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(54) **A THREE-DIMENSIONAL BIODEGRADABLE FIBRE NETWORK PRODUCT OF NATURAL ORGANIC FIBRES, METHOD OF PREPARATION AND USE THEREOF**

(57) The invention relates to a method for the preparation of three-dimensional biodegradable fibre network product, the method comprising foaming natural organic fibres in aqueous solution, mould filling with the foamed natural organic fibres, wherein the mould has a plurality of pores, forming a three-dimensional biodegradable fibre network product by providing electromagnetic energy to the foamed natural organic fibres, wherein the plurality

of pores is adapted to evacuate water and steam generated by providing electromagnetic energy to the foamed natural organic fibres.

The invention also relates to a three-dimensional biodegradable fibre network product and use thereof as a plant growth substrate, filtration medium, filling and/or acoustic and mechanical damping structure, thermal isolator, efficient moisture absorber and evaporator.

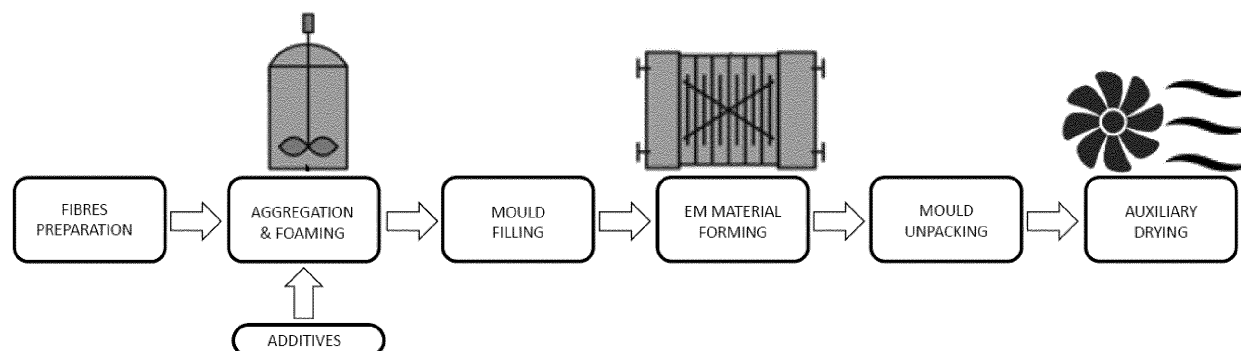


Fig. 1

Description

Technical Field

[0001] The present invention relates to a biodegradable three-dimensional fibre network product from defibrated natural organic fibres, method of preparation of the fibre network product using electromagnetic (EM) energy and use thereof.

Background Art

[0002] Natural organic fibres of plants or animal origin have been widely used for many years in many industries, such as paper and wood industry, due to advantageous mechanical properties including good tensile strength, low weight and specific stiffness, but also due to its renewable character and ability to be broken down by bacteria making them environment friendly.

[0003] In the paper industry, cellulose fibres used as raw material are wetted, converted into a pulp, pressed, and dried giving sheets of paper having a substantially flat shape, wherein the fibres are oriented substantially in the sheet-plane direction, which results in a product having a good tensile strength in said sheet-plane direction. Compression into thin sheets of paper allows for effective removal of water from the material, whereas production of thicker sheets is limited and requires more energy to dry the final product.

[0004] Recently, foam forming techniques become the subject of interest in the production of lightweight products for thermal insulation and sound absorption. Burke, et al., "Properties of lightweight fibrous structures made by a novel foam-forming technique", (2019), Cellulose 26, 2529-2539, describes a method for the production of lightweight fibrous structures of low densities. The method is based on the use of liquid foam as a carrier medium for dispersed Kraft fibres by slow draining and drying until all foam has disappeared. The procedure resulted in bulk samples whose height (up to 25 mm) and density were controlled by initial fibre concentration and liquid fraction of the foam. The problem with this method is degradation of the foam during draining of water excess and long time required for draining and drying to preserve as much of the structure in the initial web form secured by the foam.

[0005] Timofeev, et al., "Drying of foam-formed mats from virgin pine fibers", (2016), Drying Technology, 34:10, 1210-1218, describes drying of foam-formed mats from virgin pine fibres using the steps of fibre foam preparation, draining of the liquid, and drying with the use of different drying methods, namely convective drying in the oven, impingement drying assisted by vacuum, combined impingement-infrared drying, and through-air drying. Shrinkage of the final product was observed in all tested drying methods with the lowest shrinkage observed for combined techniques.

[0006] Alimadadi, et al., "3D-oriented fiber networks made by foam forming", (2016), Cellulose 23, 661-671,

describes form forming method to create the networks of 3D fibre orientation (3DFN). The described method results in the production of sheets having out-of-plane fibre orientation with high bulk and low density by creation of the 3D fibre orientation in the foam and maintaining the orientation during the forming, pressing, and drying stages.

[0007] Wood fibres are widely used for the manufacturing of fibreboards (such as MDF or HDF), however, in order to obtain desired properties of the fibreboard, fibres are mixed with a synthetic binder and formed into panels by hot-pressing. Synthetic binders used in the production of fibreboards are not environmentally friendly and such materials have limited uses.

[0008] There is a need to provide methods of manufacturing of biodegradable three-dimensional fibrous web structures of arbitrary shapes and easily controllable properties, such as density and stiffness. Forming of such materials using defibrated cellulose fibres and other biodegradable components still presents a challenge.

Summary of Invention

[0009] An aspect of the present invention is to provide a method for the preparation of a three-dimensional biodegradable fibre network product using electromagnetic energy.

[0010] According to this aspect, the method allows to control density and anisotropy inside the product and forming it into any shape. In a particular aspect, the method has very short forming times of the product.

[0011] The method according to this aspect of the present invention is defined by claim 1. Preferable embodiments of the method according to this aspect are defined in dependent claims.

[0012] Another aspect of the present invention is to provide a three-dimensional biodegradable fibre network product as defined by claim 12. Preferable embodiments of the product according to this aspect are defined in dependent claims.

[0013] According to this aspect, the product having good mechanical strength and stability is provided. In a particular aspect, the product has high over 95% porosity and low material density down to 8 kg/m³.

[0014] Another aspect of the present invention is to provide a use of said product as defined in claim 15.

Brief Description of Drawings

[0015] Preferred embodiments of the present invention are subsequently described with respect to the accompanying drawings, in which:

Fig. 1 is a block diagram illustrating the method according to an embodiment,

Fig. 2 illustrates a closed mould according to an embodiment,

- Fig. 3 is an example illustrating the interior of a mould,
- Fig. 4 illustrates a mould according to an embodiment in cross-section with indication of pressure gradients,
- Fig. 5 shows water retention curve for the material according to an embodiment,
- Fig. 6 illustrates different embodiments of a mould integration with electromagnetic (EM) field delivery device, cross-sectional view,
- Fig. 7 illustrates the product prepared according to example 1,
- Fig. 8 illustrates the product prepared according to example 2,
- Fig. 9 illustrates the product prepared according to example 3.

Description of Embodiments

[0016] It is noted that references in the specification to "an embodiment", "one embodiment", "another embodiment", etc., indicate that the embodiment described may include one or more features. Additionally, when features are described in connection with one embodiment, it should be understood that such features may also be used in connection with other embodiments whether or not explicitly described unless clearly stated to the contrary.

[0017] According to the first aspect, the present invention provides a method for the preparation of a three-dimensional biodegradable fibre network product, the method comprising:

foaming natural organic fibres in aqueous solution,

mould filling with the foamed natural organic fibres, wherein the mould has a plurality of pores,

forming a three-dimensional biodegradable fibre network product by providing electromagnetic energy to the foamed natural organic fibres,

wherein the plurality of pores is adapted to evacuate water and steam generated by providing electromagnetic energy to the foamed natural organic fibres.

[0018] The method according to the present invention allows for controlling of density gradient of the three-dimensional biodegradable fibre network product in any direction in the whole space of the mould.

[0019] Density gradient of the three-dimensional biodegradable fibre network product prepared by the method according to the present invention is controlled by the arrangement of the plurality of pores in a mould. Density

gradient of the three-dimensional biodegradable fibre network product prepared by the method according to the present invention is also controlled by the kind and/or power density of electromagnetic energy provided to the foamed natural organic fibres. Preferably, density gradient of the three-dimensional biodegradable fibre network product prepared by the method according to the present invention is controlled by the arrangement of the plurality of pores in a mould and by the kind and/or power density of electromagnetic energy provided to the foamed natural organic fibres. Mould with fewer pores having foamed fibres subjected to electromagnetic energy with higher power densities of electromagnetic energy results in obtaining higher density gradient in the three-dimensional biodegradable fibre network product.

[0020] The density of the fibre network product prepared by the method according to the present invention is also controlled by the density of the foam. Lowering foam density, leads to a fibre network product with lower density, and with higher density gradient.

[0021] Providing electromagnetic energy to the foamed natural organic fibres in a mould with a plurality of pores results in volumetric heating of the foamed natural organic fibres, that leads to the generation of steam and increase in pressure. This results in reorientation of the fibres in certain direction from the inside of the mould. These directions are controlled by the arrangement of the pores adapted to evacuate water and steam. The arrangement of the pores comprises pore size, pore shape, pore direction, number of pores in the mould, distance from the pores. The arrangement of the pores sets direction of the steam release path causing compaction of the fibres at the walls of the mould, leading to reinforcement of the final fibre network product. Density gradient of the fibre network product is therefore controlled in a wide range. Keeping the pressure uniformly spread inside the mould and at low level provides a more uniform density of the fibre network product. On the other hand, with higher pressures and/or less uniform pressure distribution higher density gradient are obtained in the fibre network product. In one specific example, the method provides a three-dimensional biodegradable fibre network product, which is empty inside. Also, the density of the structure is controlled by the amount of natural organic fibres put into the mould.

[0022] Providing electromagnetic energy comprises one or more phases, preferably an initial phase and a final phase. During the initial phase electromagnetic energy is delivered intensively to reach water boiling point, which results in forcing the excess of water out of the mould. This initial phase saves energy and time required to evaporate the remaining water, which should be removed and preserves the fibrous web/mesh structure from collapsing/shrinking inside the mould. During the final phase of providing electromagnetic energy, bonds between natural organic fibres are created forming fibre network product. Depending on desired properties of the fibre network product, different electromagnetic energy

levels can be required in consecutive phases, for instance, in order to prevent local overheating of the material.

[0023] In the present invention, only natural organic fibres are used as raw material. These natural organic fibres can be of any type and size. Physical and mechanical properties of the fibre network product prepared according to the present invention, such as strength and flexibility can be controlled by the proportion of different fibres. A similar situation occurs with the biological properties of the fibre network product obtained with the present invention. Combination of different proportions of fibres will have a significant impact on biological resistance.

[0024] As an example, pure cellulose fibres as well as ligno-cellulose fibres that have been defibrated mechanically can be used as natural organic fibres for the present invention. Cellulose fibres similar to those used in paper production (after removing the lignin), plant fibres, and other organic fibres, multiversity of which is expected due to their nature can be used. Also, ligno-cellulose fibres that are fractioned mechanically (without removing the lignin) are suitable.

[0025] Natural organic fibres of one type or as mixture of different type of fibres can be used with the present invention (e.g., by weight: 50% cellulose fibres, 50% hemp fibres - a composition that is more crack resistant than 100% cellulose). Crack resistance is achieved by incorporating long (up to 30mm) natural fibres into the foam. The likely mechanism is that there is an increase in the interaction between a greater number of fibres per volume of the product.

[0026] In one embodiment, the content of natural organic fibres in three-dimensional fibre network product is at least 95% on a dry basis.

[0027] In one embodiment, the length of natural organic fibres is from 0.1 cm to 3.0 cm.

[0028] In one embodiment, natural organic fibres are cellulose fibres.

[0029] In another embodiment, natural organic fibres are ligno-cellulose fibres.

[0030] Yet, in another embodiment, natural organic fibres are a combination of cellulose fibres and ligno-cellulose fibres.

[0031] According to the method of the present invention, foaming natural organic fibres is performed in aqueous solution. The parameters of the foam, and particularly the degree of foaming, have a significant effect on the internal structure of the final fibre network product prepared by the method of the present invention. Foam is a good dispersing medium for fibres in the three-dimensional network and any suitable method of foaming known in the prior art can be used for the method according to the present invention. The size of the foam bubbles determines the distribution of fibres in the three-dimensional space. Therefore, controlling the bubbles allow for obtaining a controlled density gradient in the fibre network product prepared by the method of the present in-

vention.

[0032] In one embodiment, foaming natural organic fibres in aqueous solution is performed by introducing a gas into the pulp.

[0033] The size and homogeneity of the foam bubbles are influenced by the different phases of the forming process. The stage of preparing the batch of material gives the possibility of shaping the character of the foam by adding to the mass some additives: blowing agents increase the amount of the gas filling the bubbles, surfactants control the foam's susceptibility to foaming. The appropriate selection of foam stabilizers allows the foam to maintain the desired properties until the fibres stiffen and take over the role of a supporting skeleton a structure that has so far been held by vanishing bubbles.

[0034] In one embodiment, aqueous solution used for foaming natural organic fibres further comprises at least one biodegradable non-fibrous additive comprising a foam stabilizer, foaming agent, biodegradable blowing agent or combination thereof.

[0035] Biodegradable foam stabilizers in form of polysaccharides can be used with the method of the present invention. In particular, chitosan and/or agar are preferable biodegradable foam stabilizers. Their main goal of foam stabilizers is to extend the life of wet foam, and to support the mechanical stability of the final product.

[0036] Biodegradable foaming agents meeting environmental standards can be used with the method of the present invention. In particular, coco glucoside is a preferred foaming agent.

[0037] Biodegradable blowing agents introduced into the water solution can also be used as an aid in the formation of foam. The preferred blowing agents are sodium carbonate and sodium bicarbonate, which have minimal impact on the environment.

[0038] In another embodiment, aqueous solution used for foaming natural organic fibres further comprises at least one further additive for controlling biomechanical properties of the obtained fibre network product, wherein said further additive comprise a polysaccharide, polysaccharide derivative, lignin, lignin derivative, cellulose, and a cellulose derivative.

[0039] Other non-fibrous additives can be used with the method of the present invention to define end parameters of the material. Some of the additives have a double role, as a material stabilizer and foam enhancers. Preferably, biomaterials, that are at least partially dissolvable in water are used. Preferred non-fibrous additives are agar and chitosan, polysaccharides, that are helping with moisture control and stiffness of the product. Agar gel acts as a foam stabilizer, that extends the life of wet foam, and after electromagnetic forming it acts as a gluing agent, improving the strength of bonds between fibres.

[0040] Hydrophilic additives (e.g., chitosan) can be added for agricultural uses of the product obtained by the method according to the present invention, for maintaining moisture for a prolonged time. Biological additives

(e.g., grapefruit extract) can be used for extension of the material lifecycle.

[0041] Water insoluble, hydrophobic additives (e.g., mineral powders) can be added for creating solutions for construction applications for water repellence.

[0042] In one embodiment, polysaccharides are used as a foam additive to increase the durability of the fibre network product after the forming process. The suitable polysaccharides comprise agarose, chitosan and combination thereof. Agarose mechanically stabilizes the material after forming, by strengthening the bonds between the fibres and securing their surface mechanically. Chitosan, in addition to performing the function of mechanical strengthening, is known for its biocidal properties, protects the material against excessive biological aging.

[0043] In another embodiment, starch is also used as a foam additive, which increases the stiffness of the material after the molding process.

[0044] Starch is a potential additive, that has impact on mechanical properties of the final material. It makes the outer layer more rigid and brittle.

[0045] In one embodiment lignin is used as an additive. Lignin may be introduced to increase mechanical strength and water resistance of final product.

[0046] Furthermore, in the present invention, chemical and mechanical derivatives of cellulose can be used as mechanical stabilizers or modifiers of the fibre surface. Examples include cellulose ethers, for example, methyl cellulose and ethyl cellulose, known for their use as industrial rheology modifiers. They can be used as foam stabilizing agents and modifier of interactions between the fibre network product and solvents, either polar or nonpolar. Other cellulose derivatives including hydroxypropylmethylcellulose and cellulose nanofibrils are suitable additives using the method according with the present invention.

[0047] Referring to Fig. 1, the method of the present invention is illustrated, wherein in the initial fibres preparation step, stock natural organic fibres, such as cellulose fibres are being defibrated using already known methods. The obtained defibrated natural organic fibres are suspended in water to obtain an aqueous solution.

[0048] Next, aggregation and foaming of the natural organic fibres in aqueous solution is performed. There are many methods supporting the foam creation during this phase. It could be done by injecting a gas through nozzles, shaking/ultrasounds, mechanical mixing or increasing the gas saturation by increasing the pressure in the mixing chamber (generating overpressure relative to the forming process pressure). At this step, additional additives can be added.

[0049] Next, mould filling is performed and foamed natural organic fibres are placed in a mould of arbitrary size and shape. Three-dimensional mould is used to control the of shape the fibre network product as well as to limit the foamed material expansion during the fast thermodynamical process (scaling of production speed). The

mould has a plurality of pores to allow for evaporation of steam and gases during forming. The number of pores and its size allows for control of the density gradient and other physical characteristics of the fibre network product obtained in the method according to the present invention. Pores can be small, but also can have a form of missed walls or parts of the walls of the mould. The mould is made of material, having a softening point above 100°C. In one embodiment the mould is at least partially made of a dielectric material selected from PVC, PVL, silicon, PTFE, PTFE GF30, PP-H, PEEK, ceramics, or combination thereof.

[0050] Next, EM material forming is performed as depicted in Fig. 1. During this step electromagnetic energy is provided to the foamed natural organic fibres in the mould. Kind of electromagnetic energy in terms of frequency and power density is adapted to desired properties of the final fibre network product prepared.

[0051] In the EM material forming step, some of the foam bubbles are degraded, however most bubbles grow as a result of an increase in the volume of gases with increasing temperature. When the process reaches the boiling point, the degradation of the old bubbles does not matter anymore, as new ones are intensely formed in the entire volume of the mold. They do not allow the fibre network to collapse until it is rigid enough to maintain a stable structure.

[0052] In one embodiment, electromagnetic energy having a frequency in a range of 10 - 100 MHz is provided to the foamed natural organic fibres. This frequency range is preferable for implementation of electromagnetic energy delivery device in a form of parallel plate capacitor or almost parallel plate capacitor. Relation of wavelength to the device size allows for such implementation. Such implementation allows for automation of the material forming in continuous process, while the material is moving along parallel plates. The capacitor is popular implementation in the industry around 27 - 35 MHz frequency range. Another advantage of this frequency range is that electromagnetic energy can be better dissipated in losses in natural organic fibrous material and polymer additives.

[0053] In another embodiment, electromagnetic energy having a frequency in a range of 300 MHz - 10 GHz is provided to the foamed natural organic fibres. This frequency range is preferable for implementation of electromagnetic energy delivery device in a form of a resonator, usually built as a closed cavity or a tunnel with the resonance inside the tunnel. Another advantage of this frequency range is that electromagnetic energy can be better dissipated in water, especially at 2.4 GHz resonance of water particles. The tunnel resonances are popular implementation in the industry around 900 MHz frequency.

[0054] Preferably, in the method according to the invention electromagnetic energy is provided uniformly. This can be achieved by a combination of uniform electromagnetic field generation technique and physical

movement (longitudinal or rotations) of the mould within the semi-uniform electromagnetic field.

[0055] Preferably, during the foamed natural organic fibres is subjected to electromagnetic energy, a ventilation system is used, allowing for removal of moisture from the space surrounding the form. The efficiency of the ventilation increases for shorter forming times (higher powers of electromagnetic energy can be applied). The delivery of warm air can further optimize the forming process in combination with the delivery of electromagnetic energy.

[0056] When bonds between fibres start to form and the foam starts to disappear (stable shape of the material begins), mould may be optionally unpacked and further drying of the obtained fibre network product can be done by applying a flow of dry air and conventional heating (with or without applying of electromagnetic energy). This optional step is marked as auxiliary drying on Fig. 1.

[0057] Referring to Fig. 2, the shape of a closed mould according to an embodiment is illustrated. Three of the four walls are flat and perpendicular to each other, the fourth is spherical. All of the outer surfaces have different normal vectors. On the two flat walls and the spherical one there are pores in a form of round holes drilled through the walls. Diameters of those holes and distribution density are varied.

[0058] In Fig. 3, the interior of a mould from Fig. 2 is illustrated for better understanding of the invention. The walls of the mould limit and determine the shape of the formed material. They are themselves impermeable to water vapor, but thanks to the holes, water vapor escapes through three of the four walls of the mould.

[0059] Fig. 4 shows a cross section of the mould according to an embodiment (the same as shown in Fig. 3 and Fig. 4) with an indication of pressure gradients depending on mould shape and pore placements. During the EM forming process, the temperature of the foamy material inside the mould increases simultaneously throughout the entire volume of the mould. It is because EM energy is accumulated over the entire volume of foamy material, i.e. by all the mass contained in the mould. When the temperature reaches the boiling point of water, an intense process of water vapor formation begins, the more intense the higher the power density used in the process. This creates a pressure build-up that seeks to escape through the pores in the mould walls. The lines of the steam flow currents are shaped by pressure gradients, and those in the area of the pores coincide with the vectors normal to the wall surfaces in these places. Those lines of steam flow generate pressure on fibres, which are wet in the first phase of the process and susceptible to displacement and crushing. That is why the density of the final fibre network product may be non-uniform. Moreover, we can distinguish many directions along which the density increases. These density gradients coincide with the pressure gradients shown in Fig. 4.

[0060] It is worth noting, the greater the local total open area of the mould, the smaller the pressure gradients it

generates. The steam leakage rate, however, also depends on the size of individual pores, their shape, and the density of their distribution. As illustrated in Fig. 4, more steam will flow through the area of the pores on the spherical wall at the same time than through the remaining open areas of the mould. This is due to the much larger diameter of the pores on the spherical wall and a relatively large number of them. However, the density distribution in the product obtained from such mould has a smaller material density gradient from the spherical side, but it remains much more even over a large area - similar to the pressure gradient distribution shown in Fig. 4. Fragments of a final fibre network product obtained according to an embodiment, which was located adjacent to the flat walls in the regions corresponding to the pores in the mould have a greater density of the material, the fibre network product is strengthened, but only in a small area covered by the "action" of the mould pores. It is significant that from the side of the third flat wall, which is adjacent to the solid wall (without pores) of the mould, it is more difficult to distinguish a clear differentiation of density, the density gradient is absent, and the obtained fibre network product is softer.

[0061] In the state of the art, pores in bottom part of the mould could normally serve as drainage holes for water excess removal by gravitation or by additional application of vacuum. However, such process usually leads to some degradation of the foam. In presented forming method, the draining step is not used and excess of water is forced out of the mould by application of electromagnetic energy at a level which causes water boiling inside the mould.

[0062] In one embodiment, parts of the mould are composed of metal parts of the electromagnetic field delivery device. For example, parallel metal plates of a capacitor can also serve as upper or lower walls of the mould allowing to form larger sheets of material.

[0063] In another embodiment, parts of the mould or electromagnetic field delivery device have movable elements which allow automatizing the manufacturing process of material filling into the mould, travelling through the mould or removing it out of the mould after the formation of the final fibre network product.

[0064] According to the second aspect of the invention a three-dimensional biodegradable fibre network product is provided, wherein the fibre network product is prepared from foamed natural organic fibres using electromagnetic energy, wherein the fibre network product has a density of 8 - 150 kg/m³ and total porosity of more than 90%.

[0065] In one embodiment, the product has a density of 8 - 90 kg/m³, preferably a density of 8 - 70 kg/m³, more preferably a density of 8 - 50 kg/m³, the most preferably a density of 8 - 30 kg/m³.

[0066] The three-dimensional biodegradable fibre network product according to the present invention can be further characterized by one or more of the following features:

- Local inhomogeneity of material density - the density gradient can be controlled by power input and mould geometry;
- High stiffness relation to the weight by increasing the density in the outer layer of the structure, effectively forming shell;
- High porosity - the structure is bone-like and is made of intertwined and entangled fibres, providing a solid mechanical support, with a large share of free space for potential implementation of other substances;
- Shape memory - the structure behaves very resiliently in a wide range of deformations (e.g., a wet blanket with dimensions $h = 4$ cm, $d = 4$ cm, when compressed to $h = 70\%$, returns to approx. $h = 96\%$);
- High dimensional stability under the influence of moisture - after saturation with water, change of linear dimensions preferably does not exceed approx. 1%;
- fully biodegradable.

[0067] The physical properties of the structure according to the invention can be determined by the method described by the Research Station in Naaldwijk, Netherlands (Wever '2002). Used standards: PN-EN 13039 - determination of organic matter content, PN-EN 13041 - determination of total porosity, volume density, shrinkage, water and air capacity at a water potential of -10 cm H_2O .

[0068] In one embodiment, there is provided a fibre network product prepared by the method of the present invention having the following characteristics:

Density: 65-75 [kg/m³];

pH 6.3-6.8;

EC (electrical conductivity) 0.07-0.10 [mS/cm];

General porosity of more than 95%;

The volume of water at the water potential of -10 cm of more than 45%;

The volume of air at the water potential of -10 cm of more than 48%;

[0069] Fig. 5 shows water retention curve for the fibre network product according to an embodiment of the present invention. The X-axis represents a potential from 0 to -10 cm H_2O , the Y-axis represents water volume (vol %). The fibre network product according to the embodiment of the present invention is characterized by high water and air capacity of more than 45%, which favours the growth of young plants such as seedlings. The tested fibre network product in a form of cubes also have an appropriate pH of 6 - 7 and are characterized by a very low EC, which greatly facilitates the selection of optimal fertilization. In an embodiment, the preferred density of

the fibre network product is about 70 kg/m³ (in the range of 65 - 75 kg/m³). The fibre network product with such a density has the most advantageous air-water properties, similar to those of mineral wool.

[0070] Fig. 6 illustrates different embodiments a), b), c) and d) of a mould integration with electromagnetic field delivery device, cross-sectional view:

a) EM device is a mould at the same time and material fills fully the EM cavity; this version is suitable for implementation as open-closed mould

b) mould is made separately of EM cavity and is inserted inside it,

c) mould is introduced between parallel plates of EM capacitor or into a resonant tunnel,

d) mould is missing upper and lower walls which are replaced by EM device parts; this embodiment may also not have vertical mould walls for continuous bulk material production captured between parallel plates or through the EM tunnel

[0071] The three-dimensional biodegradable fibre network product of the present invention can be preferably used as a plant growth substrate, filtration medium, filling and/or acoustic and mechanical damping structure.

[0072] The embodiments and examples of the present invention are to be regarded in all respects as merely illustrative and not restrictive. Therefore, the present invention may be embodied in other specific forms without deviating from its essence and the present invention, which is to be limited only by the scope of the claims.

Examples

Example 1

[0073] The product prepared according to example 1 is illustrated in Fig. 7.

[0074] The product was prepared according to the following steps:

1. Wet defibrated paper cellulose with average fibre dimensions 3 mm in length and 0.01 mm in diameter was suspended in water, reaching a cellulose concentration of 12%.

2. Pure coco glucoside was used as a foaming agent.

3. As the binder 1, increasing the stiffness of the finished product, a 10% aqueous solution of corn starch, prepared by dissolving the starch in boiling water, was used.

4. Sodium carbonate was used as the blowing agent.

5. 170 g of 12% cellulose, 0.8 g of coco glucoside, 20 g of sodium carbonate and 120 g of 10% starch were combined in a vessel.

6. The mixture was foamed on a high-speed mixer to obtain a foam with a density of 600 g/dm³. 5

7. 10 g of foam was placed in a Teflon cuboid mould with dimensions of 5 cm x 6.5 cm x 8 cm and 1.5 mm ventilation holes uniformly distributed on entire surface, with density 4 holes/cm². Foam in mould was then placed in 2.4 GHz, 1850 W electromagnetic field, for 8 minutes. 10

8. After 8 minutes, the product was removed from the mould. 15

9. Finished product weighted 5.7 g.

[0075] The method presented in example 1 makes it possible to obtain structures with high mechanical strength and high impact strength in relation to their mass. 20

Example 2 25

[0076] The product prepared according to example 2 is illustrated in Fig. 8.

[0077] The product was prepared according to the following steps: 30

1. Wet defibrated papermaking cellulose with average fibre dimensions of 3 mm in length and 0.01 mm in diameter was adjusted to a concentration of 12%. 35

2. Hemp fibres of average dimensions 15 mm in length and 0.1 mm in diameter, were sterilized for 60 minutes in a boiling solution of 1% hydrogen peroxide, dried to 5% moisture, and then dry defibrated in a high-speed laboratory mill. 40

3. A 1% agar solution in demineralized water was used as binder 1.

4. As binder 2, a 1.5% chitosan solution in 1% acetic acid was used. 45

5. Pure coco glucoside was used as a foaming agent.

6. 170 g of 12% cellulose, 7 g of hemp fibres, 0.4 g of coco glucoside, 150 g of 0.5% agar solution, 10 g of 1.5% chitosan solution in 1.5 acetic acid were combined in a vessel. 50

7. The mixture was foamed on a high-speed mixer to obtain a foam with a density of 600 g/dm³. 55

8. 50 g of the foam was placed in a Teflon cuboid

with dimensions of 3 cm x 6 cm x 10 cm and 1.6 mm ventilation holes uniformly distributed on entire surface, with density 5 holes/cm². Foam in mould was then placed in a 2.4 GHz, 1850 W electromagnetic field for 5 minutes.

9. Finished product weighted 8.7 g.

10. The highest density gradient is at the outer walls of the product and reaches 15 kg/m³ on each 1 mm towards outside direction. The method in example 2 allows to obtain a material with higher flexibility and is characterized by high acoustic insulation.

Example 3

[0078] The product prepared according to example 3 is illustrated in Fig. 9.

[0079] The product was prepared according to the following steps:

1. Pulp of wood with an average length of 3 mm and an average thickness of 0.1 mm was suspended in water to obtain a concentration of 12%.

2. Linen fibres 15 mm in length and 0.1 mm in diameter were dry defibrated in a high-speed laboratory mill.

3. As a binder, a 1% agar solution in demineralized water was used.

4. 100% pure coco glucoside was used as foaming agent.

5. 175 g of 12% wood pulp, 14 g of flax fibres, 0.8 g of coco glucoside, 150 g of 1% agar solution were combined in a vessel.

6. The mixture was foamed on a high-speed mixer to obtain a foam with a density of 600 g/dm³.

7. 40 g of the foam was placed in a Teflon multi-form in 4 cylindrical moulds having internal dimensions: length = 4 cm, height = 4 cm and 2 mm ventilation holes uniformly distributed on entire surface, with density 7 holes/cm². Foam in mould was then placed in 27 MHz, 1 kW electromagnetic field for 4 minutes.

8. Finished product weighted 3.8 g.

[0080] The method provided in example 3 allows to obtain a material with good water absorption and favourable air-water relation for plant growth.

Claims

1. A method for the preparation of three-dimensional biodegradable fibre network product, the method comprising:

foaming natural organic fibres in aqueous solution,
mould filling with the foamed natural organic fibres, wherein the mould has a plurality of pores, forming a three-dimensional biodegradable fibre network product by providing electromagnetic energy to the foamed natural organic fibres,

wherein said plurality of pores is adapted to evacuate water and steam generated by providing electromagnetic energy to the foamed natural organic fibres.

2. The method according to claim 1, wherein the content of natural organic fibres in three-dimensional fibre network product is at least 95 % on a dry basis.
3. The method according to any preceding claim, wherein the length of natural organic fibres in foamed natural organic fibres is from 0.1 cm to 3.0 cm.
4. The method according to any preceding claim, wherein natural organic fibres are selected from cellulose fibres, ligno-cellulose fibres or combination thereof.
5. The method according to any preceding claim, wherein foaming natural organic fibres in aqueous solution is performed by introducing a gas into the pulp.
6. The method according to any preceding claim, wherein aqueous solution used for foaming natural organic fibres further comprises adding at least one biodegradable non-fibrous additive selected from a foam stabilizer, surfactant, biodegradable blowing agent or combination thereof.
7. The method according to any preceding claim, wherein aqueous solution used for foaming natural organic fibres further comprises adding at least one further additive for controlling biomechanical properties of the fibre network product, wherein said further additive is selected from a polysaccharide, polysaccharide derivative, lignin, lignin derivative, cellulose, and a cellulose derivative.
8. The method according to any preceding claim, wherein the mould is made of dielectric material, having a softening point above 100°C.

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9. The method according to any preceding claim, wherein density gradient of the three-dimensional biodegradable fibre network product is controlled by the arrangement of the plurality of pores in a mould and by the kind and/or power density of electromagnetic energy provided to the foamed natural organic fibres.

10. The method according to any of claims 1 - 9, wherein electromagnetic energy used for forming a three-dimensional biodegradable fibre network product has a frequency in a range of 10 - 100 MHz.

11. The method according to any of claims 1 - 9, wherein electromagnetic energy used for forming a three-dimensional biodegradable fibre network product has a frequency in a range of 300 MHz - 10 GHz.

12. A three-dimensional biodegradable fibre network product, wherein the fibre network product is prepared from foamed natural organic fibres using electromagnetic energy, wherein the fibre network has a density of 8 - 150 kg/m³ and total porosity of more than 90%.

13. The three-dimensional biodegradable fibre network product according to claim 12, wherein the fibre network product is prepared using the method according to claim 1.

14. The three-dimensional biodegradable fibre network product according to claim 12 or 13, wherein the product has a density of 65 - 75 kg/m³ and total porosity of more than 95% and pH of 6 - 7 and volume of both water and air at the water potential of -10 cm of more than 45%.

15. Use of a three-dimensional biodegradable fibre network product according to any of the claims 12 - 14 as a plant growth substrate, filtration medium, filling and/or acoustic and mechanical damping structure, thermal isolator, efficient moisture absorber and evaporator.

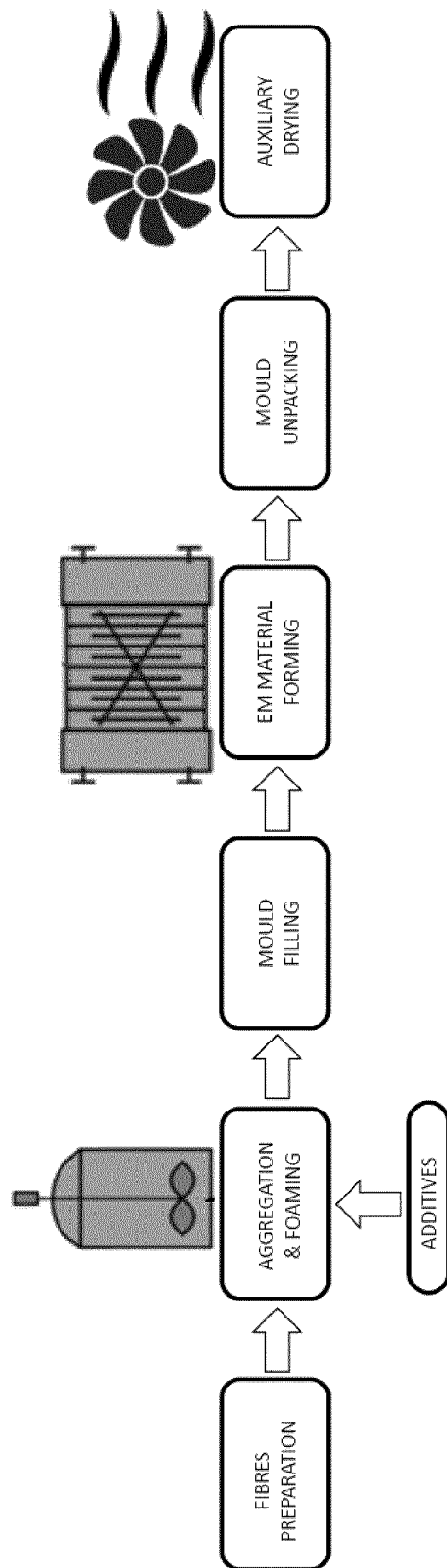


Fig. 1

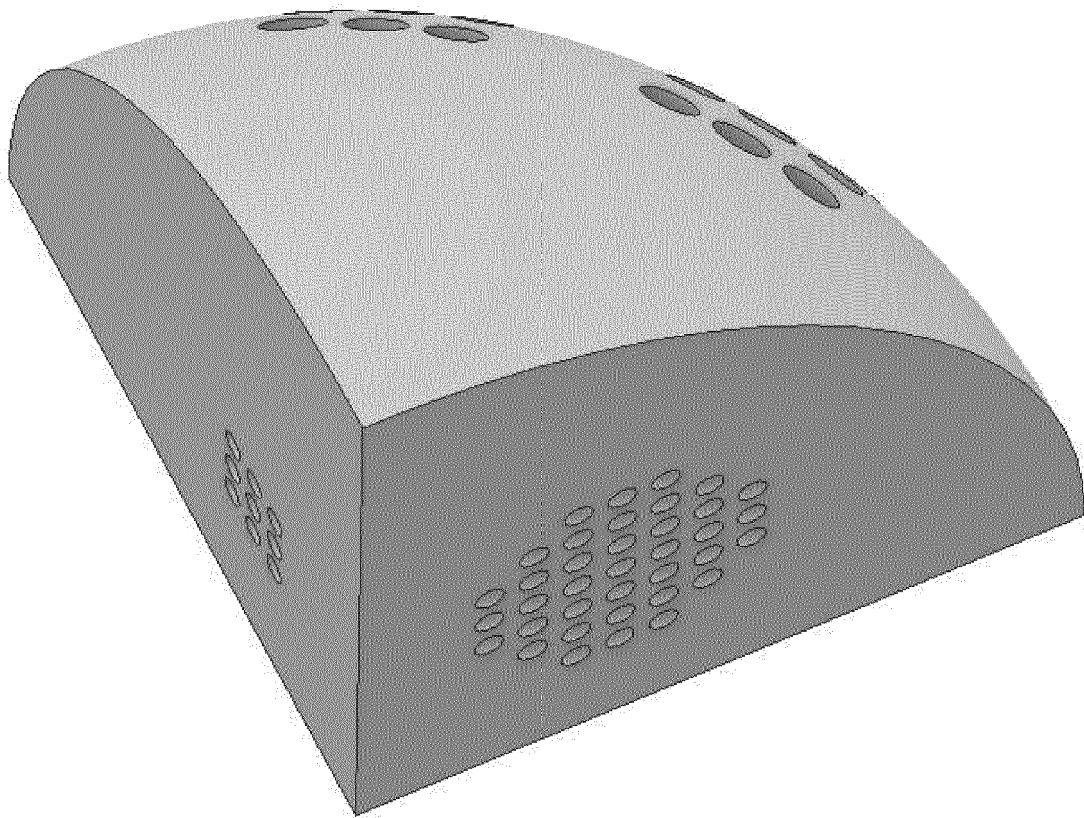


Fig. 2

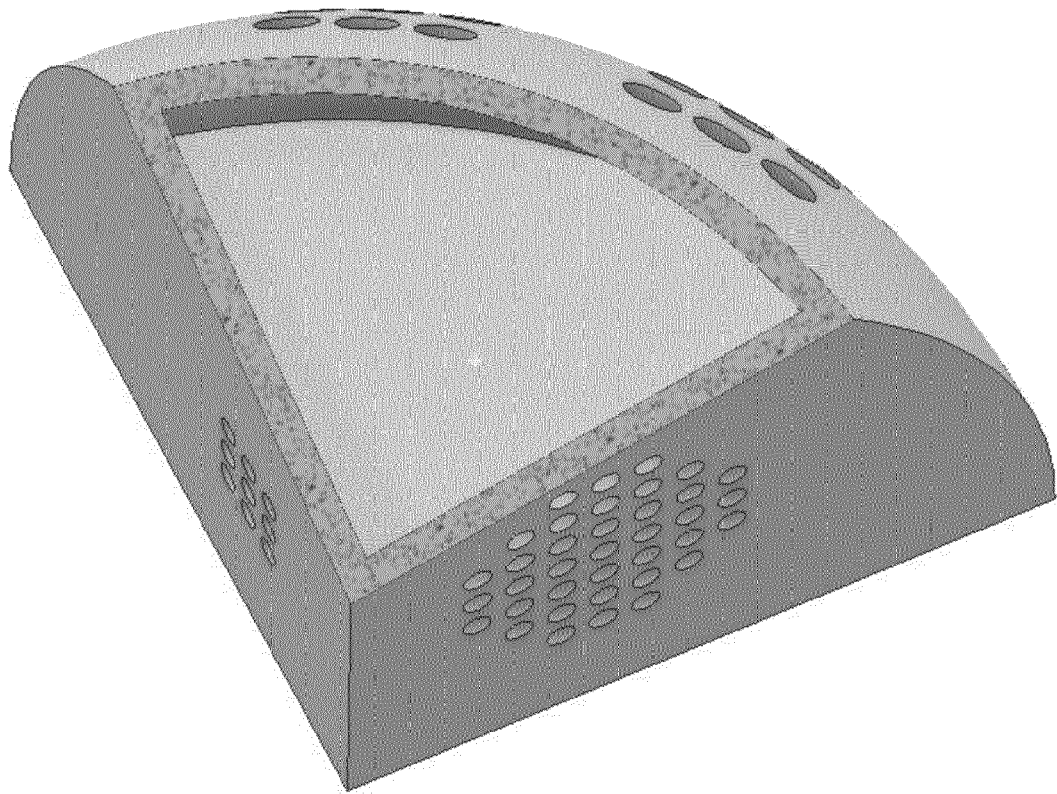


Fig. 3

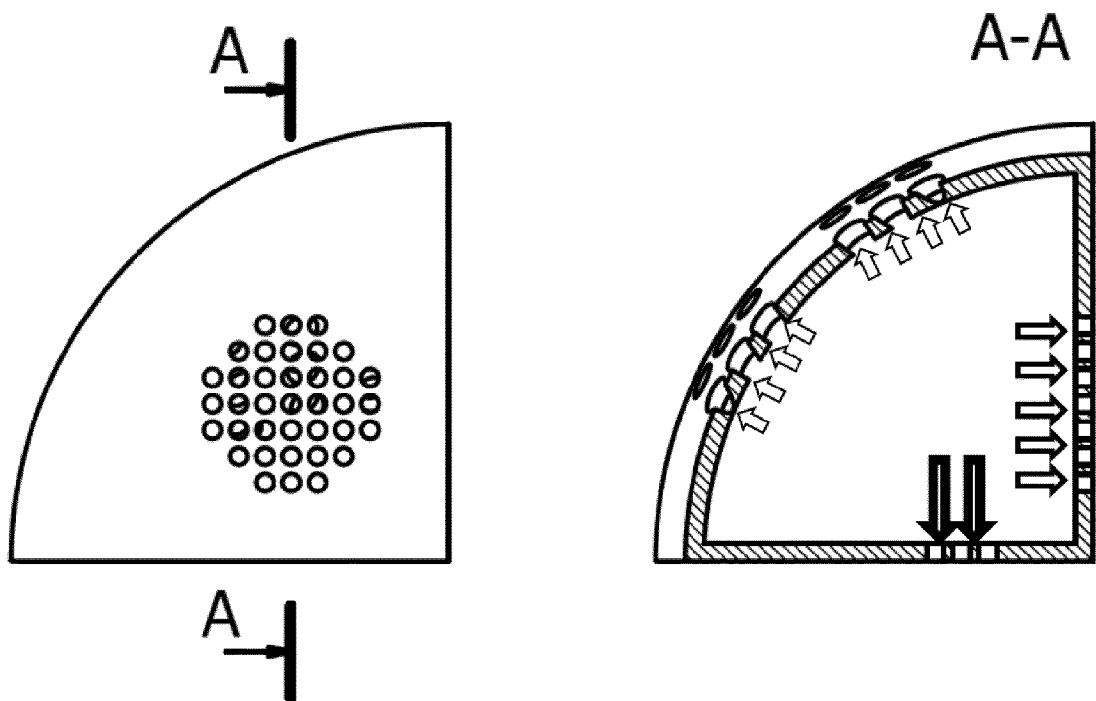


Fig. 4

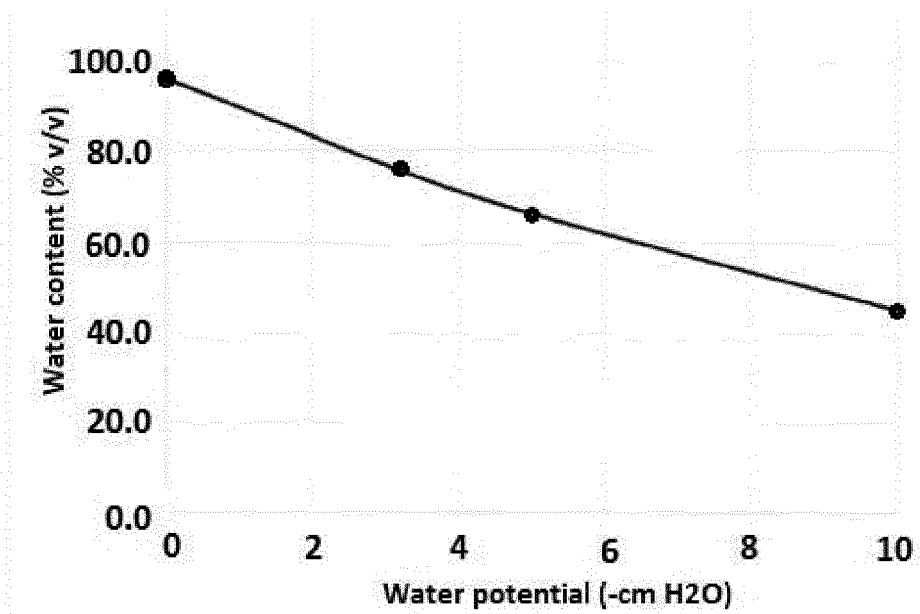


Chart 1 - Water retention in relation to water potential

Fig. 5

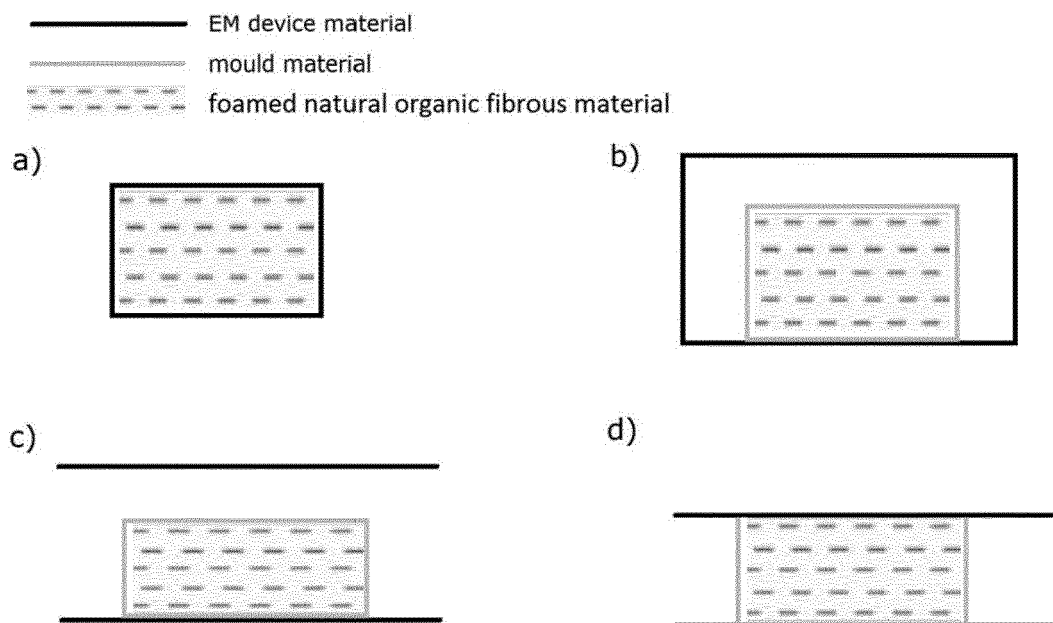


Fig. 6

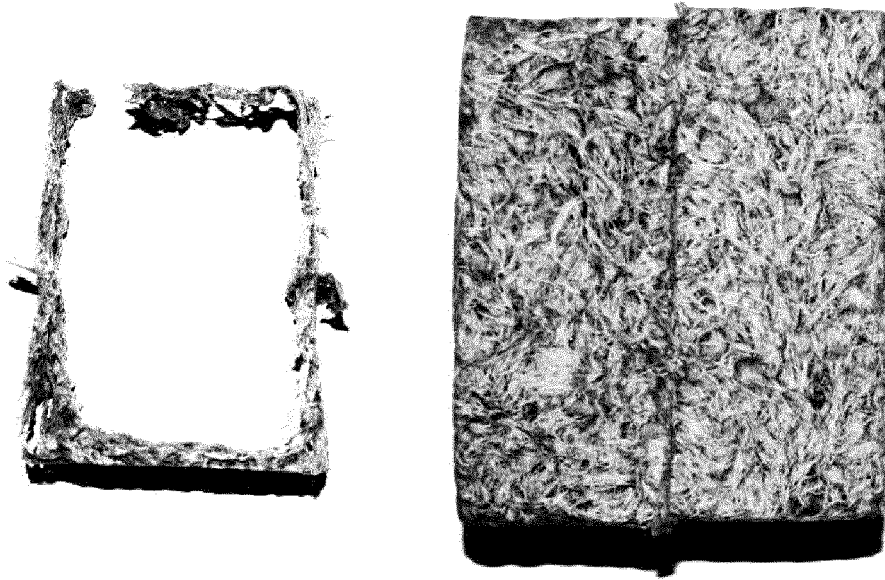


Fig. 7



Fig. 8

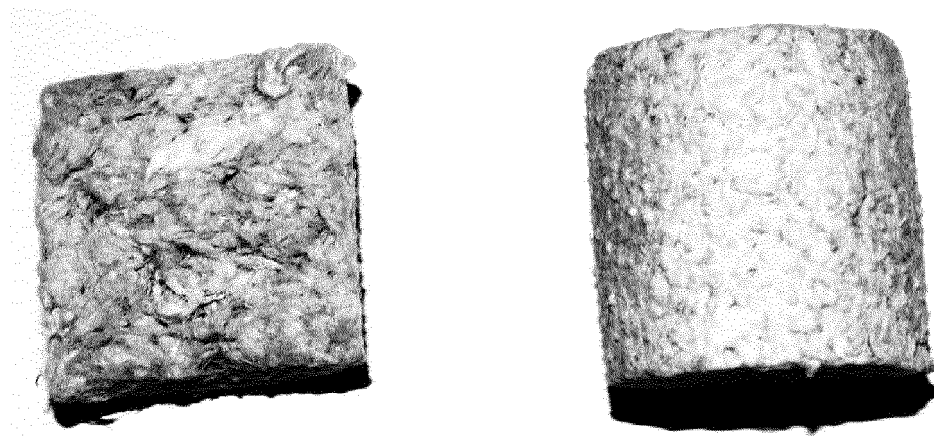


Fig. 9



EUROPEAN SEARCH REPORT

Application Number
EP 21 15 4700

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			TECHNICAL FIELDS SEARCHED (IPC)
			D21J
The present search report has been drawn up for all claims			
Place of search Munich		Date of completion of the search 22 June 2021	Examiner Pregetter, Mario
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