 of bleached chemical softwood can have a bulk density close to 55 kg/m³, which corresponds to a bulk value in the range of 18 to 20 cm³/g, when measured according to standard SCAN-C 33:80. Typical fluffed pulp FLF1 of bleached chemical hardwood can have a bulk density close to 60 kg/m³, which corresponds to a bulk value in the range of 15 to 17 cm³/g (SCAN-C 33:80). The bulk value as expressed herein refers

to the specific volume of a material and is the inverse of bulk density. In addition to separating the cellulose fibres from each other, the fluffing also reduces the moisture content of the dewatered pulp DEW1, thereby providing fluffed pulp FLF1 having a moisture content of less than 50 wt.%. Advantageously, the fluffing is preceded by refining of the pulp, in order to obtain maximum exposed surface for the fibres and optimum heat for the fluffing.

[0054] The remaining excess moisture within the dewatered pulp DEW1 or the fluffed pulp FLF1 can be evaporated in a flash drying unit F, thereby obtaining flash dried pulp DRY1 having a pulp content of equal to or higher than 80 wt.% and a moisture content of less than 10 wt.%. Advantageously, the flash drying is preceded by a fluffing, in order to increase the available surface area of the cellulose fibres to flash drying unit F. The flash drying may be arranged in two consecutive phases, each phase being controlled by temperature controller devices arranged to maintain constant temperature of exhaust drying air. In the 'flash off' - phase the dewatered pulp DEW1 or the fluffed pulp FLF1 is contacted with a stream of inlet drying gas, typically air, having a temperature in the range of 250 to 300°C. When the fibres contact with the hot drying gas, moisture evaporates rapidly from free wet surfaces of the cellulose fibres in direct contact with the inlet drying gas. As the dewatered pulp DEW1 or the fluffed pulp FLF1 may still contain close to 50 wt.% of moisture, most of the drying takes place at the 'flash off' - phase, near the inlet part of the flash drying unit F. The temperature and moisture difference between the inlet drying gas and the fibres creates a very high evaporation rate, and the surface water is flashed off from the fibres within a few seconds. The short contact time with the inlet drying gas also protects the pulp against the high temperatures, which could otherwise seriously degrade the hemicelluloses or cellulose fibre structure. The 'flash off' - phase is advantageously followed by a more gentle drying phase, wherein the dewatered pulp DEW1 or the fluffed pulp FLF1 is contacted with a second stream of heated drying gas having a temperature in the range of 150 to 200°C, such that the moisture evaporation continues inside the cellulose fibres, while the temperature of the now dry cellulose surface containing the adsorption agent CHEM1 begins to increase. The temperature of the latter drying phase is designed for lower temperatures, to prevent overheating of the pulp. Advantageously, the flash drying unit F may contain drying towers, which increase the retention time of heavier particles, which thereby travel more slowly through the flash drying unit F. The increased retention time based on particle weight may be arranged to enhance the drying effect to obtain uniform dryness. Advantageously, cyclone separator is used for separating the flash dried pulp DRY1 from the exhaust drying gas containing moisture RES3, such that the exhaust drying gas RES3 from the latter phase can be reheated and recirculated back into the 'flash off' - phase as the inlet drying gas. Thereby, flash dried pulp DRY1 containing cellulose fibres that have been treated with the adsorption agent CHEM1, wherein the flash dried pulp DRY1 has a moisture content of less than 10 wt.% may be obtained. When needed, the flash dried pulp DRY1 may be cooled after the last drying phase.

Mixing of surface treated cellulose fibres with thermoplastic compatibilizer

[0055] The pulp precursor material FIB1 may be obtained from the flash dried pulp DRY1 by mixing less than 12 wt.%, preferably equal or less than 8 wt.%, most preferably equal to or less than 7 wt.% of thermoplastic compatibilizer POL1 into the flash dried pulp DRY1, thereby obtaining pulp precursor material FIB1 which has a pulp content of equal to or higher than 80 wt.% and is suitable for compounding with a polymer POL2. As an example, the mixing may comprise,

- feeding the flash dried pulp DRY1 and the thermoplastic compatibilizer POL1 into the mixing unit G and
- mixing the flash dried pulp DRY1 and the thermoplastic compatibilizer POL1 in the mixing unit G together, such that an even dispersion of pulp precursor material FIB1 is formed and
- discharging the pulp precursor material FIB1 from the mixing unit G.

[0056] The thermoplastic compatibilizer POL1 may be a polymer. Preferably, the thermoplastic compatibilizer POL1 is selected from the group of biopolymers, such as bio-polyamide, polylactic acid and cellulose acetate, or synthetic polymers, such as synthetic polyamide, polycarbonates, polyethylene terephthalate, polystyrene, polystyrene copolymers, acrylonitrile-butadiene-styrene copolymer, styrene block copolymers and polyvinyl chloride, or polyolefins, such as polyethylene, high density polyethylene, low density polyethylene, linear low density polyethylene and polypropylene. Bio-polyamides such as polyamide 1010, which is the polycondensation product of 1,10-decamethylene diamine and 1,10-decanedioic diacid, may be obtained by chemical processing of castor oil, thereby providing a biopolymer that has been produced of fully natural raw materials. On the other hand, synthetic polyamides, such as polyamide PA12, which may be obtained from a multi-stage process of butadiene via laurolactam, may be advantageous due to a lower processing temperature around 180°C and excellent thermomechanical properties of the polymer, despite being a synthetic, non-biodegradable polyamide. A further advantage of polyamide as a thermoplastic compatibilizer POL1 may be the chemical compatibility with adsorption agents CHEM1 containing terminal amine groups (-NH₂), such as polyethyleneimine PEI.

[0057] A preferred thermoplastic compatibilizer POL1 suitable for use with polyolefin-based compounding polymers is a polyolefin or a polyolefin copolymer that has been modified to contain functional groups, such as maleic anhydride. Advantageously, the thermoplastic compatibilizer POL1 may be used to provide pulp precursor material FIB1 that has

improved dispersion characteristics at a compounding unit I, when the pulp precursor material FIB1 is compounded with a further polymer POL2, such that a composite product CMP1 with improved mechanical properties may be obtained. The improved mechanical properties may be obtained without concomitant melting of the thermoplastic compatibilizer POL1 with the flash dried pulp DRY1 upon mixing. Therefore, the thermoplastic compatibilizer POL1 preferably has an average particle size of equal to or less than 1 mm, preferably in the range of 100 to 800 micrometres. Preferably, the thermoplastic compatibilizer POL1 is polypropylene, preferably polypropylene that has been grafted to contain a coupling agent, such as maleic acid anhydride or a functional silane, for example a vinyl silane or methacrylic silane. The coupling agent may be used to provide a lower melting temperature or glass transition temperature for the thermoplastic compatibilizer POL1. A lower melting temperature or glass transition temperature facilitates the dispersion of the thermoplastic compatibilizer POL1 with the flash dried pulp DRY1. Since some coupling agents may be sensitive to residual moisture in the flash dried pulp DRY1, the pulp precursor material FIB1 may advantageously comprise at least 6 wt.% of the thermoplastic compatibilizer POL1, when the thermoplastic compatibilizer POL1 is a polymer that has been grafted to contain a coupling agent. Further, when the thermoplastic compatibilizer POL1 is a polymer that has been grafted to contain a coupling agent, an average particle size equal to or higher than 200 micrometres may be advantageous. When the average particle size of the thermoplastic compatibilizer POL1 is larger, the activity of the coupling agent may be better preserved. An advantage of using a polypropylene as a highly non-polar polymer with low surface energy, is the compatibility of the material with many commonly used compounding polymers POL2, in particular with other polyolefins. A further advantage of using a polypropylene based thermoplastic compatibilizer POL1 is the improved water repellence, which may be obtained with even small additions of polypropylene based thermoplastic compatibilizer POL1, such as less than 12 wt.%.

[0058] The advantage of adding only a small amount of thermoplastic compatibilizer POL1, such as an amount between 1 and 12 wt.%, preferably in the range of 3 to 10 wt.%, most preferably in the range of 6 to 8 wt.% of the pulp precursor material FIB1, is that while the small amount of thermoplastic compatibilizer POL1 facilitates the fibre-based polymer compounding, the compounder who manufactures a fibre-based composite product CMP1 from the pulp precursor material FIB1, has still the freedom to decide the type and amount of polymer to be used in a fibre-based composite product CMP1.

[0059] The mixing unit G may be, for example a fluffer or a mixer that can be used to provide a masterbatch and has thus been configured to admix thermoplastic compatibilizer POL1 polymer into the flash dried pulp DRY1. Thereby, pulp precursor material FIB1 which has a pulp content of equal to or higher than 80 wt.% and is suitable for compounding with a polymer is obtainable. Suitable mixers are, for example, batch or continuous mixers which are used for mixing dry ingredients with each other, thereby providing dry-blends, and which mixers normally do not melt the materials. Advantageously, due to the temperature-sensitive nature of the cellulose fibres, the mixer element geometry is adjustable, thereby allowing to control the mixing operation and the intensity of the mixing. The mixing unit G may be coupled with a compacting unit H to compact the material and reduce the moisture content of the material to be less than 0.5 wt.%. When needed, the mixing unit G and/or the compacting unit H may therefore comprise heating and cooling stages, such as in a hot-cold type of mixer. The mixing may thus further comprise heating the flash dried pulp DRY1 and the thermoplastic compatibilizer POL1 in the mixing unit G to a temperature, which is near the glass transition temperature of the polymer for enhancing the dispersion of the cellulose fibres with the thermoplastic compatibilizer POL1.

[0060] When needed, the flash dried pulp DRY1 may be compacted such that the bulk density of the flash dried pulp DRY1 is higher than 100 kg/m³, preferably in the range of 150 to 300 kg/m³, prior to mixing the thermoplastic compatibilizer POL1 into the flash dried pulp DRY1. Advantageously, however, the compacting is performed on the pulp precursor material FIB1, such that upon storage, the bulk density of the pulp precursor material FIB1 is higher than 100 kg/m³, preferably in the range of 150 to 300 kg/m³. When a specific size or shape of pulp precursor material FIB1 is necessitated by the composite CMP1 manufacturing method, the pulp precursor material may be compressed into granulates or pellets in the compacting unit H, such as a pelletizer, thereby forming an intermediate product GR1 of the pulp precursor material FIB1. The intermediate product GR1 of the pulp precursor material FIB1 may have a density of bulk higher than 300 kg/m³. A granulate or pellet having a bulk density of 600 kg/m³ or higher may be advantageous for storage and transportation, but has a disadvantage of being very hard. Pulp precursor material FIB1 compacted into a granulate or pellet having a density of 600 kg/m³, furthermore, can be difficult to disintegrate upon compounding at the compounding unit I, which is undesired. Therefore, advantageously, the pulp precursor material FIB1 is processed into a form of a granulate or pellet having a bulk density higher than 300 kg/m³, such as a bulk density between 300 and 600 kg/m³. An example of a compacting unit H is a strand pelletizer, wherein the pulp precursor material FIB1 can be heated, compressed and conveyed to a die head, wherein the compressed material coming from the die head is converted into strands that are cut into intermediate products GR1, such as pellets, after cooling and solidification. Alternatively, a pelletizing process may be used, wherein the compressed material coming from the die head is cut directly into intermediate products GR1, such as pellets, followed by cooling, for example by means of an air-cooled die-face pelletizer.

Adsorption of silane based adsorption agents onto cellulose fibres

[0061] Reference is made to Figure 7, which shows an isotherm (1h) of vinylsilane VTES (vinyltriethoxysilane, CAS number: 78-08-0, Wacker) and aminosilane APTES (3-aminopropyltriethoxysilane, CAS number: 919-30-2, Wacker) adsorption to bleached primary chemical pulp fibres (g/100g), measured as a function of the chemical dosage per dry pulp (wt.%). Reference is further made to Figure 8, which shows an isotherm (1h) of the adsorbed fraction (%) of vinyl silane and aminosilane to bleached primary chemical pulp fibres, measured as a function of the chemical dosage per dry pulp (wt.%).

[0062] As evidenced, silane based adsorption agents show nearly linear adsorption behavior in bleached primary chemical pulp. Of interest, aminosilane presented higher adsorption amounts in the range of 0.3 to 0.9 wt.%, compared to the adsorption amounts of vinyl silane in the range of 0 to 0.3 wt.%. This is further evidenced by the adsorbed fraction of the silane based adsorption agents, as aminosilane presented a higher adsorbed fraction in the range of 30 to 60%, whereas the adsorbed fraction of vinyl silane was less than 10 %.

[0063] The adsorption of the silane based adsorption agents was further evidenced by scanning electron microscopy, with reference made to Figures 3, 4, 5 and 6, which show images of bleached primary chemical pulp fibres treated with 0.2 or 0.8 wt.% addition of either vinylsilane VTES or aminosilane APTES. The samples in Figures 3 to 6 were imaged with scanning electron microscope (Sigma VP, Zeiss) using an acceleration voltage of 3.0 kV. The samples were attached onto the sample holder with carbon tape and coated with gold for 2 minutes (coating current 25 mA, K100X, Emitech).

[0064] The experimental results together demonstrate, that excellent results were also obtained with small additions of silane based adsorption agent. Figures 3 and 5 show that vinyl silane VTES or aminosilane APTES which is added in an amount of 0.2 wt.% of the flash dried pulp display excellent coverage and even adsorption of the adsorption agent CHEM1 onto the surface of bleached cellulose fibres.

[0065] The experimental results further demonstrate, that shorter adsorption times display higher adsorption, as can be observed from Figure 9. In the experiment, a 2 wt.% dosages of vinyl silane VTES and aminosilane APTES were used. The highest adsorption rate was achieved ca. 30 minutes after addition of the adsorption agent. When the reaction time was prolonged, the adsorption rate was decreased, which was likely caused by the aqueous environment in the bleached chemical pulp suspension, that led to silane polymerization.

[0066] The adsorbed fraction of aminosilanes and vinylsilanes furthermore increases as a function of the specific refining energy, as evidenced by Figure 10, wherein a 2 wt.% dosages of vinyl silane VTES and aminosilane APTES were used in three different suspensions of bleached primary chemical pulp fibres, wherein specific refining energy was either 0 kWh/t, 40 kWh/t or 80 kWh/t. The results demonstrate that a moderate refining up to 100 kWh/t, can improve the adsorption of the adsorption agent onto the fibre surface.

[0067] Reference is further made to Figure 11, which shows flash dried pulp having a pulp content of equal to or higher than 80 wt.% that has been treated with an adsorption agent and to Figure 12, which shows pellets made of pulp precursor material that has been compressed and pelletized with an industrial cutter (D-7000 Stuttgart 30, C F Scheer & Cie). The pellets have a bulk density of 600 kg/m³ and a moisture content of less than 0.5 wt.%. The pellets have an average diameter d1 of less than 10 mm.

[0068] Together, the experimental results demonstrate, that instead of using only a traditional grafted coupling agent upon compounding, bleached chemical pulp containing cellulose fibres can be first coated with small amount of an adsorption agent and subsequently mixed with a thermoplastic compatibilizer to improve the dispersion and miscibility of the cellulose fibres, prior to compounding with a polymer that contains a traditional grafted coupling agent. The experimental results further demonstrate, that it is not necessary to use high amounts of the adsorption agent CHEM1, in fact it may be beneficial to avoid oversaturating the cellulose fibre surface, as this may have a negative impact on the mechanical strength of a subsequently manufactured cellulose fibre-based polymer composite CMP1.

Mechanical properties of composite samples containing adsorption agent

[0069] Reference is made to Figure 13. The effect of pulp precursor material to the properties of a cellulose fibre-based polymer composite was studied in an experimental setup, wherein the tensile and impact strengths of cellulose fibre-based polymer composite samples was studied as a function of adsorption agent addition. The abbreviations CS-i, CL-i and C2-i in Figure 13 refer to the impact strength values (kJ/m²) and the abbreviations CS-t, CL-t and C2-t to the tensile strength values (MPa). The cellulose fibre-based polymer composite samples contained 43 wt.% pulp precursor material, of which the amount of thermoplastic compatibilizer (maleic anhydride grafted polypropylene) was 3 wt.%. The matrix polymer upon compounding had been polypropylene. The test samples were prepared with an Engel ES 200/40 injection moulding machine. The tensile strengths of the fibre-sheets were measured according to ISO 1924-3:2005 with extension rate of 100 mm/min. The sheets were conditioned at 23°C/50% relative humidity for so long that the mass of the sheets didn't change in two consecutive measurements which were one hour apart. The breaking length of the fibres was measured according to T 494 om-01. The tensile properties were measured with L&W Tensile Tester (Lorentzen

& Wettre).

[0070] Tensile analysis of the composites was conducted according to ISO 527. The samples were conditioned at 65% relative humidity and 20°C for 48 hours before conducting the testing with a tensile testing apparatus (Instron 3366) and a video extensometer. The measurement was performed with an extension rate of 50 mm/min using a 10 kN load cell. The tensile measurements were carried out with 5 parallel samples. The impact testing was performed via un-notched Charpy procedure according to ISO 179. The samples were conditioned at 65.0% relative humidity and 20°C for 48 hours before performing the testing with Instron Dynatup impact testing apparatus. The measurements were conducted to 10 parallel samples with an impact velocity of 2.9 m/s. The impact strength values (kJ/m²) and tensile strength values (MPa) were determined from samples having adsorption agent addition of 0.2 wt.%, 0.8 wt.%, and 1.5 wt.% of the flash dried pulp, wherein the adsorption agent was either a cationic surfactant having a short hydrocarbon chain CS, a long hydrocarbon chain CL or two hydrocarbon chains C2.

[0071] Without an adsorption agent addition the fibre-based polymer composite C2-t, CS-t had a tensile strength of 28 Mpa and an impact strength of 15 kJ/m². When the fibre-based polymer composite samples comprising pulp precursory material had been treated with cationic surfactant CS, C2 in an amount of 0.2 wt.% of the flash dried pulp, the composites presented a significant increase in the tensile strength CS-t, C2-t and in the impact strength CS-i, C2-i. At an adsorption agent addition of 0.2 wt.% of the flash dried pulp, the impact strength of the fibre-based polymer composite containing a cationic surfactant with two hydrocarbon chains C2-i was 51 Mpa and the impact strength was 33 kJ/m², while the tensile strength of the fibre-based polymer composite containing a cationic surfactant with short hydrocarbon chain was 48 Mpa and the impact strength was 32 kJ/m². The results suggest that small amounts of adsorption agent reduce the aggregation of the cellulose fibres in composite compounding. When an adsorption agent was added in amounts of equal to or higher than 0.2 wt.% of the flash dried pulp, such as in the range of 0.2 to 1.5 wt.% of the flash dried pulp, the fibre-based polymer composites had an impact strength equal to or higher than 27 kJ/m² and tensile strength equal to or higher than 44 MPa. However, when the adsorption agent was added in an amount higher than 0.8 wt.% of the flash dried pulp, the tensile and impact strengths of cellulose fibre-based polymer composite samples no longer improved.

[0072] Reference is made to Figure 14. The effect of pulp precursor material containing refined cellulose fibres was studied in an experimental setup, wherein the tensile and impact strengths of cellulose fibre-based polymer composite samples was studied as a function of specific refining energy of bleached primary chemical pulp. In the composite samples, the adsorption agent was added in an amount of 0.8 wt.% of the flash dried pulp. The samples contained pulp refined with specific refining energy at 0 kWh/t, 40 kWh/t or 80 kWh/t. The '0 kWh/t, Dried sheet' sample was a reference sample wherein dried sheet was refined instead of a never-dried pulp suspension. Interestingly, according to the experimental results, once-dried cellulose fibres have an reduced reinforcing effect compared to never-dried cellulose fibres, such as fibres of bleached chemical pulp obtained after washing and acidification, prior to the last high consistency bleaching tower of the pulping process. A moderate refining at 80 kWh/t presented the highest impact strength of 33.7 kJ/m². A refining of cellulose fibres prior to adding an adsorption agent induced a higher impact strength in the composite samples, indicating a better coupling between the cellulose fibres and the polymer matrix. However, the tensile strength decreased likely due to the refining-induced damage in the cellulose fibres. Thus a method for manufacturing pulp precursor material, wherein the specific refining energy is equal to or less than 100 kWh/tonne, more preferably equal to or less than 80 kWh/tonne, most preferably equal to or less than 40 kWh/tonne, is preferred, when tensile strength is desired.

Claims

1. A pulp precursor material for compounding with a polymer, the pulp precursor material comprising

- dried bleached chemical pulp containing cellulose fibres,
- adsorption agent on the surface of the cellulose fibres, said adsorption agent being for reducing hydrogen bonding of the cellulose fibres towards each other, and
- less than 12 wt.% of thermoplastic compatibilizer which has been mixed with the dried bleached chemical pulp, said thermoplastic compatibilizer being for improving miscibility of the cellulose fibers with hydrophobic polymers;

wherein the pulp content of the pulp precursor material is equal to or higher than 80 wt.% of the pulp precursor material and the moisture content of the pulp precursor material is less than 10 wt.%.

2. A method for manufacturing pulp precursor material for compounding with a polymer, the method comprising:

- adding adsorption agent that is suitable for surface interactions with cellulose fibres to bleached chemical pulp,

said adsorption agent being for reducing hydrogen bonding of the cellulose fibres towards each other,
 - dewatering the pulp by pressing or filtering such that a pulp content in the range of 40 to 50 wt.% is obtained,
 - evaporating moisture from the pulp in a flash drying unit, thereby obtaining dried pulp having a moisture content
 of less than 10 wt.%, and

- mixing less than 12 wt.% of thermoplastic compatibilizer into the dried pulp, thereby obtaining pulp precursor
 material which has a pulp content of equal to or higher than 80 wt.% and which is suitable for compounding
 with a polymer, said thermoplastic compatibilizer being for improving miscibility of the cellulose fibers with
 hydrophobic polymers.

3. The method according to claim 2, further comprising increasing the cellulose fibre surface area of bleached chemical
 pulp by refining, prior to adding the adsorption agent; and preferably, wherein, upon refining,

- the bleached chemical pulp has a pulp content in the range of 4 to 12 wt.%, preferably equal to or less than
 6 wt.%; and/or, preferably, wherein

- the specific refining energy of the refining is equal to or less than 100 kWh/tonne, more preferably equal to or
 less than 80 kWh/tonne, most preferably equal to or less than 40 kWh/tonne.

4. The method according to any of the claims 2 to 3, further comprising

- after dewatering the pulp, separating cellulose fibres from each other in a fluffing unit, thereby decreasing the
 bulk density and obtaining fluffed pulp having a bulk value in the range of 15 to 20 cm³/g and a moisture content
 of less than 50 wt.%; and/or

- compacting the dried pulp, such that the bulk density of the dried pulp is higher than 100 kg/m³, preferably in
 the range of 150 to 300 kg/m³; and/or

- compressing the pulp precursor material into granulates or pellets having a bulk density higher than 300 kg/m³,
 such as a between 300 and 600 kg/m³, and a moisture content of less than 0.5 wt.%.

5. The method according any of the claims 2 to 4, wherein the adding of the adsorption agent is performed

- in a refining unit and/or
 - in a reactor unit before or after dewatering the pulp, and/or
 - in a dewatering unit arranged to comprise an inlet for adding the adsorption agent, and/or
 - in a fluffing unit, when decreasing the bulk density of the pulp,

thereby providing cellulose fibres that contain adsorption agent on the fibre surface.

6. The method according to any of the claims 2 to 5, wherein the bleached chemical pulp is

- a suspension of bleached primary chemical pulp obtained after washing and acidification, prior to the last high
 consistency bleaching tower of the pulping process, such that the pulp content of the suspension is in the range
 of 3 to 12 wt.%, preferably equal to or less than 6 wt.%, most preferably equal to or less than 4 wt.%; and/or
 - a water-based suspension of never-dried Kraft pulp from a pulping process.

7. The pulp precursor material according to claim 1 or the method according to any of the claims 2 to 6, wherein the
 bleached chemical pulp is hardwood pulp or softwood pulp.

8. The pulp precursor material or the method according to any of the preceding claims, wherein the adsorption agent is

- a cationic surfactant or a polyelectrolyte; or
 - a polyethyleneimine or a polyallylamine; or
 - a silane based compound, such as aminosilane or vinylsilane.

9. The pulp precursor material or the method according to any of the preceding claims, wherein the amount of the
 adsorption agent is

- equal to or less than 0.8 wt.% of the dried pulp, such as in the range of 0.05 to 0.8 wt.% of the dried pulp;
 preferably,
 - equal to or less than 0.3 wt.% of the dried pulp, such as in the range of 0.05 to 0.3 wt.% of the dried pulp;

more preferably,

- in the range of 0.1 to 0.3 wt.% of the dried pulp, such as 0.2 wt.% of the dried pulp.

10. The pulp precursor material or the method according to any of the preceding claims, wherein the thermoplastic compatibilizer is selected from the group of

- biopolymers, such as polyamide, polylactic acid and cellulose acetate, or
- synthetic polymers, such as polycarbonates, polyethylene terephthalate, polystyrene, polystyrene copolymers, acrylonitrile-butadiene-styrene copolymer, styrene block copolymers and polyvinyl chloride, or
- polyolefins, such as polyethylene, high density polyethylene, low density polyethylene, linear low density polyethylene and polypropylene.

11. The pulp precursor material or the method according to any of the preceding claims, wherein the thermoplastic compatibilizer

- has an average particle size of equal to or less than 1 mm, preferably in the range of 100 to 800 micrometres; and/or
- is polypropylene, preferably polypropylene that has been grafted to contain a coupling agent, such as maleic acid anhydride.

12. A system for manufacturing pulp precursor material which has a pulp content of equal to or higher than 80 wt.% for compounding with a polymer, the system comprising:

- an inlet configured to receive bleached chemical pulp containing cellulose fibres from a pulping process,
- adding means for adding adsorption agent to the surface of cellulose fibres,
- a dewatering unit configured to decrease the water content of the pulp such that a pulp content in the range of 40 to 50 wt.% is obtainable,
- a flash drying unit configured to evaporate moisture from the pulp such that dried pulp having a pulp content of equal to or higher than 80 wt.% and a moisture content of less than 10 wt.% is obtainable, and
- a mixing unit configured to admix thermoplastic compatibilizer polymer into the dried pulp, such that pulp precursor material which has a pulp content of equal to or higher than 80 wt.% and is suitable for compounding with a polymer is obtainable.

13. The system according to claims 12, further comprising

- a refining unit configured to increase the surface area of the cellulose fibres in the bleached chemical pulp; and/or
- a fluffing unit configured to decrease the bulk density of the pulp, such that fluffed pulp having a moisture content of less than 50 wt.% is obtainable; and/or
- a compacting unit configured to provide granulates or pellets having a bulk density higher than 300 kg/m³, such as between 300 and 600 kg/m³, and a moisture content of less than 0.5 wt.%.

14. The system according to any of the claim 12 to 13, wherein the adding means for adding the adsorption agent is a reactor unit, such as a bleaching reactor; and/or an inlet to the refining unit and/or dewatering unit and/or fluffing unit, which inlet is further configured to receive the adsorption agent.

Patentansprüche

1. Zellstoffvorläufermaterial zum Compoundieren mit einem Polymer, wobei das Zellstoffvorläufermaterial umfasst:

- getrockneten gebleichten chemischen Zellstoff, der Cellulosefasern enthält;
- ein Adsorptionsmittel auf der Oberfläche der Cellulosefasern, wobei das Adsorptionsmittel zur Verringerung einer Wasserstoffbrückenbindung der Cellulosefasern miteinander ist, und
- weniger als 12 Gew.-% eines thermoplastischen Verträglichkeitsvermittlers, der mit dem getrockneten gebleichten chemischen Zellstoff gemischt wurde, wobei der thermoplastische Verträglichkeitsvermittler zur Verbesserung einer Mischbarkeit der Cellulosefasern mit hydrophoben Polymeren ist;

wobei der Zellstoffgehalt des Zellstoffvorläufermaterials gleich oder größer als 80 Gew.-% des Zellstoffvorläufer-

materials ist und der Feuchtigkeitsgehalt des Zellstoffvorläufermaterials kleiner als 10 Gew.-% ist.

2. Verfahren zur Herstellung eines Zellstoffvorläufermaterials zum Compoundieren mit einem Polymer, wobei das Verfahren umfasst:

- Zugabe eines Adsorptionsmittels, das für Oberflächenwechselwirkungen mit Cellulosefasern geeignet ist, zu gebleichtem chemischem Zellstoff, wobei das Adsorptionsmittel zur Verringerung einer Wasserstoffbrückenbindung der Cellulosefasern miteinander ist,
- Entwässern des Zellstoffs durch Pressen oder Filtrieren, so dass ein Zellstoffgehalt im Bereich von 40 bis 50 Gew.-% erhalten wird,
- Abdampfen von Feuchtigkeit von dem Zellstoff in einer Schnelltrocknungseinheit, wodurch getrockneter Zellstoff mit einem Feuchtigkeitsgehalt kleiner als 10 Gew.-% erhalten wird, und
- Mischen von weniger als 12 Gew.-% eines thermoplastischen Verträglichkeitsvermittlers in den getrockneten Zellstoff, wodurch ein Zellstoffvorläufermaterial erhalten wird, das einen Zellstoffgehalt gleich oder größer als 80 Gew.-% aufweist und das zum Compoundieren mit einem Polymer geeignet ist, wobei der thermoplastische Verträglichkeitsvermittler zur Verbesserung einer Mischbarkeit der Cellulosefasern mit hydrophoben Polymeren ist.

3. Verfahren nach Anspruch 2, weiterhin umfassend

ein Erhöhen des Zellulosefaseroberflächenbereichs von gebleichtem chemischem Zellstoff durch Veredeln vor dem Zugabe des Adsorptionsmittels; und vorzugsweise wobei nach dem Veredeln

- der gebleichte chemische Zellstoff einen Zellstoffgehalt im Bereich von 4 bis 12 Gew.-%, vorzugsweise gleich oder kleiner als 6 Gew.-% aufweist; und/oder vorzugsweise wobei
- die spezifische Veredelungsenergie des Veredelns gleich oder kleiner als 100 kWh/Tonne, mehr bevorzugt gleich oder kleiner als 80 kWh/Tonne, am meisten bevorzugt gleich oder kleiner als 40 kWh/Tonne ist.

4. Verfahren nach einem der Ansprüche 2 bis 3, weiterhin umfassend

- nach dem Entwässern des Zellstoffs ein Trennen von Cellulosefasern voneinander in einer Fluffing-Einheit, wodurch die Füllichte verringert wird und ein gebluffter Zellstoff mit einem Füllwert im Bereich von 15 bis 20 cm³/g und einem Feuchtigkeitsgehalt kleiner als 50 Gew.-% erhalten wird; und/oder
- Verdichten des getrockneten Zellstoffs, so dass die Füllichte des getrockneten Zellstoffs höher als 100 kg/m³ ist, vorzugsweise im Bereich von 150 bis 300 kg/m³ liegt; und/oder
- Komprimieren des Zellstoffvorläufermaterials zu Granulat oder Pellets mit einer Füllichte größer als 300 kg/m³, wie zwischen 300 und 600 kg/m³, und einem Feuchtigkeitsgehalt kleiner als 0,5 Gew.-%.

5. Verfahren nach einem der Ansprüche 2 bis 4, wobei das Zugabe des Adsorptionsmittels

- in einer Veredelungseinheit und/oder
- in einer Reaktoreinheit vor oder nach dem Entwässern des Zellstoffs und/oder
- in einer Entwässerungseinheit, die dazu eingerichtet ist, einen Einlass zum Zugabe des Adsorptionsmittels zu umfassen, und/oder
- in einer Fluffing-Einheit, wenn die Füllichte des Zellstoffs verringert wird, durchgeführt wird,

wodurch Cellulosefasern bereitgestellt werden, die Adsorptionsmittel auf der Faseroberfläche enthalten.

6. Verfahren nach einem der Ansprüche 2 bis 5, wobei der gebleichte chemische Zellstoff

- eine Suspension von gebleichtem primärem chemischem Zellstoff, die nach einem Waschen und einem Ansäuern vor dem letzten Dickstoffbleichturm des Aufschlussvorgangs erhalten wird, so dass der Zellstoffgehalt der Suspension im Bereich von 3 bis 12 Gew.-% liegt, vorzugsweise gleich oder kleiner als 6 Gew.-%, am meisten bevorzugt gleich oder kleiner als 4 Gew.-% ist; und/oder
- eine wasserbasierte Suspension von nie getrocknetem Kraftzellstoff aus einem Aufschlussvorgang ist.

7. Zellstoffvorläufermaterial nach Anspruch 1 oder Verfahren nach einem der Ansprüche 2 bis 6, wobei der gebleichte chemische Zellstoff Laubholzhalbstoff oder Nadelholzhalbstoff ist.

8. Zellstoffvorläufermaterial oder Verfahren nach einem der vorhergehenden Ansprüche, wobei das Adsorptionsmittel

- ein kationisches Tensid oder ein Polyelektrolyt; oder
- ein Polyethylenimin oder ein Polyallylamin; oder
- eine silanbasierte Verbindung, wie Aminosilan oder Vinylsilan, ist.

9. Zellstoffvorläufermaterial oder Verfahren nach einem der vorhergehenden Ansprüche, wobei die Menge des Adsorptionsmittels

- gleich oder kleiner als 0,8 Gew.-% des getrockneten Zellstoffs ist, wie im Bereich von 0,05 bis 0,8 Gew.-% des getrockneten Zellstoffs; vorzugsweise
- gleich oder kleiner als 0,3 Gew.-% des getrockneten Zellstoffs ist, wie im Bereich von 0,05 bis 0,3 Gew.-% des getrockneten Zellstoffs; mehr bevorzugt
- im Bereich von 0,1 bis 0,3 Gew.-% des getrockneten Zellstoffs liegt, wie 0,2 Gew.-% des getrockneten Zellstoffs.

10. Zellstoffvorläufermaterial oder Verfahren nach einem der vorhergehenden Ansprüche, wobei der thermoplastische Verträglichkeitsvermittler aus der Gruppe von

- Biopolymeren, wie Polyamid, Polymilchsäure und Celluloseacetat, oder
- synthetischen Polymeren, wie Polycarbonaten, Polyethylenterephthalat, Polystyrol, Polystyrol-Copolymeren, Acrylnitril-Butadien-Styrol-Copolymer, Styrol-Blockcopolymeren und Polyvinylchlorid, oder
- Polyolefinen, wie Polyethylen, Polyethylen hoher Dichte, Polyethylen niedriger Dichte, lineares Polyethylen niedriger Dichte und Polypropylen, ausgewählt ist.

11. Zellstoffvorläufermaterial oder Verfahren nach einem der vorhergehenden Ansprüche, wobei der thermoplastische Verträglichkeitsvermittler

- eine durchschnittliche Teilchengröße gleich oder kleiner als 1 mm, vorzugsweise im Bereich von 100 bis 800 Mikrometer aufweist; und/oder
- Polypropylen, vorzugsweise Polypropylen, das gepropft wurde, um ein Kopplungsmittel zu enthalten, wie Maleinsäureanhydrid, ist.

12. System zur Herstellung eines Zellstoffvorläufermaterials, das einen Zellstoffgehalt gleich oder größer als 80 Gew.-% aufweist, zum Compoundieren mit einem Polymer, wobei das System umfasst:

- einen Einlass, der dazu konfiguriert ist, gebleichten chemischen Zellstoff, der Cellulosefasern enthält, aus einem Aufschlussvorgang aufzunehmen,
- ein Zugabemittel zum Zugeben eines Adsorptionsmittels zu der Oberfläche von Cellulosefasern,
- eine Entwässerungseinheit, die dazu konfiguriert ist, den Wassergehalt des Zellstoffs zu verringern, so dass ein Zellstoffgehalt im Bereich von 40 bis 50 Gew.-% erhalten werden kann,
- eine Schnelltrocknungseinheit, die dazu konfiguriert ist, Feuchtigkeit aus dem Zellstoff abzdampfen, so dass getrockneter Zellstoff mit einem Zellstoffgehalt gleich oder größer als 80 Gew.-% und einem Feuchtigkeitsgehalt kleiner als 10 Gew.-% erhalten werden kann, und
- eine Mischeinheit, die dazu konfiguriert ist, thermoplastisches Verträglichkeitsvermittlerpolymer in den getrockneten Zellstoff zu mischen, so dass ein Zellstoffvorläufermaterial, das einen Zellstoffgehalt gleich oder größer als 80 Gew.-% aufweist und zum Compoundieren mit einem Polymer geeignet ist, erhalten werden kann.

13. System nach Anspruch 12, weiterhin umfassend

- eine Veredelungseinheit, die dazu konfiguriert ist, den Oberflächenbereich der Cellulosefasern in dem gebleichten chemischen Zellstoff zu vergrößern; und/oder
- eine Fluffing-Einheit, die dazu konfiguriert ist, die Füllichte des Zellstoffs zu verringern, so dass gebluffter Zellstoff mit einem Feuchtigkeitsgehalt kleiner als 50 Gew.-% erhalten werden kann; und/oder
- eine Verdichtungseinheit, die dazu konfiguriert ist, Granulat oder Pellets mit einer Füllichte größer als 300 kg/m³, wie zwischen 300 und 600 kg/m³, und einem Feuchtigkeitsgehalt kleiner als 0,5 Gew.-% bereitzustellen.

14. System nach einem der Ansprüche 12 bis 13, wobei das Zugabemittel zum Zugeben des Adsorptionsmittels eine Reaktoreinheit, wie ein Bleichreaktor; und/oder ein Einlass zu der Veredelungseinheit und/oder der Entwässerungseinheit, umfasst.

serungseinheit und/oder der Fluffing-Einheit ist, wobei dieser Einlass weiterhin dazu konfiguriert ist, das Adsorptionsmittel aufzunehmen.

5 Revendications

1. Matériau précurseur de pulpe pour composition avec un polymère, le matériau précurseur de pulpe comprenant

- de la pulpe chimique blanchie séchée contenant des fibres de cellulose,
- un agent d'adsorption sur la surface des fibres de cellulose, ledit agent d'adsorption étant pour réduire les liaisons hydrogène des fibres de cellulose les unes vers les autres et
- moins de 12 % en poids d'agent compatibilisant thermoplastique qui a été mélangé avec la pulpe chimique blanchie séchée, ledit agent compatibilisant thermoplastique étant pour améliorer la miscibilité des fibres de cellulose avec des polymères hydrophobes ;

la teneur en pulpe du matériau précurseur de pulpe étant supérieure ou égale à 80 % en poids du matériau précurseur de pulpe et la teneur en humidité du matériau précurseur de pulpe étant de moins de 10 % en poids.

2. Procédé de fabrication d'un matériau précurseur de pulpe pour composition avec un polymère, le procédé comprenant :

- l'addition d'un agent d'adsorption qui est approprié pour les interactions de surface avec les fibres de cellulose à la pulpe chimique blanchie, ledit agent d'adsorption étant pour réduire les liaisons hydrogène des fibres de cellulose les unes vers les autres,
- la déshydratation de la pulpe par pressage ou filtration de telle manière qu'une teneur en pulpe dans la plage de 40 à 50 % en poids est obtenue,
- l'évaporation de l'humidité de la pulpe dans une unité de séchage instantanée, obtenant ainsi de la pulpe séchée ayant une teneur en humidité de moins de 10 % en poids et
- le mélange de moins de 12 % en poids d'un agent compatibilisant thermoplastique dans la pulpe séchée, obtenant ainsi un matériau précurseur de pulpe qui a une teneur en pulpe supérieure ou égale à 80 % en poids et qui est appropriée pour la composition avec un polymère, ledit agent compatibilisant thermoplastique étant pour améliorer la miscibilité des fibres de cellulose avec des polymères hydrophobes.

3. Procédé selon la revendication 2, comprenant en outre l'augmentation de la surface de fibre de cellulose de pulpe chimique blanchie par raffinage, avant d'ajouter l'agent d'adsorption ; et de préférence, après raffinage,

- la pulpe chimique blanchie ayant une teneur en pulpe dans la plage de 4 à 12 % en poids, de préférence inférieure ou égale à 6 % en poids ; et/ou, de préférence,
- l'énergie de raffinage spécifique du raffinage étant inférieure ou égale à 100 kWh/tonne, davantage de préférence inférieure ou égale à 80 kWh/tonne, de préférence entre toutes inférieure ou égale à 40 kWh/tonne.

4. Procédé selon l'une quelconque des revendications 2 à 3, comprenant en outre

- après déshydratation de la pulpe, séparation des fibres de cellulose les unes des autres dans une unité de gonflage, diminuant ainsi la masse volumique et obtenant la pulpe gonflée ayant une valeur de volume dans la plage de 15 à 20 cm³/g et une teneur en humidité de moins de 50 % en poids ; et/ou
- le compactage de la pulpe séchée, de telle manière que la masse volumique de la pulpe séchée est supérieure à 100 kg/m³, de préférence dans la plage de 150 à 300 kg/m³ ; et/ou
- la compression du matériau précurseur de pulpe en granulés ou comprimés ayant une masse volumique supérieure à 300 kg/m³, tel qu'entre 300 et 600 kg/m³ et une teneur en humidité de moins de 0,5 % en poids.

5. Procédé selon l'une quelconque des revendications 2 à 4, dans lequel l'addition de l'agent d'adsorption est effectuée

- dans une unité de raffinage et/ou
- dans une unité de réacteur avant ou après la déshydratation de la pulpe et/ou
- dans une unité de déshydratation disposée pour comprendre une entrée pour ajouter l'agent d'adsorption et/ou
- dans une unité de gonflage, quand on diminue la masse volumique de la pulpe,

fournissant ainsi des fibres de cellulose qui contiennent de l'agent d'adsorption sur la surface de la fibre.

6. Procédé selon l'une quelconque des revendications 2 à 5, dans lequel la pulpe chimique blanchie est

- une suspension de pulpe chimique primaire blanchie obtenue après lavage et acidification, avant la dernière tour de blanchiment de haute consistance du processus de pulpage, de telle manière que la teneur en pulpe de la suspension se situe dans la plage de 3 à 12 % en poids, de préférence inférieure ou égale à 6 % en poids, de préférence entre toutes inférieure ou égale à 4 % en poids ; et/ou
- une suspension à base d'eau de pulpe Kraft jamais séchée à partir d'un processus de pulpage.

7. Matériau précurseur de pulpe selon la revendication 1 ou procédé selon l'une quelconque des revendications 2 à 6, dans lequel la pulpe chimique blanchie est de la pulpe de bois dur ou de la pulpe de bois tendre.

8. Matériau précurseur de pulpe ou procédé selon l'une quelconque des revendications précédentes, dans lequel l'agent d'adsorption est

- un tensioactif cationique ou un polyélectrolyte ; ou
- une polyéthylèneimine ou une polyallylamine ; ou
- un composé à base de silane, tel qu'un aminosilane ou un vinylsilane.

9. Matériau précurseur de pulpe ou procédé selon l'une quelconque des revendications précédentes, dans lequel la quantité de l'agent d'adsorption est

- inférieure ou égale à 0,8 % en poids de la pulpe séchée, tel que dans la plage de 0,05 à 0,8 % en poids de la pulpe séchée ; de préférence,
- inférieure ou égale à 0,3 % en poids de la pulpe séchée, tel que dans la plage de 0,05 à 0,3 % en poids de la pulpe séchée ; davantage de préférence,
- dans la plage de 0,1 à 0,3 % en poids de la pulpe séchée, tel que 0,2 % en poids de la pulpe séchée.

10. Matériau précurseur de pulpe ou procédé selon l'une quelconque des revendications précédentes, dans lequel l'agent compatibilisant thermoplastique est choisi dans le groupe de

- les biopolymères, tels que le polyamide, l'acide polylactique et l'acétate de cellulose ou
- les polymères synthétiques, tels que les polycarbonates, le téréphtalate de polyéthylène, le polystyrène, les copolymères de polystyrène, le copolymère acrylonitrile-butadiène-styrène, les copolymères séquencés de styrène et le chlorure de polyvinyle ou
- les polyoléfines, telles que le polyéthylène, le polyéthylène haute densité, le polyéthylène basse densité, le polyéthylène basse densité linéaire et le polypropylène.

11. Matériau précurseur de pulpe ou procédé selon l'une quelconque des revendications précédentes, dans lequel l'agent compatibilisant thermoplastique

- a une taille de particule moyenne inférieure ou égale à 1 mm, de préférence dans la plage de 100 à 800 micromètres ; et/ou
- est le polypropylène, de préférence le polypropylène qui a été greffé pour contenir un agent de couplage, tel que l'anhydride d'acide maléique.

12. Système de fabrication de matériau précurseur de pulpe qui a une teneur en pulpe supérieure ou égale à 80 % en poids pour composition avec un polymère, le système comprenant :

- une entrée configurée pour recevoir de la pulpe chimique blanchie contenant des fibres de cellulose d'un processus de pulpage,
- des moyens d'addition pour ajouter l'agent d'adsorption à la surface des fibres de cellulose,
- une unité de déshydratation configurée pour diminuer la teneur en eau de la pulpe de telle manière qu'une teneur en pulpe de 40 à 50 % en poids peut être obtenue,
- une unité de séchage instantané configurée pour évaporer l'humidité de la pulpe de telle manière que la pulpe séchée ayant une teneur en pulpe supérieure ou égale à 80 % en poids et une teneur en humidité de moins de 10 % en poids peut être obtenue et

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- une unité de mélange configurée pour mélanger le polymère agent compatibilisant thermoplastique dans la pulpe séchée, de telle manière que le matériau précurseur de pulpe qui a une teneur en pulpe supérieure ou égale à 80 % en poids et est approprié pour la composition avec un polymère peut être obtenu.

5 **13.** Système selon la revendication 12, comprenant en outre

- une unité de raffinage configurée pour augmenter la surface des fibres de cellulose dans la pulpe chimique blanchie ; et/ou

10 - une unité de gonflage pour diminuer la masse volumique de la pulpe, de telle manière qu'une pulpe gonflée ayant une teneur en humidité de moins de 50 % en poids peut être obtenue ; et/ou

- une unité de compactage configurée pour fournir des granulés ou des comprimés ayant une masse volumique supérieure à 300 kg/m³, telle qu'entre 300 et 600 kg/m³ et une teneur en humidité de moins de 0,5 % en poids.

15 **14.** Système selon l'une quelconque des revendications 12 à 13, dans lequel le moyen d'addition pour ajouter l'agent d'adsorption est

une unité de réacteur, telle qu'un réacteur de blanchiment ; et/ou une entrée à l'unité de raffinage et/ou l'unité de déshydratation et/ou l'unité de gonflage, laquelle entrée est en outre configurée pour recevoir l'agent d'adsorption.

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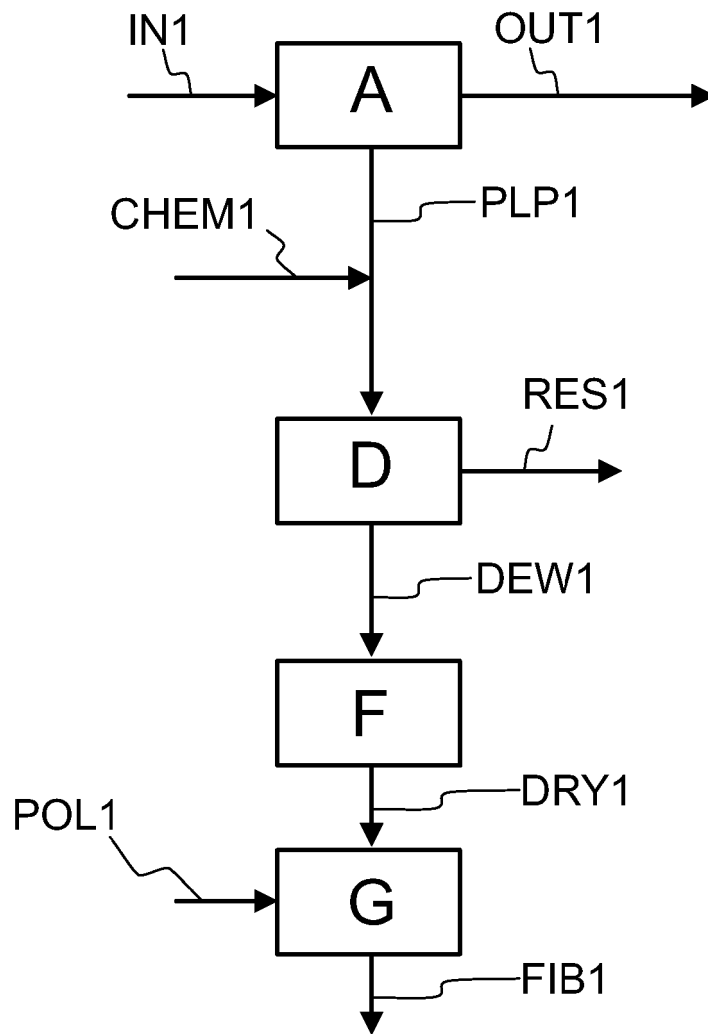


Fig. 1a

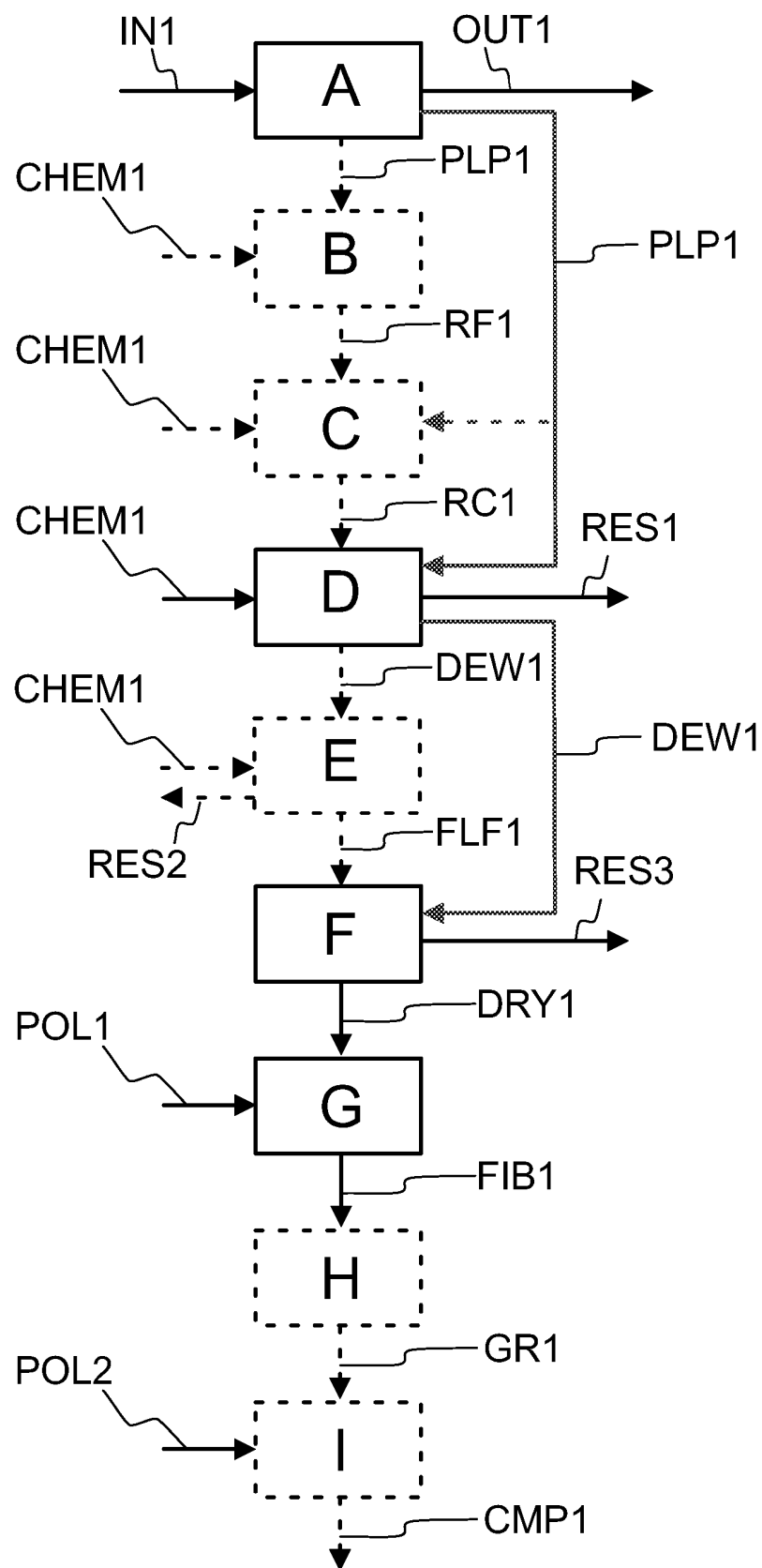


Fig. 1b

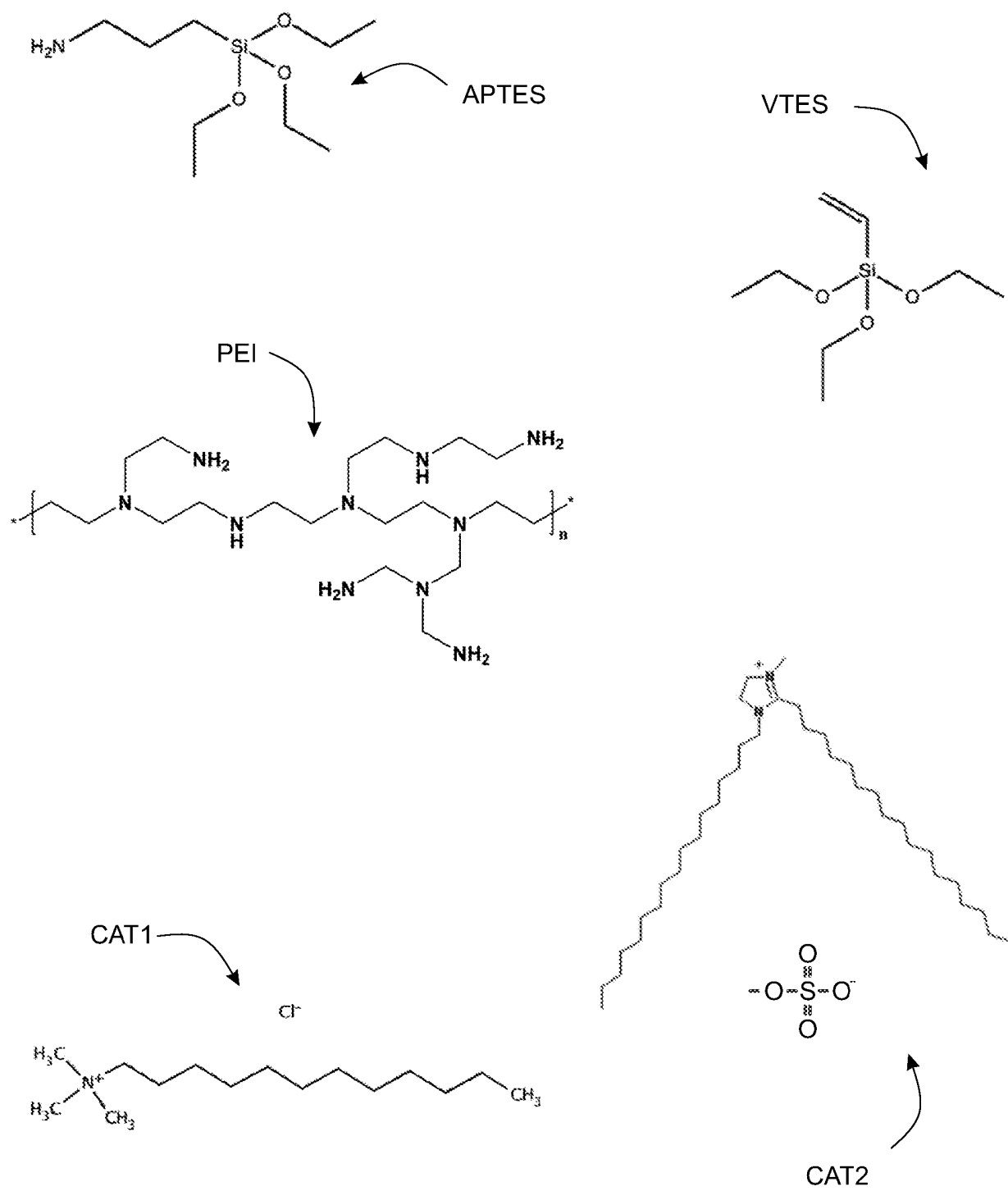


Fig. 2

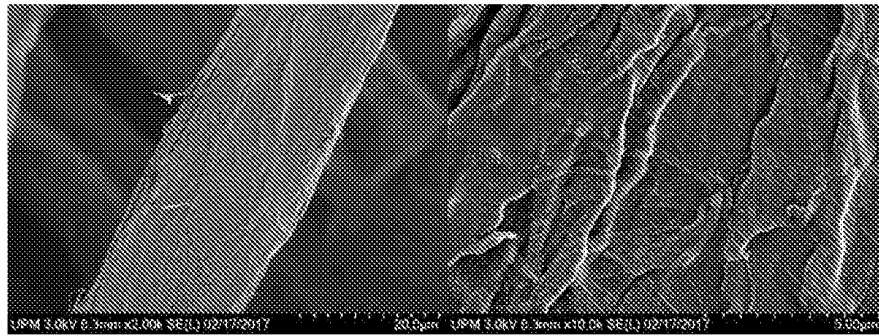


Fig. 3

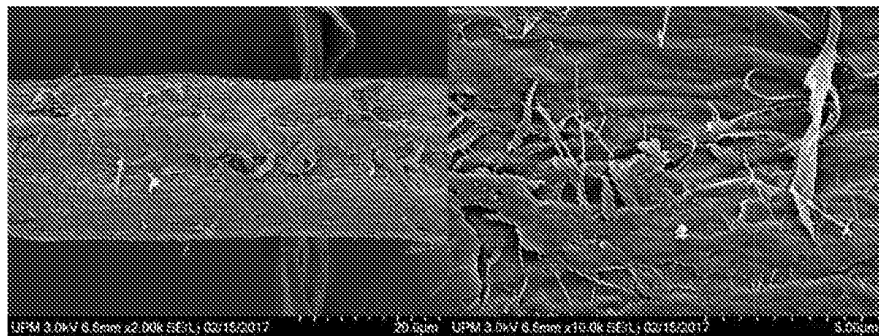


Fig. 4

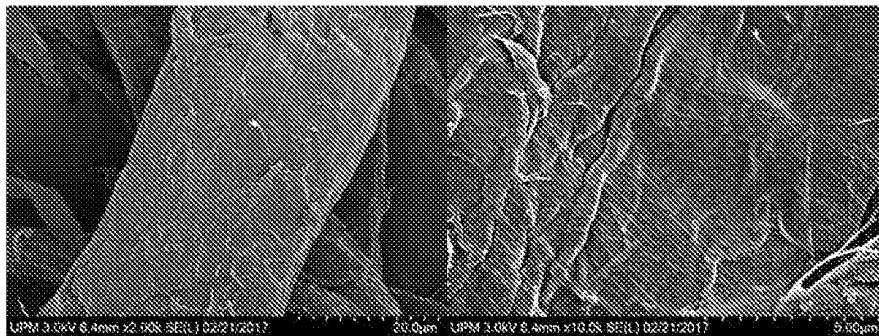


Fig. 5

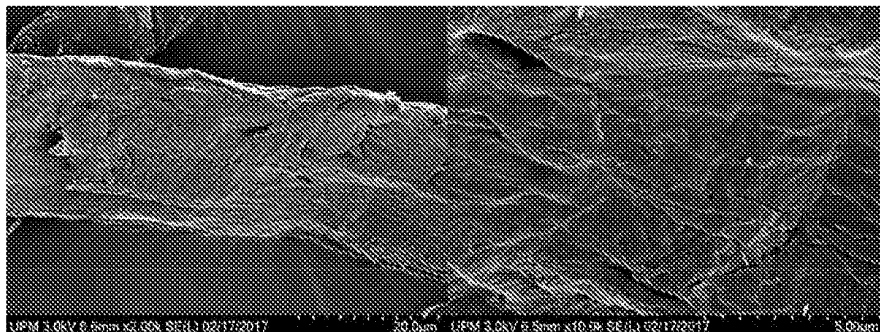


Fig. 6

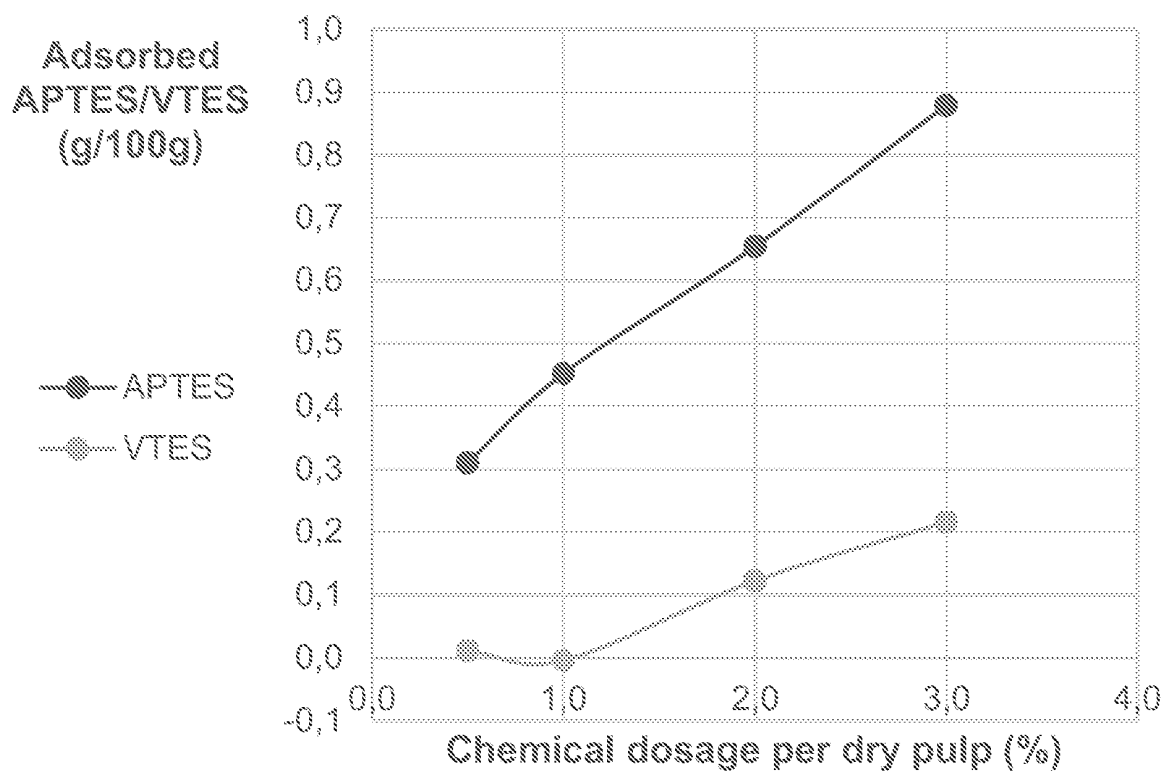


Fig. 7

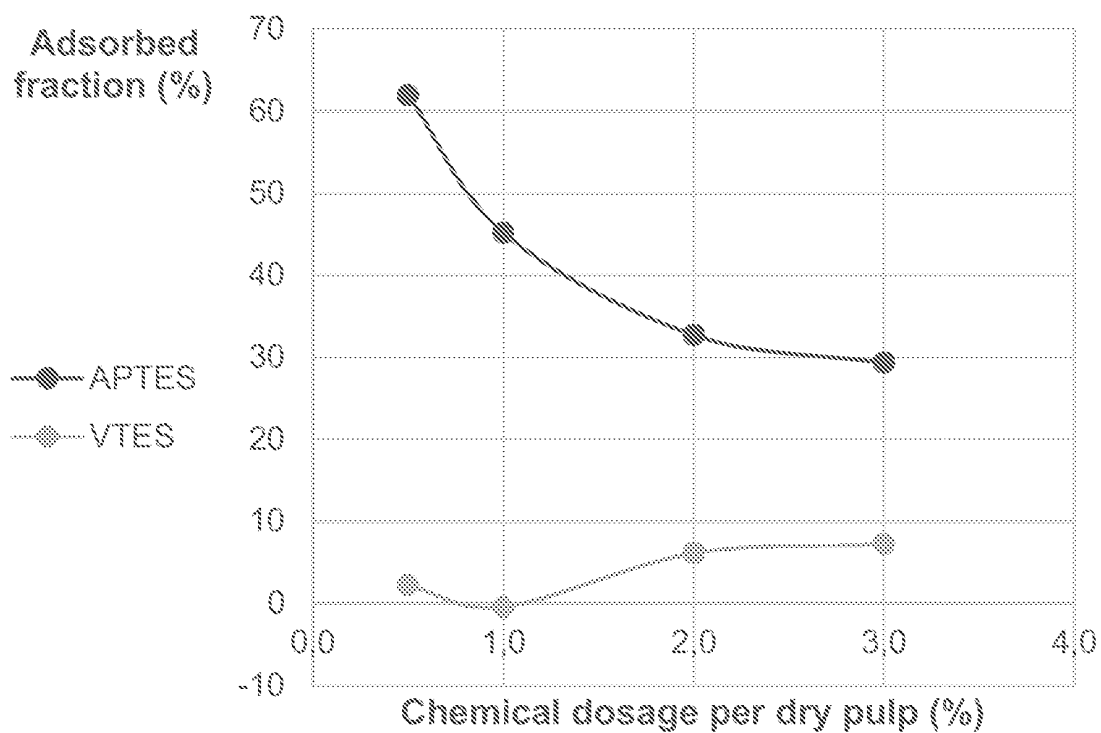


Fig. 8

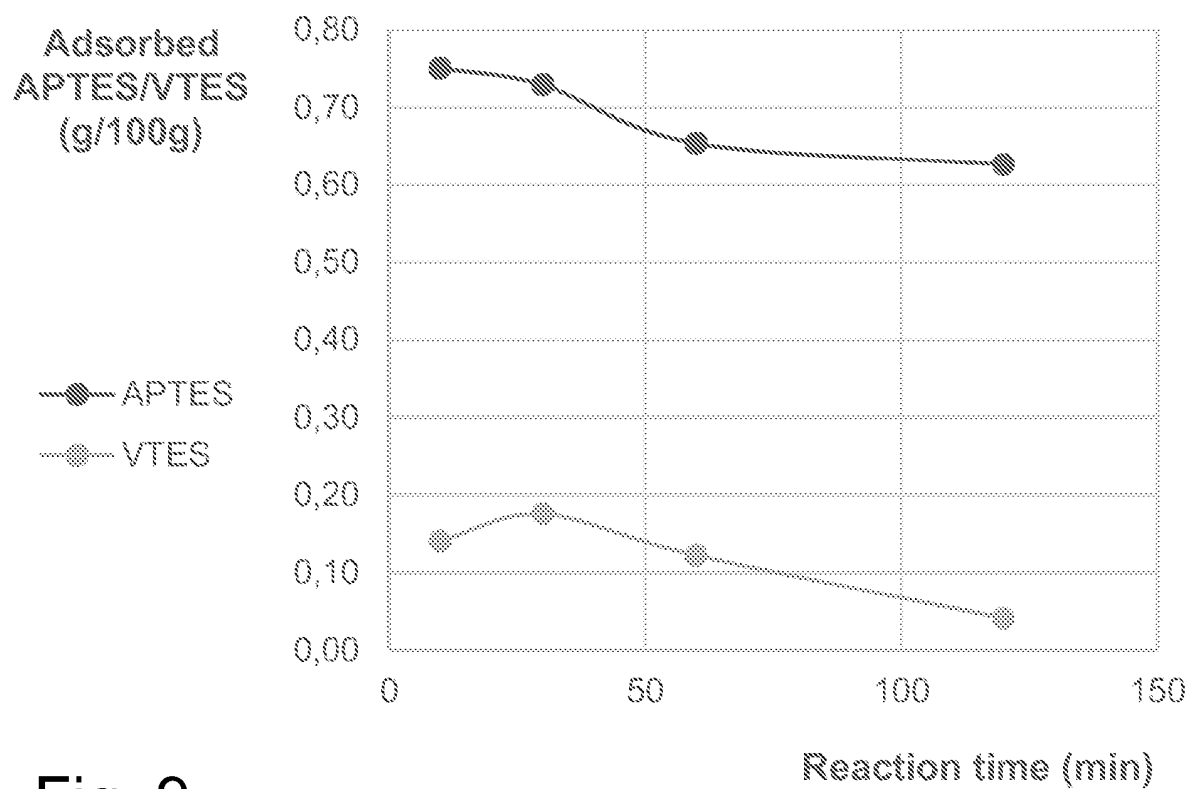


Fig. 9

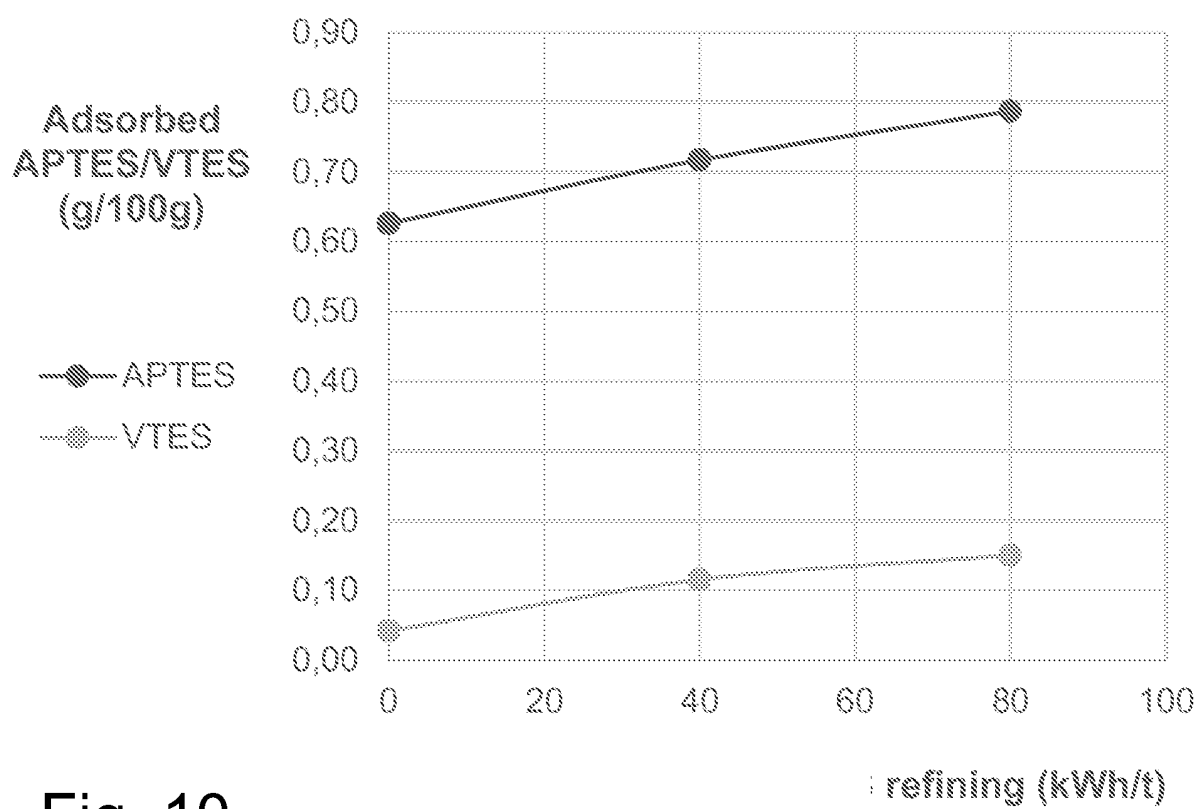


Fig. 10

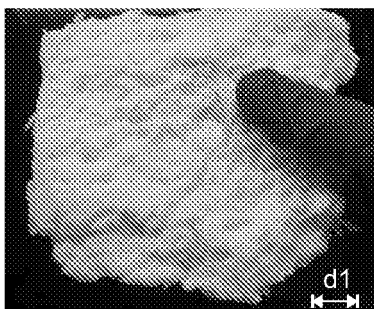


Fig. 11

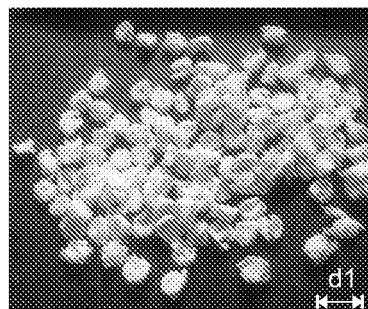


Fig. 12

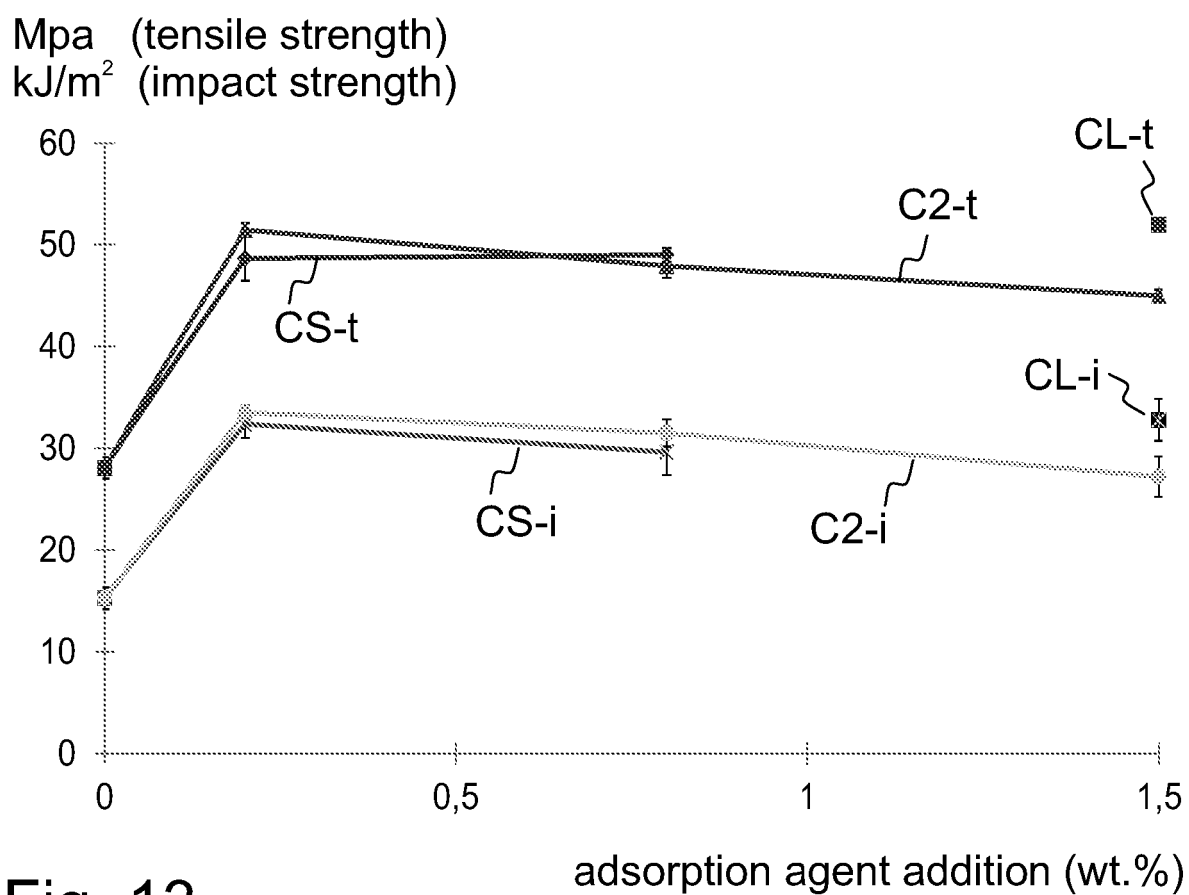


Fig. 13

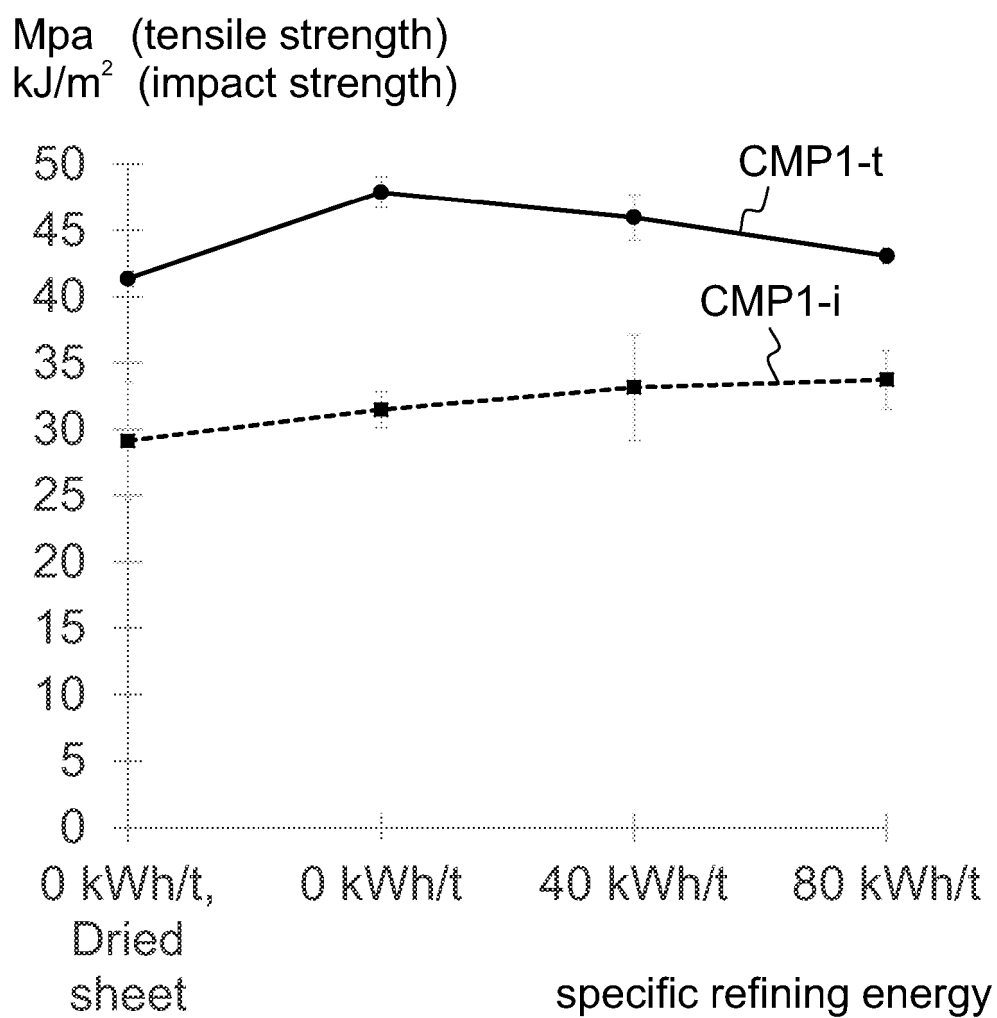


Fig. 14

REFERENCES CITED IN THE DESCRIPTION

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