



- (51) **International Patent Classification:** Not classified
- (21) **International Application Number:**
PCT/FI2012/050582
- (22) **International Filing Date:**
11 June 2012 (11.06.2012)
- (25) **Filing Language:** English
- (26) **Publication Language:** English
- (30) **Priority Data:**
20115570 9 June 2011 (09.06.2011) FI
- (71) **Applicant** (for all designated States except US):
EKOLITE OY [FI/FI]; Tykistökatu 4D, FI-20520 Turku (FI).
- (72) **Inventors; and**
- (75) **Inventors/Applicants** (for US only): **KESOLA, Ilkka** [FI/FI]; c/o Ekolite Oy, Tykistökatu 4D, FI-20520 Turku (FI). **ROMMI, Vesa** [FI/FI]; c/o Ekolite Oy, Tykistökatu 4D, FI-20520 Turku (FI). **TALLING, Bob** [FI/FI]; c/o Ekolite Oy, Tykistökatu 4D, FI-20520 Turku (FI).
- (74) **Agent:** **IPR PARTNERS OY**; Bulevardi 2-4, FI-00120 Helsinki (FI).
- (81) **Designated States** (unless otherwise indicated, for every kind of national protection available): AE, AG, AL, AM,

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

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

Declarations under Rule 4.17:

— of inventorship (Rule 4.17(iv))

Published:

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

(54) **Title:** PROCESS FOR A NATURAL FIBER COMPOSITE MATERIAL MANUFACTURING, PRODUCTS OBTAINED AND METHODS OF APPLICATION THEREOF

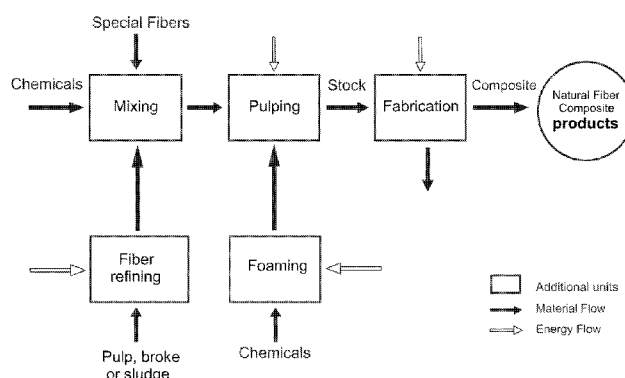


Figure 1

(57) **Abstract:** A versatile method for the manufacturing of a natural fiber composite is provided, and a product thereof. The method, in accordance to the preferred embodiment, comprises crush-or impact grinding of fibrous feedstock, followed by classifying, mixing with diluting and/or bonding agent, optional foaming and shaping of the end product. A composite product manufactured by said method is provided. Treatment of fibrous feedstock by torrefaction may be utilized prior to crush-grinding in order to provide manufactured composites with novel properties. Natural fiber composite materials thus produced retain heat-, sound-and/or fire- insulating properties and may be utilized in different industries from automotive to decorative. The method is employed to re- utilize waste, recycled and side products of various industries, including forest, chemical-, mineral-and agro-industries.



PROCESS FOR A NATURAL FIBER COMPOSITE MATERIAL MANUFACTURING, PRODUCTS OBTAINED AND METHODS OF APPLICATION THEREOF

5

FIELD OF THE INVENTION

The present invention relates to fiber composite-, preferably natural fiber composite- and natural fiber insulate materials, comprising fibrous feedstock, bonding agent and additives and allowing a multilayered structure; and methods of manufacture- and application thereof.

BACKGROUND

Term "fiber" accounts for fine, single, continuous filament. Fibrous materials are generally classified to natural and synthetic. Traditionally, the term "natural fiber" or "biofiber" relates to the fibrous material derived from the tree or plant source. Natural fibers, embedded in a natural or synthetic polymeric matrix, are known as natural fiber composites, and have gained recent interest because of global availability, low material and production costs, as well as for possessing mechanical strength, and good thermal and acoustic insulating properties.

Nowadays market for natural fiber reinforced composites is growing as well as worldwide consumers become oriented to more ecological but still low-cost alternatives of end user goods. Following same ecological concerns recycling and reuse of waste materials continue to gain its importance. For this reason, new methods employing reutilization of synthetic fibers along with recycled natural fiber materials have to be developed. Prior art includes examples of production of natural fiber reinforced composite materials employing a reutilization step.

30

Document WO9416145 discloses a method of preparation of cellulose-based composite material by mixing together chopped recycled paper/cardboard and corn starch adhesive. The method includes pulp pre-heating step, compression step at 120-150 bars and a final drying step of few days, and is most suitable for producing products in a shape of rod, plate or equivalent. However, the absolute requirement of this method is that by no circumstances a paper/cardboard must not be wet.

35

The development of natural fiber composite materials caused a production of water impermeable composites (e.g. WO07104990, WO06056737), as well as fire retardant insulates (e.g. US2011095245, US20100146887, and EP0694094). However, the production process of above mentioned water-impermeable composites is multi-
5 step and requires thermal curing. Manufacturing processes of cellulosic fire retardant insulates imply, above all, the utilization of known chemical fire retardant additives, such borates, boric acid or the like, and may imply a heat compression step. Natural fiber composites with sound attenuating properties are known, for example, from US2011073253, wherein the production process involves the use of polyurethane resins.
10

Document US20020100565A1 discloses methods for preparing a laminated structural biocomposite panels from agro-fibers, which methods involve a compression step with elevated temperature. A sandwich stacking is realized in WO2008116466;
15 however the manufacturing process required a compression step in press mold under vacuum.

One way to reduce weight of the material and make it to achieve better thermo- and sound isolating characteristics is to produce a foamed structure. Consequently, a
20 production of foamed thermoplastics by means of chemical and physical foaming agents is well described in prior art (for ex. Throne, Thermoplastic Foam Extrusion, 2004). Also, WO2008006943 teaches how to increase the bulk of a fiber product formed of fibrous pulp. The fiber pulp is modified in two steps, first by basic carbonate component and then by acid. The bulk increases as a result of small-bubbled
25 gas formation. In WO02088233 the wood-plastic composite is disclosed, comprising wood flour and high-density polyethylene, which composite was foamed by introducing of CO₂, N₂ gases or air into wood-plastic mixture during an extrusion process.

30 Regardless of the fact, that multiple methods of natural fiber reinforced composite materials production from waste and/or recycled materials exist, it may still be advantageous to develop more efficient methods to process and functionalize recycled, as well as re-use and combine wood-, agro- and mineral fiber materials into ecological and cost-effective composite products. It may be also desirable to produce said
35 composite products of any shape or size, combining lightweight with stiffness and rigidity, with the opportunity to gain other properties in addition to above said by simple addition of reactants; produced by a straightforward process involving as least steps as possible and involving commonly utilized machines, and being equal-

ly suitable for various common methods of handling, for example, pressing, casting, blowing or spraying.

SUMMARY OF THE INVENTION

5

The objective of the invention is to at least partly alleviate above mentioned problems by enabling forest-, agro-, mineral, chemical and environmental industries to convert cross-industrial side-flows to produce new functional natural fiber composites and natural fiber insulates, including multilayered structures, possessing, depending on composition and processing method, such features as fire-resistance, hydrophobicity, light weight, durability, as well as sound and heat insulation properties.

15

The objective of the invention is achieved by various embodiments of a process for manufacture a natural fiber composite material and of a composite product obtained therefrom.

20

In one aspect of the invention a process for the manufacturing of a natural fiber composite material is provided.

25

In accordance with the preferred embodiment the process for the manufacturing of a natural fiber composite material involves a crush- or impact grinding of fibrous feedstock, followed by classifying, mixing with diluting and/or bonding agent, optional foaming and the fabrication of the end product; and a resulted composite product thereof. Fibrous feedstock may include wood-, agro- and optionally mineral fibers. Foaming is accomplished mechanically; however, the use of either chemical or physical foaming agents is not excluded. For the purposes of the invention, the term "foamed material" refers to a material having a plurality of distinct void spaces formed therein, and the term "foaming" refers to either introducing physical or chemical foaming agent into a defibered feedstock mass which promotes formation of distinct void spaces therein, or promoting the formation of said void spaces mechanically.

35

In one embodiment of the invention a dry process for the manufacturing of natural fiber insulates is provided, and a fibrous insulation product thereof, wherein the solid content of a fibrous feedstock is >90%. In accordance with said embodiment a fibrous feedstock may include wood- and/or agro-fibers. In accordance with said embodiment, the product may be in the form of panels, which rigidity may vary from

soft to stiff depending on additives, bonding agent and fabrication method. In accordance with said embodiment a blowable fibrous insulate may be manufactured. Insulation products, obtained in accordance with said embodiment are particularly suitable for sound and heat insulation purposes.

5

In further embodiment of the invention a process for the manufacturing of a natural fiber composite material is provided, which process utilizes, along wood- and agro-fibers also mineral fibrous feedstock. In accordance with said embodiment, a "wet" process is provided, and a composite product thereof, wherein the solid content of fibrous feedstock may either be 15-50% and the process will be referred to as a "high consistency" or the solid content of fibrous feedstock may be 2-15%, and the process will be referred to as a "medium consistency", respectively. For the purposes of the invention the term "consistency" refers to a solid content of fibrous feedstock during processing. Composite products, obtained thereof, are particularly suitable for heat insulation purposes. In accordance with said embodiment a dry process is provided and a composite product thereof, wherein said product is particularly suitable for fire retardation purposes.

In yet further embodiment of the invention a process for the manufacturing of multi-layered (sandwich) structures is provided. The sandwich structure consists of two hard faces with a middle filling layer in between. Said middle filling layer for such "sandwich" comprises a natural fiber reinforced composite material prepared according to the process of a preferred embodiment of the invention. The material of said middle filling layer may be secured to the inner walls of "sandwich" faces by means of special adhesive.

In yet further embodiment of the invention a process for manufacturing of natural fiber composite materials is provided, said process involves treatment of an unprocessed fibrous feedstock known as torrefaction.

30

In another aspect of the invention a composite material, obtained by the above described processes, is provided.

The following advantages may be achieved by utilizing manufacturing methods of various embodiments of the present invention in comparison to those described in prior art.

35

The method is flexible and enables manufacturing of various products by utilizing same equipment. The method may be easily and cost-effectively integrated into existing production lines of wood- and paper industries, for example, since the method may be adapted to utilize side products from above-mentioned industrial lines.

5

The method is employed to re-utilize waste, recycled and side products of different industries, including forest, chemical-, mineral- and agro- industries. For example, every year only in Finland 3 million tons of bark is produced as a waste product of forest industry, that bark is simply burned; or 2 million tons of clean waste paper, that is been recycled by so called wet process consuming a lot of electricity and water. Fibrous feedstock material utilized herein may be wood- and agro-fibers, such as, for example, recycled paper, carton boards and newsprints, waste wood, bark, sawdust, mechanical pulp fibers or plant fibers. The invention, according to its embodiment, includes also utilization of mineral wool fibers, such as, for example, a stone wool, obtained, as clean, chemically unmodified waste directly from the factory, and converting it into high-value composite material. Also biogas, acting as physical blowing agent, according to an embodiment of the invention, can be obtained directly from landfill, and refinement of biofiber-based material, by biogas directly on the landfill side may save costs of the end product and contribute to ecological situation. Thus, integrating a composite production to wood-, bioenergy or waste-processing industries may be beneficial from ecological and economical point of views.

The composite material manufactured in accordance with the invention may be easily adapted for different ways of handling and shaping, such as air-blowing, casting, extrusion, web formation and building laminated structures. The composite material, dependent on the production method thereof, possesses at least one property selected from such features as resistance to fire, heat- and/or sound isolation, hydrophobicity, fluid absorption capability, durability, light weight, improved strength-to-weight ratio and biological stability.

BRIEF DESCRIPTION OF THE DRAWINGS

The invention and examples are next described in more detail with reference to enclosed drawings, in which:

Figure 1 illustrates a generalized diagram for an exemplary process of fiber-reinforced composite material manufacturing according to the invention;

Figure 2 illustrates an exemplary process for manufacturing of thin and stiff fiber reinforced composite materials;

- 5 Figure 3 illustrates an exemplary dry process for manufacturing of relatively soft natural fiber composite materials and blowable sound insulates;

Figure 4 illustrates an exemplary dry process for manufacturing of fire retardant composite material; and

10

Fig.5 illustrates a general approach for manufacturing of composite materials and products by means of torrefaction.

DETAILED DESCRIPTION OF THE EMBODIMENTS

15

Some embodiments of the invention will now be described in more detail.

The process for the manufacturing of composite material according to the preferred embodiment of the invention involves the step of defibering of feedstock material, which step may preferably be implemented by crush- or impact-grinding of fibrous feedstock, followed by classifying.

20

So called crush-grinding or impact grinding is an essential stage of the composite material manufacturing process, according to the preferred embodiment of the invention, and specifies the difference of said process in comparison with that of prior art. Crush-grinding is realized on the basis of disintegrator apparatus DESI-15/16C (Desintegraator Tootmise OÜ, Estonia), with the frequency of rotor rotation 72 + 25-72 Hz. Said apparatus may also be used for the preparation of suspension and emulsions. A pre-crusher may optionally be utilized. Disintegrators DESI are adapted to perform grinding of starting material by several, usually 3-7 impacts at high speed up to 100-200 m/s. The speed is increased as the particles move from the center of the rotor to the periphery. The central part of the disintegrator comprises two rotors rotating in opposite directions, which rotors are provided with several concentrically placed rows of impact elements. When fibrous feedstock is disintegrated by said apparatus, grinded feedstock particle's surface tends to be more active in regards to chemical bonds formation so, that upon crush grinding of initial fibrous feedstock comprising several components, as for example bark, waste paper and mineral wool, said components start to react with each other already at grinding

25

30

35

stage, or right after the grinding stage. This way, new bonds between feedstock reactants are created. The grading and the microscopic texture of crush-grinded by DESI apparatus particle's surface is different, comparing to that of particles, grinded by common hammer mills. This difference in surface structure was experimentally observed by comparison of disintegrated into powder wood feedstock and grinded by conventional methods wood feedstock while testing both powders as oil filter materials. Wood powder processed by disintegration with particle surface activation demonstrated superior filtering properties in terms of providing non-viscous, almost water-like liquid in comparison to dark liquid of high viscosity filtered through wood powder obtained by conventional grinding.

During above mentioned disintegration process feedstock particles collide with one another, thus promoting the process of surface activation. Particle collision speed that may be achieved in the above mentioned disintegrator is times higher than that provided by conventional methods. While a highest particle collision speed that may be achieved by conventional grinding apparatuses, such as mortar grinder and ball mill, is far below 100 m/s, in disintegrator DESI particles may be collided at a speed of 100-350 m/s. Therefore above mentioned grinding method is largely based on feedstock particle's collision rates. Fibrous feedstock crush-grinded particles with the activated surface possess linearly enhanced reaction rates while interacting with the bonding agent. Enhanced reactivity may be detected already for larger feedstock particles. Another advantage of above mentioned disintegration method is reduced agglomeration rate, while agglomeration is a common aftereffect of grinding by conventional methods.

An important aspect to be taken into account while processing various fibrous feedstocks by above mentioned disintegration process is particle collision speed adjustment that may be determined only experimentally for individual materials.

Alternative equipment may include solutions from e.g. Megatrex Ltd, Atritor ltd, Theisen GmbH. For those skilled in art it is clear, that any equipment with similar operation mode may be suitable for the purposes of the invention.

Fibrous feedstock, in accordance with said embodiment, may include wood-, agro- and/or mineral fibers, wherein natural fiber component may consist of recycled paper, carton boards, newsprints or clean paper waste, bark and sawdust, mechanical pulp fibers and plant fibers. Mineral fiber component herein consists of clean, chemically unmodified stone wool.

Crush-grinding line preferably includes a classifier, utilized, for example, for separating bark into outer and inner layers, according to some of the embodiments of the invention.

5

A diluting agent, preferably water, and/or a bonding agent are admixed to the crush-grinded fibrous feedstock. Resulted composite blend may optionally undergo a foaming step, implemented by means of mechanical frothing or by introducing a physical or chemical foaming agent into a composite blend.

10

For clarity purposes the terms "crush-grinded fibrous feedstock" and "crush-grinded blend" are referred in this disclosure to fibrous feedstock that had undergone crush-grinding process in DESI disintegrator; and the term "composite blend" is referred to the mixture of crush-grinded fibrous feedstock with the bonding agent.

15

The term "foamed material" is referred in this disclosure to the material having a plurality of distinct void spaces formed therein; the term "foaming" is referred to the formation of a plurality of distinct void spaces in a pulp mass, accomplished by either introducing a foaming agent or mechanical frothing; and the term "foaming agent" is referred to a chemical or gas which promotes foaming in a composite material.

20

Chemical foaming agents may be, according to some embodiments of the invention, Sodium Lauryl Sulfate (SLS), or coconut dimethylamineoxide (Aromox MCD-W, Akzo Nobel). SLS, also known as Sodium Dodecyl Sulfate (SDS), has a general formula of $C_{12}H_{25}CO_4Na$ and is a common anionic surfactant and possess ability to create froth. Coconut dimethylamineoxide is a conditioning agent and foam booster in highly alkaline to highly acid cleaner formulations. Also, other common washing agents can be used as chemical foaming agent according to the preferred embodiment of the invention.

25

30

Physical foaming agents comprise mainly carbon dioxide (CO_2) and biogas, but are not generally limited to those and may also comprise of hydrogen (H_2), methane (CH_4), butane (C_4H_{10}) and others. Biogas of a typical landfill side comprises largely of carbon dioxide (CO_2) (30-50%) and methane (CH_4) (40-60%).

35

In accordance with the embodiments of the invention, bonding agents, utilized by the invention, may comprise, for example, water glass, also known as silicate glue,

or a two-component glue system from Casco (Melamine-Urea-Formaldehyde 1247/2526). It is not the purpose to limit the invention by these particular adhesives, in certain circumstances these may be replaced by more suitable bonding agents. Water-glass, known also as Sodium metasilicate, can be depicted with the chemical
5 formula Na_2SiO_3 (CAS Registry number 6834-92-0). In addition to the anhydrous form, there are hydrates with the formula $\text{Na}_2\text{SiO}_3 \cdot n\text{H}_2\text{O}$. Water glass has a proven property as a hardener and a concrete sealant; in addition it is also used as a fire retardant.

- 10 The use of biological bonding agents, such as lignin-based adhesives or starch is also possible, in accordance with the preferred embodiment of the invention. One of the examples for biological bonding agents includes a pine tree adhesive.

The invention, according to its preferred embodiment, implies the use of various
15 additives in order to improve final characteristics of the material. Additives may include calcium carbonate (chalk), slag, gypsum, special cements, for example Hac (High alumina Cement), hydrophobic agents, mold-growth prevention agents, for example pine tree bark, stabilization agents, EPS (styrox), biocoal, torrefied biomass.

20

Biocoal, a black, solid, dry material, produced by a process of torrefication or thermochemical treatment of biomass at high temperatures and without oxygen, according to a preferable embodiment of the invention, is utilized as an additive in order to provide sufficient stiffness to the composite product. Torrefication or torrefaction is
25 a process similar to pyrolysis, but proceeding in milder conditions, such as lower temperatures. The term "biomass" refers in this disclosure to the material of natural origin, derived for example from wood or plants, such as wood chips, bark, forest residues, willow chips, straw, grass and the like. Biocoal, when being crush-grinded by means of above mentioned disintegrator, preserves a fiber-like structure.

30

Torrefication of biomass, as disclosed above, is utilized as a subprocess for production of various composite materials and/or products in accordance with embodiment of the invention. Torrefied biomass materials, such as wood chips, for example, are experimentally shown to gain fascinating properties, such as hydrophobicity, biological stability and high fluid absorption capacity. Torrefied biomass-based composite products therefore demonstrate higher durability and lower weight as compared to corresponding prior art-related composites, as well as improved strength-to-weight ratio. Said composite products easier undergo recycling or disposing. In
35

addition, the torrefaction-based natural fiber composite production process retains lighter carbon and water footprints, as compared to corresponding state-of-art. As natural fiber material side products of wood, and/or paper industries may be utilized.

5

The process for manufacturing of fiber-reinforced composite material according to the preferred embodiment of the invention comprises the following phases:

- 10 1. Defibering of fibrous feedstock by crush- or impact-grinding as described above, at ambient temperature, utilizing disintegrator apparatus of DESI-type.
2. Introducing a diluting and/or bonding agent into defibered blend.
3. Foaming the resulted composite blend either mechanically or by means of introducing a physical or chemical foaming agent into the composite
- 15 blend.
4. Admixing additives.
5. Drying and shaping the product.

20 Figure 1 illustrates an example of the above mentioned process in accordance to one embodiment. For those skilled in art it is clear, that process phases are exemplary and represent a generalized overview of the production process without intentions to limit an invention in any way, so that in particular cases the process phases may be arranged in different order for achieving best results.

25 The last process phase comprising a final shaping of the composite product may be implemented in different ways. The following methods may be utilized to shape the product, in accordance with the embodiments of the invention:

- Casting into forms, press-casting.
- 30 - Light pressing by means of roller compactor or tape gun, optionally employing heating, taking care of not damaging air bubbles, formed during foaming step.
- Layering (lamination)
- Calendering
- 35 - Extrusion
- Injection or compression molding
- Blowing by airflow into frame
- Pelletization

The invention, in accordance with some embodiments, can be adapted for integration into fibrous feedstock treatment processes commonly described by professional terms "wet" and "dry". "Wet" processes imply the use of water during handling of fibrous feedstock, and can be classified as high-, medium- and low consistency, where the term "consistency" refers to a solid content of fibrous feedstock during processing. An invention, according to some embodiments utilizes high- and medium consistency processes for the production of natural fiber composites, wherein the solid content of fibrous feedstock is approximately 15-50% and 2-15%, correspondingly. Also process denoted as "dry" is utilized for the purposes of the invention, wherein the solid content of a fibrous feedstock is >90%. However, these numbers are approximate and may slightly vary depending on the production line setup.

The versatility of the invention, disclosed herein, and its adjustability to various production processes introduce certain difficulties for general disclosure. For this reason the embodiments will be further described by reference to the following detailed examples. The examples are not, however, to be considered as technical limitations and are provided with a purpose to teach those skilled in art to implement the invention in best possible way.

Examples 1 and 2 describe one of the embodiments of the invention, implemented by a dry process for the manufacturing of natural fiber composites, wherein said dry process utilizes a natural fibrous feedstock. Examples 3 and 4 describe another embodiment of the invention, implemented by wet and dry processes, characterized in that both processes utilize both natural and mineral fiber feedstock. Example 5 describes the manufacturing of multilayered structures. Example 6 describes treatment of fibrous feedstock by means of torrefaction as an additional phase in natural fiber composite manufacturing process.

Examples

Example 1. Dry process for the manufacturing of thin and stiff natural fiber composite materials.

5

The reference will now be made to the manufacturing of thin and stiff natural fiber composite materials according to Fig.2. According to one embodiment of the invention a dry process for manufacturing of stiff natural fiber composite materials is provided, and a natural fiber composite product thereof. The process implies utilization of wood fibrous feedstock obtained from, for example, bark, waste and recycled paper and a bonding agent. A fibrous feedstock is crush- or impact-grinded, according to the preferred embodiment of the invention. A bonding agent consists of two-components: an adhesive and a hardener, for example Melamine-Urea-Formaldehyde (MUF) system 1247/2526 from Casco.

15

The fibrous component is spread on fixed or moving line or other air-permeable base, underneath which a suction box is located. Spread components are held in place by means of suction, while two-component glue is sprayed over them.

20

Alternatively, a crush-grinded fibrous feedstock in an amount of < 60% may be admixed with thermoplastic polymers (40%), and, optionally with additives, and the resulted wood-plastic blend undergoes a shaping process by means of injection molding, extrusion or compression molding, followed by drying. A product thus obtained is stiff and, accordingly, particularly durable, therefore a process described herein may be used for the manufacturing of wear-resistant consumer goods.

25

The other component, providing additional stiffness, may be biocoal, produced by means of torrefaction of biomass, such as wood chips, crushed bark, willow chips, forest residues and the like, according to a preferred embodiment of the invention.

30

Example 2. Dry process for the manufacturing of relatively soft natural fiber composite materials and blowable sound insulates.

35

The reference will now be made to the manufacturing of relatively soft, natural fiber composite materials, preferably shaped as panels, as well as blowable insulation materials, according to Fig. 3. According to one embodiment of the invention, a dry process for manufacturing of relatively soft natural fiber composite material is pro-

vided, and a natural fiber composite product thereof. The fibrous feedstock herein is represented by wood- or agro fibers.

5 Fibrous feedstock components in an amount of <90% were crush- or impact-grinded by means of above mentioned disintegrator apparatus. A hydrophobic agent (0.1-0.2%) and, optionally, a mold-growth preventing agent may be added at this step. Herein, a mold-growth preventing agent is pine tree bark. The resulted blend may be referred as a blowable fibrous insulation material. A bonding agent (herein starch) in an amount of < 10% was introduced into the crush-grinded blend.

10

In parallel a foaming agent along with a stabilization agent (0.5%) were admixed and in turn added to the composite blend, and the resulted composite blend was mechanically vortexed.

15 The resulted composite blend undergoes shaping by means of press-casting, followed by a final drying. The end-product may be referred as a natural fiber composite material, and may be utilized as sound-insulation panels, or decorative elements with acoustic properties.

20 The natural fiber composite material obtained by the above described process may be utilized as a filling for so called noise barrier panels. The noise barrier panel may be built in the form of a lightweight honeycomb structure, for example, which structure is spray- or cast-filled by the composite material. The size of such panels is not limited. The density of the composite material may be adjusted in order to optimize
25 sound damping properties of noise barrier panels.

Example 3. Wet process for the manufacturing of thermal insulates.

30 According to another embodiment of the invention, a wet process for the manufacturing of a natural fiber containing composite material is provided, and a natural fiber containing composite product thereof. Herein, three equal parts of fibrous feedstock were utilized; these parts comprising bark, paper waste and mineral fiber. Each part was equal of 600 g. The bark was classified to inner and outer bark in the grinding line classifier, and only outer bark was used for the process. Fibrous feed-
35 stock components were crush-grinded by means of above mentioned disintegrator apparatus. A diluting agent, preferably water, in amount of 30 dl was admixed to defibered feedstock. A coconut dimethylaminoxide additive along with the diluting agent (water, 6 dl), were admixed to the resulted composite blend, and said compo-

site blend was mechanically foamed by stirring 10 min. High Alumina Cement (Hac) additive was added to the foamed composite blend followed by gentle stirring of the foamed composite blend. A cement accelerator (fondue) was added and the final composite material was introduced into the mold.

5

Thus obtained foamed composite material is characterized by its perfect thermal insulation parameters. Thermal conductivity parameter of the material (descriptive of an ability of the material to conduct heat) was measured in accordance to the standard EN ISO 8031:2009, Rubber and plastics hoses and hose assemblies - Determination of electrical resistance and conductivity. According to those measurements, a mean thermal conductivity (λ) of the said material at a mean temperature of 10 ° C is in the range of 0.03-0.1 W/mK (Watts/meter per kelvin).

10

The applications of the foamed composite material include thermal insulation for construction panels and bearing wall- and ceiling construction elements (instead of mineral and glass wool, EPS and PU); ecological insulation materials for manufacturers of refrigeration equipment and instruments for food industry (instead of PU); ecological insulation materials for the top layers of special purpose vehicles (instead of plastic composites and PU); ecological insulation materials for construction and inner furnishing of buses and special purpose vehicles (instead of plywood, steel, felt); light weight ecological inner- or outer parts for packaging industry (instead of EPS, LDPE).

15

20

Example 4. Dry process for the manufacturing of fire retardant composite materials.

25

The reference now will be made to the production of fire retardant composite material, according to Fig. 4.

According to a certain embodiment of the invention, a fire retardant composite material is provided, and a process for the manufacturing thereof. Herein, the wood- and mineral fibrous feedstock (stone wool) is utilized. Said fibrous feedstock was crush-grinded by means of above mentioned disintegrator apparatus, however, wood- and mineral feedstock were handled separately. Hydrophobic agent, herein Xylan, and mold-preventing agent, herein pine tree bark, were admixed to wood fibrous feedstock at crush-grinding stage. Hydrophobic agent was sprayed over the surface of the resulted blend in order to prevent moisture absorption by fibers and stabilize air bubbles. Resulted wood-fibrous blend was admixed with the excess of

30

35

crush-grinded mineral fibers, initially obtained from the factory, as a clean, chemically unmodified mineral fiber waste, in the ratio of 1 : 8.5. The fibrous blend, obtained as a result of mixing wood- and mineral fibrous feedstock, may be referred as a fire resistant fiber insulate.

5

Simultaneously to fibrous feedstock handling, in the separate apparatus a water-glass was admixed to calcium carbonate (CaCO_3 , chalk). As an alternatively to calcium carbonate, herein, slag (Calcium hydroaluminate) may be used. Sodium Lauryl Sulfate (SLS), utilized herein as a foaming agent, along with stabilizator, was added to the resulting mix. Thus created composition of bonding agent (water glass) and foaming agent (SLS) is sprayed into crush-grinded fibrous blend that was pre-hand moved to the concrete mixer, and mixed therein. At this stage, also EPS-granules (Styrox) may be added to the composite blend in order to increase its volume.

15

Thus obtained composite blend may be cast into the form or air blown into the frame. The composite material is characterized by its perfect thermal insulation parameters. Mean thermal conductivity parameter (λ) of said material, measured in accordance to the standard EN ISO 8031:2009 at a mean temperature of 10°C is in the range of 0.03-0.1 W/mK (Watts/meter per kelvin).

20

The composite material has good fire resistance properties, and may be utilized, for example, in the manufacturing of fire-proof doors for housing and boating industries; fire retardant insulators in machinery and energy industries (e.g. hatches, honeycomb and stem constructions); fire retardant panels for inner walls and ceilings for housing and boating industries; as wells as for use in lifts; or sprayable fire retardant insulators for pipe bridges and cables.

25

Example 5. Manufacturing of sandwich structures.

30

According to one embodiment of the invention a process for manufacturing of a multilayered structure is provided and a multilayered structure product thereof, wherein multilayered structure comprises two hard faces with a middle layer sandwiched in between. Hard faces may be made from steel or other hard material, depending on final application. The middle layer, which fills the "sandwich", may be produced according to previous example 4, and comprise a fire retardant fiber reinforced composite material. This process may be adapted, for example, to the manufacturing of fire-proof doors.

35

Alternatively, the middle layer may comprise a bulk filling, manufactured from natural fibrous feedstock according to example 2, wherein natural fiber component comprises wood- or agro-fibers. This process may be adapted for the manufacturing of e.g. noise barriers.

Example 6. Utilization of a torrefaction process in natural fiber composites manufacturing.

The reference now will be made to the production of natural fiber composites according to Fig. 5.

The process as described below may be integrated into common wood-processing facilities to improve cost-effectiveness and ecological situation therein, since natural fiber materials or biomass that undergoes treatment by torrefaction, are selected from side products of wood- and paper processing industries, such as wood chips, bark, saw dust and the like, and the process itself is recognized by lighter carbon- and water footprints in comparison to corresponding state-of-art processes.

Torrefaction process, as described elsewhere in this document, is introduced into natural fiber composite manufacturing line as a step prior to crush- or impact-grinding. Torrefied biomass further undergoes impact- (or crush-) grinding with particle surface activation, in accordance with some embodiment. The powder, once obtained by mechanical treatment, may be further suspended in bonding agent and/or admixed to other components required to produce a composite material with selected properties. Shaping of the resulted product in accordance with some embodiments, may comprise pelletization, extrusion or casting into forms. Depending on shaping method various products may be obtained, those include torrefied pellets, wood-plastic composite boards and gypsum boards.

Claims

1. A process for the manufacturing of composite material from fibrous feedstock with certain degree of solid content, **characterized** in that it comprises the following stages:
- 5 - defibering fibrous feedstock;
 - introducing bonding agent into defibered blend to form a composite blend;
 - admixing additive(s) into defibered- or composite- blend;
 - foaming the composite blend; and
10 - shaping resulted product,
- wherein defibering of the fibrous feedstock is implemented by impact grinding of fibrous feedstock by means of impact disintegrator with mechanical particle surface activation system adapted to promote chemical bonds formation between particles of various fibrous feedstock by subjecting said particles to collisions at high speed.
- 15 2. A process, in accordance with claim 1, **characterized** in that the collision speed of feedstock particles upon impact grinding is adjusted to be at least 100 m/s and preferably no more than 350 m/s.
- 20 3. A process, in accordance with claim 1, **characterized** in that defibering of the fibrous feedstock is implemented at ambient temperature.
4. A process, in accordance with claim 1, **characterized** in that the foaming stage may be implemented by at least one of the following means:
- 25 - mechanical frothing;
 - introducing a foaming agent into defibered blend, which agent may be physical foaming agent and comprise carbon dioxide, biogas, methane, butane and/or hydrogen gases, and/or which agent may be chemical foaming agent and comprise sodium lauryl sulfate and other washing agents, or coconut
30 dimethylamineoxide.
5. A process, in accordance with claim 1, **characterized** in that the bonding agent may comprise starch, lignin-based adhesives, water glass or two-component glue system such as Melamine-Urea-Formaldehyde system.
- 35 6. A process in accordance with claim 1, **characterized** in that the shaping stage may be implemented by at least one of the following means:
- Casting into forms;

- Press-casting;
 - Roller pressing with optional heating;
 - Layer-by-layer lamination;
 - Calendering;
 - 5 - Extrusion;
 - Injection or compression molding;
 - Air blowing into frame; and
 - Pelletization.
- 10 7. A process, in accordance with claim 1, **characterized** in that the additive may comprise calcium carbonate, slag, gypsum, high alumina cements, hydrophobic agents, mold-growth prevention agents, stabilization agents, EPS, thermoplastic polymers and biocoal, torrefied biomass, such as wood, plants, straw, cereals, such as wheat, corn, barley, and the like, or torrefied pellets.
- 15 8. A process in accordance with claim 1, **characterized** in that the solid content of defibered feedstock is at least 90%.
- 20 9. A process in accordance with claim 1, **characterized** in that the treatment known as torrefaction is applied to an unprocessed fibrous feedstock.
- 25 10. A process in accordance with claim 1, **characterized** in that it implies utilization of fibrous feedstock comprising of wood- and mineral fibers, in particular of outer bark, recycled paper and stone wool, respectively; a diluting agent, which diluting agent is preferably water; a foaming agent, in particular coconut dimethylaminoxide, and a high alumina cement additive; in which process the solid content of defibered feedstock may vary from 2-15% to 15-50%; and which process comprises the stages of fibrous feedstock defibering, admixing the diluting and foaming agents into defibered feedstock, and mechanical froth-
- 30 ing of the resulted blend followed by casting the foamed composite material into the mold.
- 35 11. A process, in accordance with claims 1 and 8, for the manufacturing of natural fiber insulates, **characterized** in that it implies utilization of fibrous feedstock comprising of wood- and agro- fibers, in particular of recycled paper, carton boards, newsprints or clean paper waste, outer bark and sawdust, mechanical pulp fibers and plant fibers; a bonding agent, in particular starch, an optional mold-growth preventing agent, in particular a pine tree bark, a hydrophobic

agent and a foaming agent; which process comprises the stages of fibrous feedstock defibering, admixing bonding agent to defibered feedstock, introducing a foaming agent into the resulted composite blend and press-casting of the resulted natural fiber composite material followed by drying.

5

12. A process, in accordance with claims 1 and 8, for the manufacturing of natural fiber composite material, **characterized** in that it implies utilization of a fibrous feedstock comprising of wood-fibers, and a two-component bonding agent, in particular Melamine-Urea-Formaldehyde system, wherein the defibered feedstock is spread or fixed on the moving line or the other air-permeable base, located over a suction box, and the two-component bonding agent is sprayed over the suction-held defibered feedstock.

10

13. A process, in accordance with claim 1, **characterized** in that it implies utilization of fibrous feedstock comprising of wood fibers, and thermoplastic polymers, wherein the defibered feedstock is mixed with the thermoplastic polymer and the resulted blend is shaped by injection or compression molding, or extrusion, followed by drying.

15

14. A process, in accordance with claims 1 and 8, for the manufacturing of fire retardant insulates, **characterized** in that it implies utilization of fibrous feedstock comprising of wood- and mineral fibers, in particular of outer bark, recycled paper and stone wool, respectively; a bonding agent, preferably water glass, a foaming agent, preferably sodium lauryl sulfate, and additives, preferably calcium carbonate, slag, pine tree bark as mold-preventing agent and Xylan as hydrophobic agent; which process comprises the stages of fibrous feedstock defibering, along with spraying mold-preventing and hydrophobic agent into the defibered blend; introducing admixed beforehand bonding agent, foaming agent and calcium carbonate into the defibered blend by spraying, and further mixing the resulted composite blend followed by shaping by means of casting or air blowing into frame.

20

25

30

15. A process for the manufacturing of multilayered composite structures, **characterized** in that a multilayered structure is provided by sandwiching a middle layer between two hard faces, wherein said hard faces may be steel, and said middle layer comprises either composite material, produced according to the process of claim 14, or natural fiber composite material, produced according to the process of claim 11.

35

16. A composite material manufactured by the process of claim 10, **characterized** in that it possesses thermal insulating properties and may be applied as thermal insulate for construction panels and bearing wall- and ceiling construction elements; for refrigeration equipment and instruments for food industry; for the top layers of special purpose vehicles and for inner- or outer parts for packaging industry.
17. A natural fiber insulate material manufactured by the process of claim 11, **characterized** in that it possesses sound insulating properties and may be used as blowable sound insulate, as acoustic panels, or as a component of a noise barrier, wherein said natural fiber insulate material is sprayed or cast into the lightweight honeycomb structure.
18. A natural fiber composite material manufactured by the processes of claims 12 or 13, **characterized** by its particular stiffness and durability, which material may be utilized for manufacturing of wear-resistant consumer goods.
19. A composite material manufactured by the process of claim 14 **characterized** in that it possesses fire retardant and thermal insulating properties and may be used as a blowable thermal insulate or as insulation material for fire resistant doors.
20. A multilayered composite structure manufactured according to the process of claim 15 **characterized** in that it comprises two hard faces and a middle layer sandwiched in between, wherein said middle layer comprises either composite insulate material of claim 19 or natural fiber insulate material of claim 17.
21. A fire proof door manufactured according to the process of claim 15 **characterized** in that it comprises two outer hard faces and a fire retardant middle layer in between, wherein said outer hard faces are made from steel and a fire retardant middle layer is produced according to the process of claim 14.
22. A natural fiber composite material manufactured by the process of claim 9 **characterized** by its strength-to-weight ratio, hydrophobicity and durability.

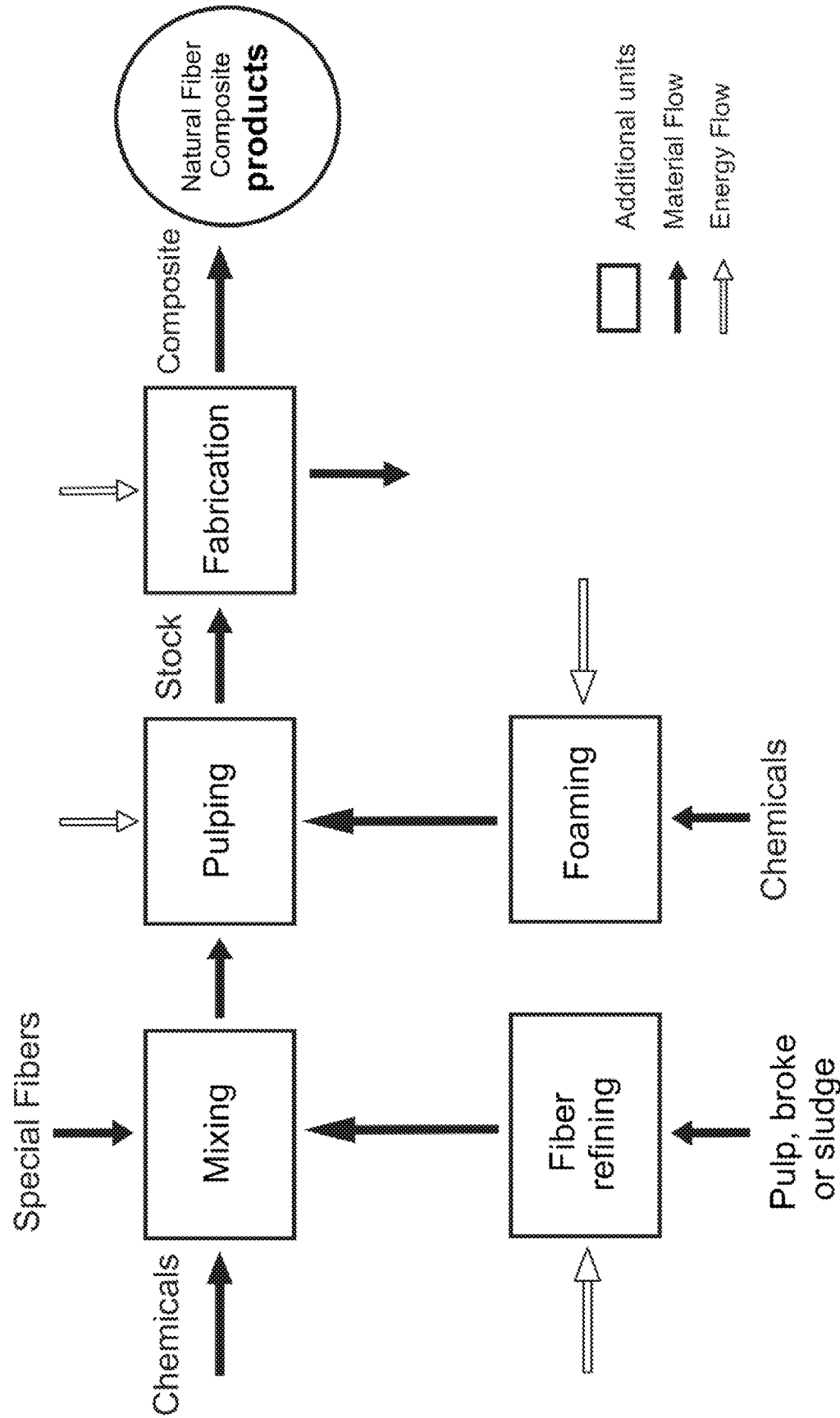


Figure 1

2/5

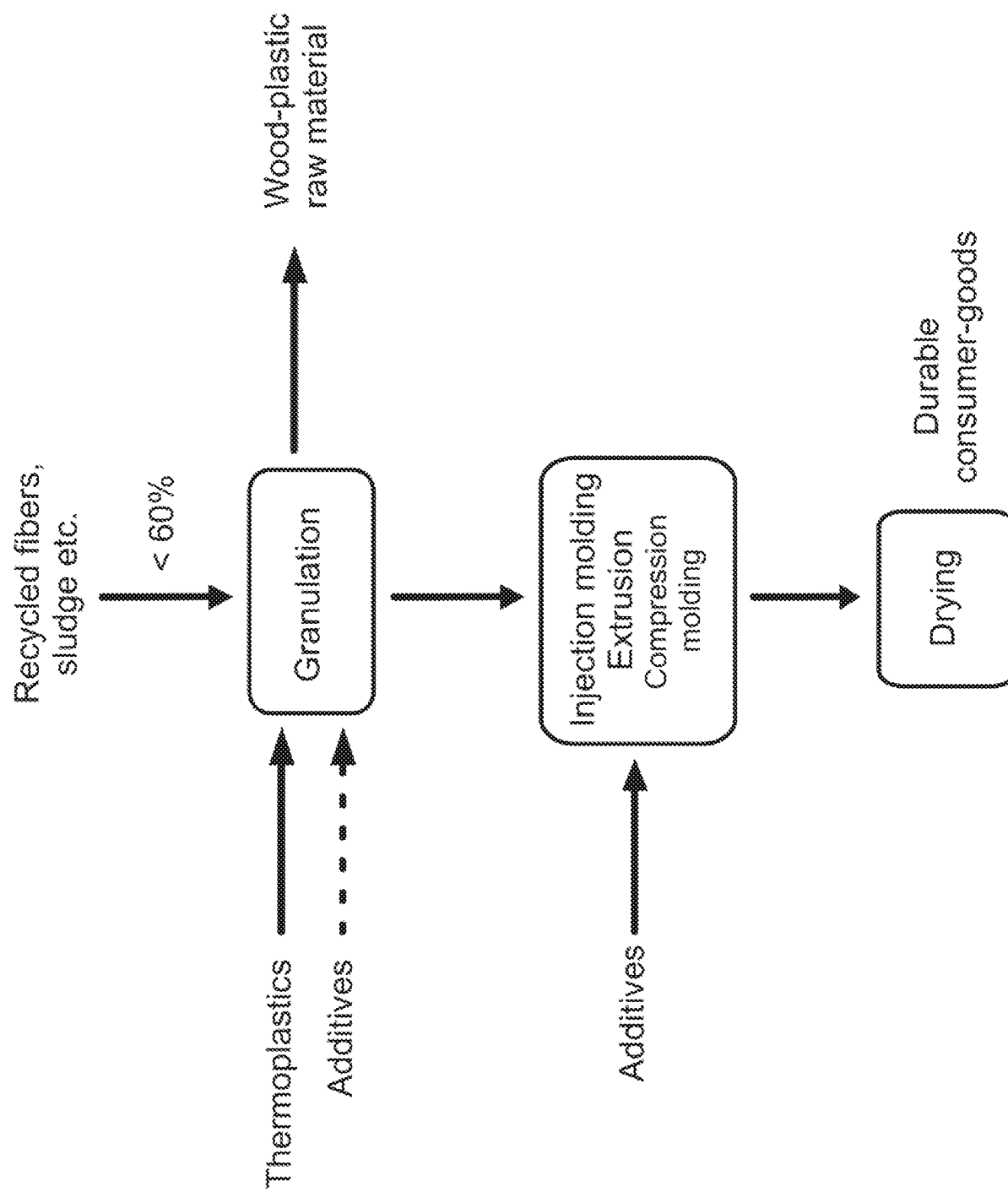


Figure 2

3/5

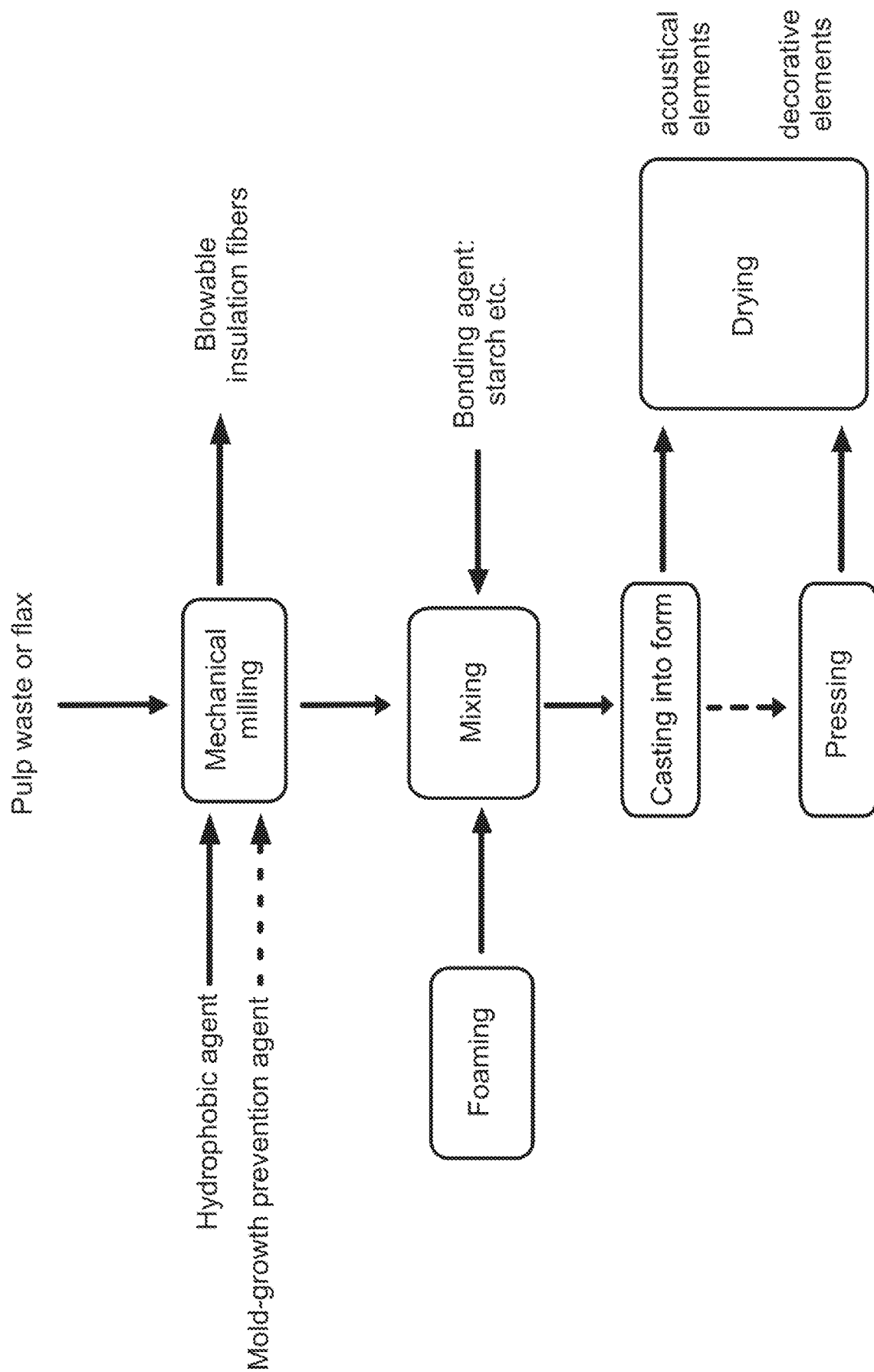


Figure 3

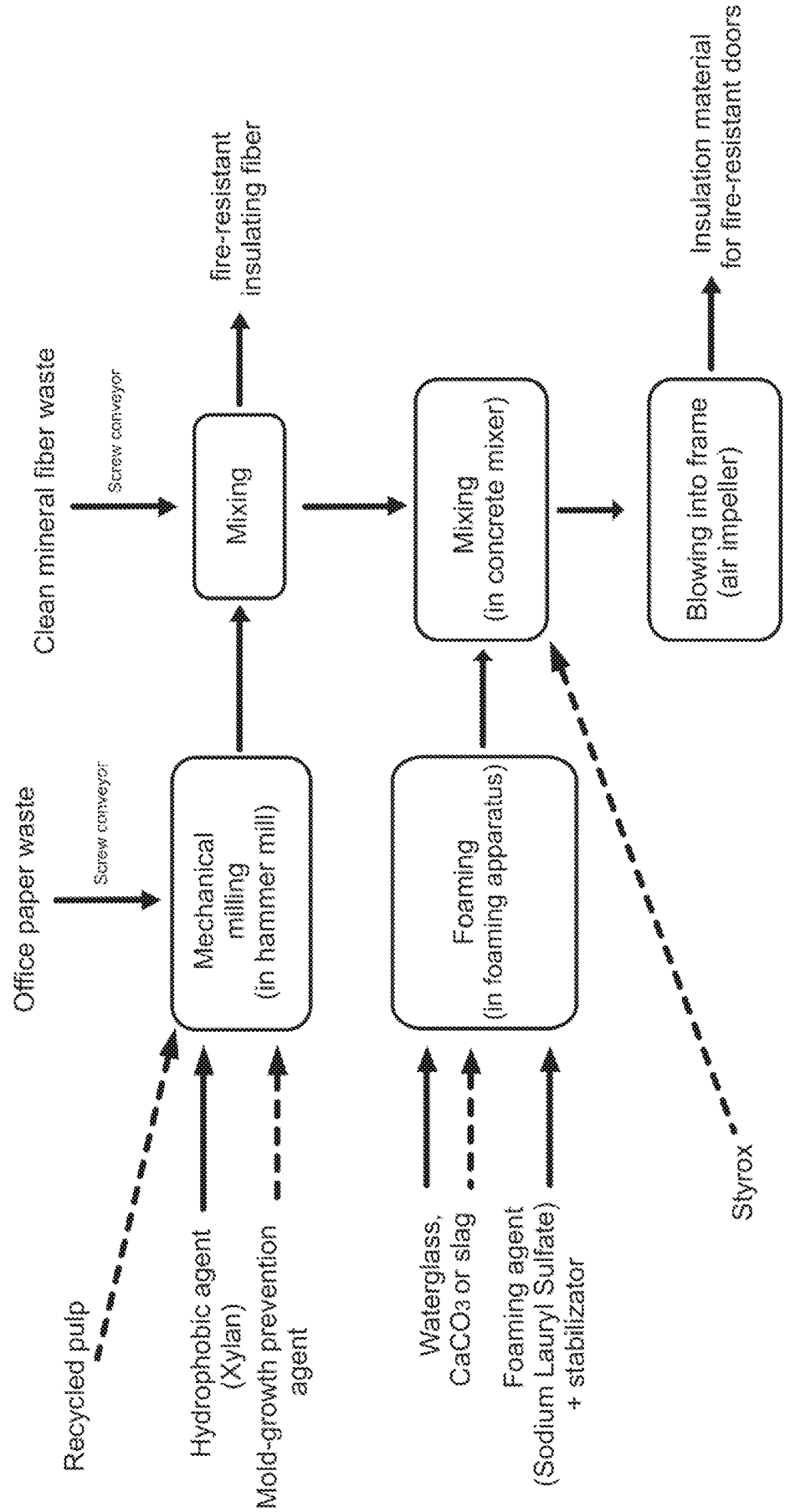


Figure 4

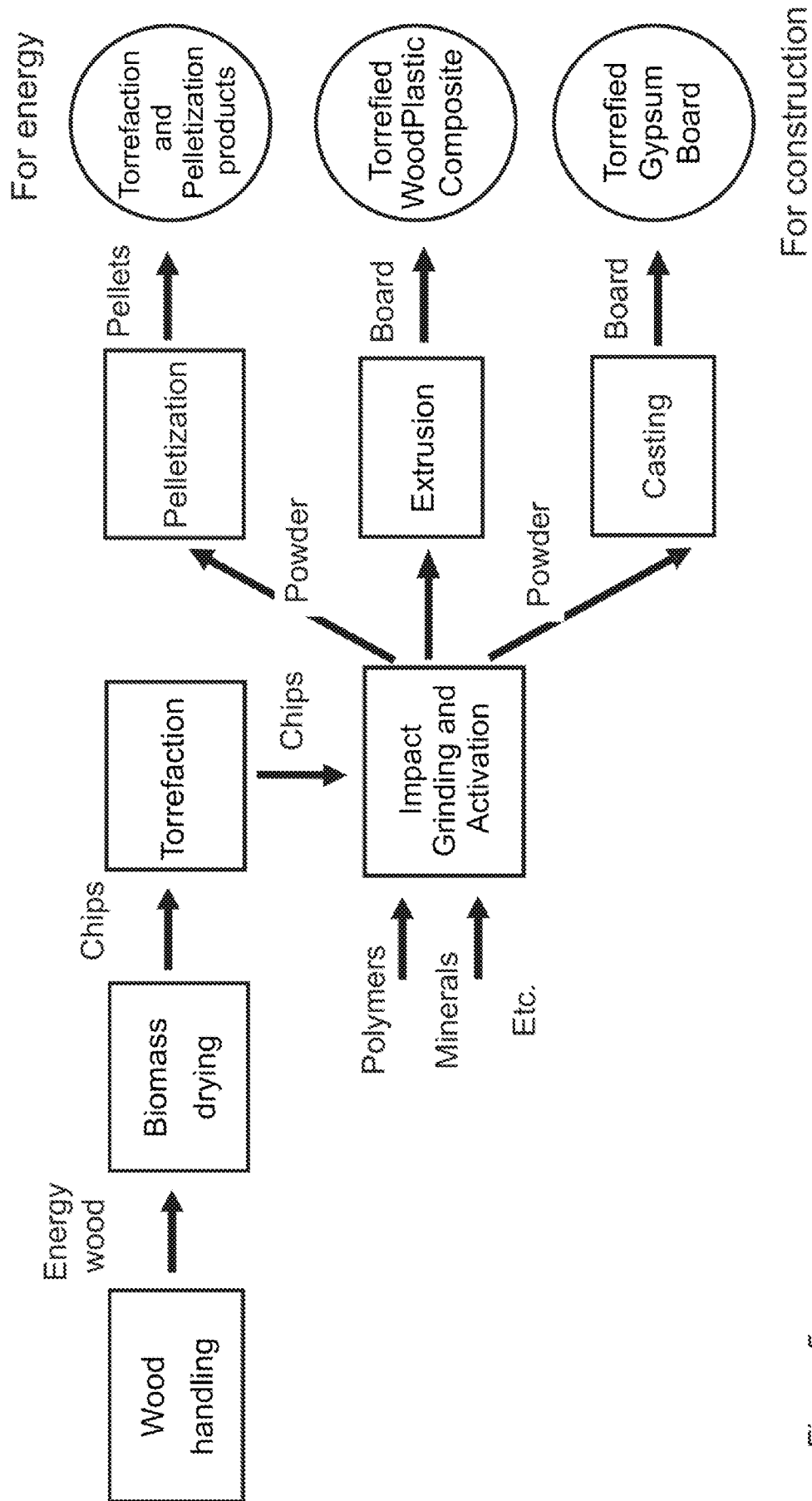


Figure 5