A method of producing a fire retarded cellulose fiber material composed of both a liquid fire retardant and a powdered fire retardant useful in producing insulation and other such products is provided.
HYBRID FIRE-RETARDED CELLULOSE MATERIALS

FIELD OF THE INVENTION

[0001] The present invention generally relates to the production of a fire-retarded cellulose insulation product, and more particularly to the manufacture of fire-retarded cellulose insulation materials using a process which involves applying both liquid and powder fire retardant compositions.

BACKGROUND OF THE INVENTION

[0002] Cellulose compositions combined with fire retardant materials are widely used in the construction industry. Specifically, fire-resistant cellulose materials are traditionally used for thermal insulation in the walls and attic spaces of homes and commercial buildings. Insulation products of this type are designed to prevent heat loss and correspondingly insulate building structures from the outside environment. Raw materials used to produce cellulose insulation products may involve many different paper compositions ranging from recycled newspaper to cardboard, paperboard, and fiberboard. These materials are physically processed to produce a finely-divided material having a low bulk density.

[0003] Burning or combustion of cellulose fiber materials such as paper, cardboard, etc., generally involves two different chemical processes: (a) flaming, which results from ignition of gases released by the pyrolysis of the cellulose fiber, and (b) smolder, a slow, high temperature, flameless combustion which results from the oxidation of the remaining carbon-rich material, as with charcoal in a barbecue. The basic difference between smoldering and flaming combustion is that smoldering combustion occurs on the surface of a solid rather than in the gas phase.

[0004] Cellulose insulation is flammable and prone to smoldering, and it is well known in the cellulose insulation industry that chemical additions to a paper source material will increase its resistance to burning. It is also known that some chemicals will extinguish flaming but not smoldering combustion. Examples of such chemicals include borax pentahydrate, hydrated magnesium sulfate and aluminum trihydrate, among others. It is further known that other chemicals can extinguish both flaming and smolder. Examples of these chemicals include ammonium sulfate, ammonium phosphate and boric acid. However,
the ammonium compounds can be corrosive under numerous conditions and boric acid can lead to infertility (according to the European and U.S. governments) and, consequently, warning labels are mandated for all cellulose bags.

[0005] Cellulose insulation is required to meet federal regulations and certain government regulations have mandated that cellulose insulation should not support burning under normal environmental conditions. The test methods that assure these properties are set forth in ASTM C-739. Testing for flaming is carried out using critical radiant flux (CRF) equipment. In that test, a "pass" is achieved if the cellulose material will not support surface flame while being subjected to radiation of 0.12 watts/cm² or less. Smolder testing is performed using a smolder box. A loss in weight of less than 15% of the original cellulose weight constitutes a "pass." In Europe, various classifications are available ranging from Category A to Category F. Chemical loadings to allow cellulose to meet ASTM C-739 would also allow it to meet a Category B. To obtain a Category C listing lesser amounts of chemical are required than would be required to meet ASTM C-739.

[0006] To achieve an approved level of flame and smolder resistance, the selected cellulose materials are combined with fire retardant compositions during the production process. Many different fire retardants may be used for this purpose, which aretraditionally applied in powder (dry) form. Fire retardant compositions include but are not limited to monoammonium phosphate, diammonium phosphate, boric acid, ammonium sulfate, sodium tetraborate, aluminum trihydrate, and mixtures thereof. These materials, as well as other fire retardant compositions and additional information regarding the production of cellulose insulation products are discussed in U.S. Pat. No. 4,168,175 to Shutt and U.S. Pat. No. 4,595,414 to Shutt. In order to meet US government standards for cellulose insulation, the loading of an all-powdered fire retardant chemical is typically about 14 to 18 % by weight (wt%) of the final insulation product, and the loading of an all-liquid fire retardant chemical is typically about 6 to 11 % by weight of the final insulation product.

[0007] After combining the selected fire retardant compositions and cellulose materials, the resulting product is physically processed using conventional mechanical devices (e.g. hammermill and or fiberization systems known in the art) to produce a fiberized, finely divided insulation product. In accordance with traditional processing technology, fire-
resistant cellulose insulation products are specifically prepared using powdered chemicals. The selected cellulose materials (e.g. recycled/used paper products) are subjected to multi-stage size reduction by grinding or other conventional processes using standard equipment including but not limited to fiberization systems. At the fiberizer, during the size reduction process, a fire retardant composition in powder (dry) form is combined/mixed with the cellulose materials. Traditionally, mixing of cellulose materials with a powder form of a fire retardant composition is undertaken in the final grinding stage of the system.

[0008] Powdered (dry) fire retardant compositions present numerous disadvantages. These disadvantages include, but are not limited to, (1) the generation of substantial amounts of dust which requires elaborate safety and environmental control systems; (2) the need to use large amounts of chemicals (e.g. fire retardants) due to production inefficiencies associated with powder-type systems; and (3) increased material costs associated with the need to use large quantities of powdered chemicals.

[0009] U.S. Pat. No. 9,045,605 to Shutt discloses an all-liquid fire retardant system that entirely avoids the use of any fire retardants in powdered (dry) form and produces a low-dust fire retarded cellulose insulation material. One of the benefits of the method described in U.S. Pat. No. 9,045,605, is a final cellulose insulation product that has been processed (e.g., screened) to eliminate dust prior to packaging and meets all applicable government requirements.

[0010] Drawbacks of using an all-liquid fire retardant system include the balancing of chemistries, high operating costs, and the need for more complicated equipment.

**SUMMARY OF THE INVENTION**

[0011] The present invention involves a unique and distinctive hybrid fire retardant system (combination of powder and liquid systems) that produces a fire-retarded cellulose material useful in producing insulation and other such products. In embodiments, the material is a low-dust, fire-retarded cellulose material.

[0012] Processes of the present invention provide advantages unattainable with either an all-powder fire retardant chemical system or an all-liquid fire retardant chemical system. Compared to all-powder fire retardant chemical systems, the advantages of the present hybrid
fire-retarded system include, but are not limited to: (1) the substantial elimination of dust and
the safety considerations associated therein (and the elimination of the corresponding
necessity to label the final product for toxicity), and (2) a substantial reduction in chemical
cost.

[0013] Compared to all-liquid fire retardant chemical systems, the principle advantage of
the present hybrid fire-retardant system is the reduction in the cost of capital equipment (e.g.,
fewer burners and sprayers, and their corresponding ducting and fans) and the associated
reduction in operating costs, due to reduced utility costs. Moreover, the present hybrid fire-
retardant system-treated cellulose material is in compliance with government requirements
for insulation. A further advantage of this hybrid system is that the fire retardant chemicals
contain neither borates nor ammonium compounds. Borate chemicals are expensive and
require a health warning on the cellulose insulation package. Ammonium compounds can be
malodorous and corrosive.

[0014] In embodiments, the invention provides methods for producing a cellulose fiber
material useful in producing insulation and other such products. In embodiments of a
method of the invention, a liquid comprising water, phosphoric acid and at least one fire-
retarding material soluble in the water (e.g., sulfuric acid or other inorganic acid other than
phosphoric acid) is applied to a cellulose source material. In embodiments the liquid consists
essentially of water, phosphoric acid and sulfuric acid, and optionally one or more additives.
In embodiments, the liquid is allowed to permeate into the cellulose source material. In
embodiments, the liquid permeated material is dried to remove the solvent while the fire-
retarded material remains in the cellulose source material.

[0015] It is a cornerstone of the invention that phosphoric acid is present in the cellulose
material. Although phosphoric acid is a flame retardant, its principle use in the present
invention is as a smolder inhibitor. It was found that compared to boric acid, ammonium
sulfate and ammonium phosphate, phosphoric acid is the only known economical chemical
that is capable of effectively inhibiting smolder to satisfy government requirements of
cellulose materials and can be used in a safe and cost effective amount to achieve an effective
fire-retarded cellulose product without the problems of offgassing associated with
ammonium compounds and health issues associated with borate chemicals.
In embodiments, the dried cellulose source material is then reduced in size to produce a partially fire-retarded cellulose fiber material. By "partially fire-retarded," it is meant that the cellulose material does not yet meet approved government requirements. Consequently, in embodiments, a powdered fire retardant chemical is added to the cellulose fiber material in a sufficient quantity to bring the cellulose insulation into compliance with government requirements for insulation. In embodiments, the partially fire-retarded cellulose fiber material can be de-dusted to reduce the amount of dust for producing a low to no dust partially fire-retarded cellulose fiber material. In embodiments, the dedusting phase can be eliminated and the powdered chemical flame retardant added at the final grinding stage to bring the cellulose insulation into compliance with government requirements for insulation.

"Composition" and like terms mean a mixture or blend of two or more components.

The term "fire" refers to the process of burning of cellulose by flame or smolder. The terms "flame" and "flammable" refer to the burning of gases resulting from pyrolysis due to heat. The term "smolder" or smoldering combustion refers to the burning of a carbon-rich material remaining after gases have devolved (e.g., as with charcoal in a barbeque). Both burning processes can be chemically tested by the methods, for example, as outlined in ASTM C-739 (Standard Specification for Cellulosic Fiber Loose-Fill Thermal Insulation) and as mandated by law.

The term "fire resistant" means resistance to flaming and smoldering combustions. The term "fire-retardant chemical" refers to a chemical substance or mixture (other than water) that reduces flammability or smolder of a cellulose material.

The term "liquid fire retardant composition" and like terms, means an aqueous solution with fire-retardant chemicals dissolved therein. A liquid fire retardant chemical composition can optionally include one or more non-fire retardant additives (e.g., surfactants).

The term "powder fire retardant chemical composition" and like terms, means a dry (non-aqueous) composition of one or more powder fire retardant chemicals. A powder fire retardant chemical composition can optionally include one or more non-fire retardant additives (e.g., pest control additives).
[0022] The term, "hybrid fire retardant system" and like terms, means the application of both a liquid fire retardant composition and a powder fire retardant composition to a cellulose-based material. In embodiments, the liquid fire retardant composition and the powder fire retardant composition are added separately to the cellulose material.

[0023] The term "bulk density" as used herein is defined to encompass the weight (traditionally in lbs. ft$^3$) of the final settled insulation product. A final product with a low bulk density is desired because it imparts less weight to the building structure in which it is used. In addition, a final product with low bulk density is more free-flowing, easier to handle, and more readily installed. In addition, since cellulose insulation is typically sold by coverage (i.e., volume), an insulation material with a lower bulk density enables a manufacturer to sell less weight without diminishing performance. Furthermore, the fiber materials in the final or completed product have a higher degree of rigidity which results in less settling of the product when used in a building structure compared with insulation products that are conventionally prepared using fire retardants in all-powdered (dry) form. Minimal settling of the insulation product is beneficial because it enables less of the product to be used, thereby providing significant cost savings. The completed insulation product can also characterized by a substantial absence of detached fibrous residue which, if present, can increase its density. Accordingly, the present invention represents an advance in the art of cellulose insulation manufacture, and provides many economic, safety, quality-control, and other benefits compared with all-liquid fire retardant systems and all-powder fire retardant systems, as further discussed below.

[0024] Unless stated to the contrary, implicit from the context, or customary in the art, all parts and percentages are based on weight.

**DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENT**

[0025] The present invention provides a unique and highly efficient method for producing de-dusted or, in embodiments, non-dedusted, fire-resistant cellulose (e.g., paper-based) insulation product. The present methods achieve the above-described benefits, at least in part, through the use of both a liquid fire retardant composition and a powder-type fire retardant composition.
To manufacture a fire-resistant cellulose insulation product utilizing a hybrid system, embodiments of the present invention are characterized by a combination of unique processing steps which (i) enable an amount of liquid fire retardant compositions to be diffused within the selected cellulose materials, (ii) permit complete drying of the cellulose materials while producing minimal amounts of fine fibrous residue, (iii) in embodiments, optionally permit the removal of a majority to substantially all dust from the dried and size-reduced fire-resistant cellulose insulation product, and (iv) enable a powdered fire-retardant chemical to be added to the dried and size-reduced fire-resistant cellulose insulation product pneumatically, such that the final fire-resistant cellulose fiber material possesses the requisite level of fire retardance to meet government standards. In embodiments, the cellulose material has a CRF value of 0.12 watts/cm² or greater, as measured in accordance with ASTM C-739. In embodiments, the final fire-resistant cellulose fiber material has a settled bulk density of 1 to 2 lbs/ft³.

Processes for applying liquid fire-retardant compositions to cellulose materials are known in the art and are described in USP 5,534,301 and USP 9,045,605.

In an embodiment, a supply of cellulose material is initially provided. In embodiments, the cellulose material will basically involve vegetable fiber materials, wood fiber compositions, or any other cellulosic substrates which are known in the art for producing insulation materials. Preferably, the supply of cellulose material will consist of virgin (unused) or recycled (used) paper, with the term "paper" encompassing commercial products derived from wood or other plant materials ranging from newspaper to cardboard, fiberboard, and paperboard. However, the present invention shall not be limited to any particular paper or cellulose compositions, with a variety of different materials being suitable for use in the system. In an embodiment, the cellulose material comprises wood or other plant materials, for example, cotton, flax, hemp, kenaf and jute, among others, known and used in the art for producing cellulose-based materials, for example but not limited to, unused or used (recycled) paper such as newspaper, cardboard, fiberboard, paperboard, Kraft paper, etc. In a preferred embodiment, paper is used as the supply of cellulose material. The term "paper" as used herein shall encompass a wide variety of vegetable or wood-based fiber materials ranging from conventional paper products to cardboard, fiberboard, and the like. Furthermore, the selected paper materials can include virgin (unused) products or, in a
preferred embodiment, recycled paper. An exemplary and preferred product suitable for processing in accordance with the invention involves recycled (used) newspaper (optimally "grade 8" newspaper).

[0029] In embodiments, the selected supply of cellulose material (e.g. recycled newspaper, Kraft paper, etc.) is then loaded onto a feed table or a feed belt, where the material is sorted and separated from non-cellulose materials and other materials. In embodiments, the cellulose material can then be routed via a conveyer belt system, for example, into a shredding and/or grinding apparatus to physically reduce the size of the material to a desired level and produce a plurality of individual pieces of paper which, in a preferred embodiment, have an average width and length of about 2-6 inches (about 5-15 cm). While these numerical values are preferred for use in the claimed process, the present invention shall not be limited to the foregoing numerical parameters which are provided solely as an example. The precise paper size to be used at this stage of the process can be determined in accordance with preliminary pilot studies on the paper material being processed and treated.

[0030] In embodiments, the individual pieces of paper are then transferred from the shredding apparatus into an air transport system as known and used in the art for material transfer, which uses an air flow to move the pieces of paper to a paper storage bin. In embodiments, from the bin, an auger can deliver paper at a fixed and constant rate to next stage of the system, i.e., a spraying system. In embodiments, a conventional spraying apparatus such as a standard spray booth can be utilized. In embodiments, the spraying system is designed to deliver at least one liquid fire retardant composition to the pieces of paper, and can include one or more spraying nozzles connected to a tank containing the selected liquid fire retardant composition. Within the spraying system (e.g., spray booth), the pieces of paper are converted into a fire retarded-soaked paper product, which comprises the initial pieces of paper soaked with the liquid fire retardant composition.

[0031] In embodiments, to ensure proper and complete diffusion of the liquid fire retardant composition within the paper, the liquid fire retardant composition is optimally delivered to the paper materials in the form of a fine mist comprising a plurality of droplets each having a diameter of about 40-200 microns. Using this approach, the selected fire
retardant composition can adequately and completely diffuse into the fibrous matrix of the paper. Spraying of the liquid fire retardant composition in a fine mist can provide many benefits including but not limited to (1) a reduction in the amount of liquid fire retardant composition that is needed; (2) greater dispersion of the composition within the internal fibrous matrix of the paper; and (3) a lack of or reduced amount of chemical fire retardant dust in the final product as discussed below.


[0033] In embodiments, acidic aqueous solutions of inorganic acids may be used as the liquid fire retardant composition, preferably sulfuric acid, phosphoric acid, and mixtures thereof. In embodiments, the liquid fire retardant composition is an aqueous solution containing an inorganic acid such as sulfuric acid and phosphoric acid, and combinations thereof. In embodiments, the liquid fire retardant composition is an aqueous solution comprising phosphoric acid and sulfuric acid, and optionally, one or more additives. In embodiments the liquid fire retardant composition is an aqueous solution consisting of, or consisting essentially of, phosphoric acid and sulfuric acid, and optionally, one or more additives.

[0034] Although inorganic acids and many organic acids can effect fire retardance, sulfuric acid and phosphoric acid are the only effective and commercially acceptable acids in that they can be used effectively in reduced quantities and in a cost effective, economical manner. Sulfuric acid is a very effective flame retardant and a modest smolder suppressant; it does not prevent re-smolder or secondary smolder in an air stream. In addition, the price of sulfuric acid is commercially acceptable. Phosphoric acid is a modest flame retardant and
ultimate smolder suppressant. While boric acid approaches the efficiency of phosphoric acid, it is more expensive and its use requires health warnings to be placed onto the container/bag of material, which serves as an impediment to sales. In addition, regulations banning the use of borates in Europe are to set to be implemented, which could follow in the USA.

[0035] In an embodiment, an inorganic acid is used alone, rather than an acid salt, for example, the use of sulfuric acid rather than ammonium sulfate, or the use of phosphoric acid rather than ammonium phosphates. The use of pure inorganic acids as the liquid fire retardant reduces costs due to reduced raw material costs, and eliminates the possibility of ammonia offgassing. Ammonia off-gassing is associated with using ammonium-containing fire retardants (e.g., ammonium sulfate) and is considered hazardous (e.g. to health). In an embodiment, the liquid fire retardant composition excludes ammonium-containing fire retardants (e.g., ammonium sulfate). Borate is known to be harmful to male reproductive health. In an embodiment, the liquid fire retardant composition excludes borate-containing fire retardants (e.g., sodium tetraborate, boric acid, etc.).

[0036] In an embodiment, aqueous solutions of many different fire retardant chemicals can be used in the described process, with the present invention not being limited to any particular agents or combinations thereof.

[0037] In embodiments, the selected liquid fire retardant composition can be formulated as an aqueous solution, preferably having about 10-40% by weight total of one or more fire retardant compounds dissolved therein. In embodiments, the application of the liquid fire retardant composition will produce a fire retardant-soaked paper product which (prior to drying) will contain about 9-40% by weight of the liquid fire retardant composition. In embodiments, upon drying, the dried insulation product will typically contain about 1-12% by weight, or, in some embodiments, 1-5% by weight, or, in some embodiments, 2-3 % by weight, of the selected fire retardant compound(s) which were applied in solution form. In an embodiment, the dried insulation product contains 1-5% by weight of the selected fire retardant compound(s) which were applied in solution form. In embodiments, the liquid fire retardant composition can further include one or more optional additives such as a wetting agent.
**Dwell time**

[0038] In embodiments, the fire retarded-soaked paper product can then be transferred into a drying chamber along with a stream of heated air. However, in embodiments, between the application of a selected liquid fire retardant composition to the paper and passage of the fire retardant-soaked paper product into the drying chamber, a given amount of "dwell" (delay) time period is allowed to lapse. A sufficient amount of dwell time ensures diffusion of the liquid fire retardant composition into the interior regions of the pieces of paper. In a preferred embodiment, a dwell time period of about 45-120 seconds will be allowed to lapse after application of the liquid fire retardant composition to the paper materials, with the exact time period depending on the type of paper being employed and other experimentally-determined factors.

[0039] The imposition of dwell time at this stage in the system can be accomplished in many ways, with the present invention not being limited to any particular method. For example, in embodiments, prior to passage of the fire retardant-soaked paper product into the drying chamber, the paper product can be allowed to reside in one or more stationary hoppers or containment vessels for a selected amount of time. Likewise, in embodiments, after production of the fire retardant-soaked paper product, the paper product can be conveyed to subsequent parts of the processing system using conventional transfer systems (e.g., feed screws, conveyor belts, and the like), which are operated at a controlled rate of speed to impart a delay in moving the paper product through the system. In embodiments, this procedure may be employed with or without the use of stationary hoppers to provide a sufficient degree of dwell time.

**Drying**

[0040] In embodiments, prior to or simultaneously with the entry of paper product into the drying chamber, a stream of heated air is passed into and through the drying chamber. In embodiments, the stream of heated air is designed to simultaneously move and dry the paper product within the chamber. In a preferred embodiment, the drying chamber is circular in cross-section and tubular in design with a longitudinal axis therethrough.

[0041] In embodiments, to achieve optimum drying of the fire retardant-soaked paper product within the drying chamber, it is preferred that the stream of heated air is introduced
(delivered) into the drying chamber in an angled and non-parallel orientation relative to the longitudinal axis of the drying chamber. In embodiments, the angle of air introduction will preferably be about 90° relative to the longitudinal axis of the drying chamber so that the stream of heated air enters the drying chamber in a direction which is perpendicular to the longitudinal axis of the chamber. However, depending on the particular configuration of the system, the angle of air introduction relative to the longitudinal axis of the drying chamber can range from about 60°-90°, with about 90° being preferred. In embodiments, it is also preferred that the stream of heated air be introduced in a manner wherein the stream is laterally offset from (e.g. to the side of) the longitudinal axis of the drying chamber. As a result, the stream of heated air entering the drying chamber will travel in a substantially helical pathway around and along the circular interior surface of the chamber wall which slows the movement of paper product passing through the chamber. In a preferred embodiment, the stream of heated air is introduced into the chamber at a flow rate of at least about 2500-3500 ft./min. (which may be varied as necessary in accordance with preliminary pilot studies on the materials being processed).

[0042] In embodiments, the fire retardant-soaked paper product is passed into and through the drying chamber in combination with the stream of heated air after completion of the dwell time period. In embodiments, the stream of heated air is designed to simultaneously move the paper product through the drying chamber to achieve complete drying of the paper product within the chamber. However, in embodiments, an additional amount of dwell time can be imparted to the paper product within the drying chamber to ensure that the paper product is completely dried. If the paper product is allowed to flow through the drying chamber with the stream of heated air in an uninterrupted manner, the paper product may not be completely dry upon leaving the chamber. Although introduction of the heated air in a helical flow path causes the paper product to pass through the chamber at a slower rate (compared with a linear flow path), in embodiments, additional dwell time may be needed to ensure complete drying.

[0043] To completely dry the fire retardant-soaked paper product, in embodiments, the process involves temporarily interrupting passage of the fire retardant-soaked paper product and heated air through the drying chamber periodically (e.g., at least once and preferably multiple times) during movement of these components through the drying chamber. This
step slows the flow of the paper product and heated air through the drying chamber, which enables greater contact between the heated air and paper product. Since interruption of these components is temporary and periodic (e.g., at selected intervals), once the paper product and air begin moving again after being interrupted, the stream of air accelerates faster than the paper product. This occurs because the air is lighter and less dense than the paper product. As a result, the stream of heated air flows over the surface of the slower-moving paper product, causing more intimate contact and increased drying of the paper product. In this regard, the more interruptions of the foregoing paper product and the air, the greater the drying capacity of the system. Without temporarily and periodically interrupting (e.g. slowing) the foregoing components as they move through the drying chamber, an inadequately-dried material would be generated. As a result of the above-described process, a dried fire-resistant cellulose insulation product is generated within the drying chamber.

[0044] There are numerous ways for temporarily and periodically interrupting the flow path of the fire retardant-soaked paper product and the stream of heated air as they pass through the drying chamber. Accordingly, the present invention shall not be limited to any particular method or apparatus for this purpose. In a preferred embodiment, temporary interruption of the paper product and air as they flow through the drying chamber can be accomplished through the use of a chamber which includes one or more stationary or movable baffle members therein. In a preferred embodiment, the drying chamber includes moveable (e.g. rotatable) baffle members that are continuously moved within the drying chamber during passage of the heated air and paper product therethrough. As a result, the paper product passing through the drying chamber comes in contact with (e.g. physically engages) at least one and preferably multiple baffle members during movement of the baffle members within the chamber. Engagement of the paper product with the baffle members temporarily interrupts the transportation and flow of the paper product through the drying chamber. The same situation occurs regarding the stream of heated air as it moves through the drying chamber. As a result, passage of the paper product through the drying chamber is substantially slowed (compared with a chamber which lacks any baffle members therein). While a delay also occurs regarding the stream of air as it encounters the baffle members and moves through the chamber, this delay is less compared with the paper product due to the minimal weight and density of air. This process in which the paper product experiences a
greater degree of delay or "dwell" (delay) time within the chamber compared with the stream of heated air enables a more continuous and sustained level of air flow over and in contact with the paper product. As a result, the paper product is completely dried so that an adequately dried fire-resistant cellulose insulation product can be produced within the drying chamber.

[0045] In embodiments, the wetted cellulose material is transferred by a stream of air (e.g., heated air) into a drying chamber such as a rotary drier and a tumble drier, among others. In embodiments, the dried cellulose material is "air dry" (e.g., 90 to 95% dry) having a 5% to 10% moisture content.

Fiberizer/Hammer mill

[0046] In embodiments, after passage of the cellulose material through the drying chamber, it is further processed as desired to create a material with additional size reduction and specific size characteristics. In embodiments, size reduction can be accomplished, for example, using one or more fiberizing systems or hammermill units, or other comparable systems known in the art for this purpose, to further reduce the size of the shredded material into smaller pieces, for example, to an average length and width of about 0.01-0.2 inch (about 0.025-0.5 cm) to produce fiberized product (i.e., a size-reduced insulation product).

[0047] In embodiments, the dry, fire retarded-treated cellulose material can be transferred, for example, by air flow, to a hammer mill, fiberizer or both. In embodiments, the cellulose material can be dried, for example, by applying heat to the wetted material prior to or during the transfer of the material.

[0048] Processing through a fiberizer reduces the size of the cellulose material to a desired, final reduced size, fluffy form. In embodiments, for a cellulose insulation product, it is preferred that the final product will have a settled bulk density of 1 to 2 lbs/ft³ (e.g., 1.2 to 1.6 lbs/ft³).

[0049] In an embodiment, a powdered (dry) fire retardant chemical (or chemical composition) can be added to the dried cellulose material within the fiberizer/hammer mill.

[0050] In another embodiment, the dried cellulose material can be reduced in size within the fiberizer/hammer mill without the addition of powdered fire retardant chemical. In
embodiments, the reduced size cellulose material can then be subjected to a dust removal (de-dusting step) and subsequently combined with the powdered fire retardant chemical.

[0051] In an embodiment, as the dried cellulose material is metered into the fiberizer/hammer mill (e.g., using a metered delivery system such as an auger), a powdered fire retardant chemical (or chemical composition) can be metered into the apparatus (e.g., using a device such as an auger).

[0052] In embodiments, nonlimiting examples of powdered fire retardant chemicals that can be applied include gypsum (dihydrate), Epsom salts (magnesium sulfate heptahydrate), and combinations thereof. These chemicals present no danger of corrosion, dust generated during a de-dusting step can be used in agriculture, and warning labeling on the cellulose product would not be required.

[0053] The amount of the powdered fire retardant chemical (or chemical composition) added to the liquid fire-retarded cellulose material is associated with the effectiveness of the powdered chemical and the level of fire retardancy needed to meet government standards for insulation. In embodiments, from 5, or 8 or 10, up to 18, or 20, or 23 or 25 parts of powdered fire retardant is added per 100 parts of the dried cellulose material. In embodiments, 5 to 25 parts of powdered fire retardant is added per 100 parts of the cellulose material. In embodiments, as the dried cellulose material is reduced in size, it mixes with the powdered fire retardant whereby the powdered fire retardant chemical (or chemical composition) uniformly coats and adheres to the reduced-size, cellulose material.

[0054] In an embodiment, as an optional step after processing the dried cellulose material in a fiberizer/hammer mill in combination with a powdered fire retardant chemical, the material can then be subjected to a dust removal (de-dusting) process, as described below.

[0055] In embodiments, in a cellulose insulation product that has been processed using a dry powder fire-retardant chemical, the reduced size dry product will contain about 14-26 % by weight dust based on the total weight of the material, composed of about 10-20 % by weight of cellulose-based (e.g., paper) dust and about 4-6 % by weight of fire-retardant dry chemical dust. Consequently, in embodiments, de-dusting of a reduced-size cellulose insulation product processed using a dry powder fire-retardant chemical (or chemical composition) removes a significant amount of fire-retardant chemical material from the
product resulting in a product that does not meet federal or industry standards for a fire-retarded material.

[0056] In embodiments, if the powdered fire retardant chemical (or chemical composition) is added in a fiberizer to the liquid fire retardant treated cellulose material, and the material is then de-dusted, depending upon the added percentage of liquid fire retardant chemical, an amount of powdered fire retardant chemical (or chemical composition) can be combined with the fiberized cellulose material to meet government standards for insulation. In embodiments, after de-dusting, about 2 to 10 parts of powdered fire retardant can be added per 100 parts of the dried cellulose material.

[0057] The combined amounts of the liquid fire retardant composition and the powdered fire retardant composition are effective to provide a sufficient level of fire retardance to the cellulose material to meet government standards for both the smoldering and combustion test and the flame spread burn test, as outlined in ASTM C-739 (Standard Specification for Cellulosic Fiber Loose-Fill Thermal Insulation), and/or European standards EP Category B and/or EP Category C.

[0058] In an embodiment, the cellulose product can then be discharged (e.g., blown) from the fiberizer/hammermill (or the de-dusting apparatus if utilized) and deposited into a holding bin or conveyed to a bale press or baler or to a bagging apparatus as known and used in the art, and packaged for transport and future use. The resulting product contains an amount of the liquid fire retardant chemical and the powdered fire retardant chemical (or chemical composition) such that it meets government standards for fire-retarded insulation materials (e.g., a CRT value of 0.12 watts/cm² or greater, as measured in accordance with ASTM C-739).

[0059] In another embodiment, the dried cellulose material can be reduced in size within the fiberizer/hammer mill without the addition of powdered fire retardant chemical. In embodiments, the reduced size cellulose material can then be subjected to a dust removal (de-dusting step) and subsequently combined with the powdered fire retardant chemical.

[0060] In embodiments, the fiberizing or size reduction processing to produce the dried fire-resistant cellulose insulation product can produce a substantial quantity of dust (i.e., material with very small particle size), which contains residues and chemicals that can be
easily inhaled. In general, dust material is finely divided solid particles that can be readily suspended in the air and/or remain suspended in the air for a time after the bulk material has substantially settled. Although the dust that is generated is generally about 10% less than the amount produced in powder-type fire-retardant systems, the amount of dust associated with the dried product can pose significant problems such as lack of visibility and personal nuisance due to a high amount of air-borne dust particles when the material is applied as an insulation, particularly for people with sinus, asthma and other respiratory problems.

[0061] In embodiments, the reduced size cellulose material can be subjected to a dust removal (de-dusting) process. Such a dust removal (de-dusting process) is described in USP 9,045,605(Shutt). The de-dusting can be performed by any suitable process, for example, by screening, air classification, and/or other known separation techniques. In embodiments of a de-dusting process, the dried insulation product can be conveyed, for example by means of a conduit or chute to a screening apparatus, for example. In embodiments, the reduced size cellulose material can be processed to eliminate a major amount (i.e., at least 50% by volume or more) to substantially all (i.e., about 90-100% by volume) of the dust, and produce a low-dust fire-retarded cellulose fiber material. In embodiments, the de-dusting removes about 15 to 30 wt%, or 15 to 25 wt% of the overall weight of the final product. The de-dusted product can be characterized by a substantial absence of detached fibrous residue which, if present, can increase its density. In a screening process, for example, waste dust material will pass through the open mesh screening of the screening unit and into a collection unit (e.g., a bin, etc.) for disposal or further processing.

[0062] In embodiments, a cellulose material that has been processed using a liquid fire-retardant chemical, the reduced-size dry product will contain about 8-15 % by weight dust composed of about 7.25-14.75 % by weight cellulose-based (e.g., paper) dust with about 0.25-0.75% by weight of liquid fire-retardant chemical adhered to the dust, and de-dusting according to the invention can remove a substantial amount of dust (up to 100% of the dust) from the reduced-size dry product with substantially no loss of fire-retardant properties provided by the liquid fire-retardant chemical.

[0063] In an embodiment, the de-dusted, liquid fire retardant chemical treated cellulose material can be delivered into a metered delivery system (such as an auger) which meters the
material into an apparatus that generates a current of air, such as a fan or blower, etc. In embodiments, at the same time, powdered fire retardant chemical (or chemical composition) can be metered into the apparatus, i.e., using a device such as an auger.

[0064] In embodiments, nonlimiting examples of powdered fire retardant chemicals include hydrated borax, gypsum (dihydrate), Epsom salts (magnesium sulfate heptahydrate), and combinations thereof. In embodiments, from 5, or 8 or 10, up to 18, or 20, or 23 or 25 parts of powdered fire retardant is added per 100 parts of the de-dusted cellulose material. In embodiments, 5 to 25 parts of powdered fire retardant is added per 100 parts of the de-dusted cellulose material.

[0065] In embodiments, the de-dusted cellulose material and powdered fire retardant chemical (or chemical composition) can be mixed (e.g., pneumatically blended) within the apparatus for an effective time period such that the powdered fire retardant chemical (or chemical composition) uniformly coats the de-dusted cellulose material and adheres to the de-dusted cellulose material. In an embodiment, the de-dusted cellulose material and the powdered fire retardant chemical (or chemical composition) can be introduced into a mixing device that generates a current of air therethrough to blend the materials together.

[0066] In embodiments, the blended material is then discharged (e.g., blown) to a bagging apparatus as known and used in the art, and packaged for transport and future use. In embodiments, the resulting product contains an amount of the liquid fire retardant chemical and the powdered fire retardant chemical such that it meets government standards for fire-retarded insulation materials (e.g., a CRF value of 0.12 watts/cm² or greater, as measured in accordance with ASTM C-739).

[0067] In embodiments, adding powdered fire retardant pneumatically to the liquid fire retarded treated cellulose material after de-dusting the cellulose material adds a minor amount of dust to the final product, which is less than the amount of dust contained in the dried liquid fire retarded treated cellulose material before de-dusting.
**Additives**

[0068] In an embodiment, the powdered fire retardant composition contains one or more additives. In an embodiment, the powdered fire retardant composition contains one or more non-fire retardant additives.

[0069] In an embodiment, the powdered fire retardant composition can contain an additive, such as a pest control additive. In an embodiment, the cellulose material contains 1 to 5 wt% of a pest control additive, based on the total weight of the cellulose material. In embodiments, a pest control additive can be added in the bagger bin just before the cellulose material is bagged.

**Applications**

[0070] In an embodiment, the blended material is discharged (e.g., blown) to a bagging apparatus as known and used in the art, and packaged for transport and future use.

[0071] In use, in embodiments, the fire retarded insulation product can be placed into a hopper and mechanically fluffed up, and then "blown" in place directly into an attic, into stud spaces in an existing wall, and other applications.

[0072] In an embodiment, the fire-retarded cellulose fiber material contains, based on the total weight of the cellulose fiber material, from 95 to 80 wt%, or 94 to 80 wt%, or 93 to 80 wt%, or 95 to 88 wt% cellulose fibers comprising a liquid fire retardant chemical absorbed therein, from 2 to 20 wt%, or 5 to 19 wt%, or 5 to 18 wt%, or 5 to 16 wt%, or 5 to 12 wt% powdered fire retardant chemical (or chemical composition), and from 0 to 0.2 wt% surfactant.

[0073] Although specific embodiments have been illustrated and described herein, it will be appreciated by those of ordinary skill in the art that any arrangement which is calculated to achieve the same purpose may be substituted for the specific embodiments shown. This application is intended to cover any adaptations or variations that operate according to the principles of the invention as described. Therefore, it is intended that this invention be limited only by the claims and the equivalents thereof. The disclosures of patents, references and publications cited in the application are incorporated by reference herein.
**What is claimed is:**

1. A method of producing a fire-retarded cellulose fiber material, comprising:
   - applying a liquid fire retardant composition to a cellulose source material, the liquid fire retardant composition comprising water, phosphoric acid and optionally one or more additional inorganic acids other than phosphoric acid;
   - allowing the liquid fire retardant composition to permeate into the cellulose source material to form a liquid-permeated cellulose material;
   - drying the liquid-permeated cellulose material to remove the solvent, wherein an effective amount of the fire-retardant material remains in the cellulose source material, to form a dried, fire-retarded cellulose material;
   - processing the dried, fire-retarded cellulose material with a powdered fire retardant chemical or chemical composition to produce a reduced size, fiberized fire-retarded cellulose material; and
   - optionally, removing dust material from the fiberized fire-retarded cellulose material.

2. The method of Claim 1, wherein the liquid fire retardant chemical composition consists essentially of water, phosphoric acid, optionally one or more additional inorganic acids other than phosphoric acid, and optionally one or more additives.

3. The method of any of the foregoing claims, wherein the liquid fire retardant chemical composition consists essentially of water, phosphoric acid, sulfuric acid, and optionally one or more additives.

4. The method of any of the foregoing claims, wherein the powdered fire retardant chemical is selected from the group consisting of gypsum (dihydrate), Epsom salts (magnesium sulfate heptahydrate), and a combination thereof.

5. The method of any of the foregoing claims, wherein the reduced size, fiberized fire-retarded cellulose material comprises, based on the total weight of the material,
   - 80 to 95 wt% cellulose fibers comprising a liquid fire retardant chemical absorbed therein,
2 to 20 wt% powdered fire retardant chemical; and
0 to 0.2 wt% surfactant.

6. The method of any of the foregoing claims, further comprising, after removing the
dust material, adding a powdered fire retardant chemical or chemical composition to the
fiberized-fire-retarded cellulose material.

7. The method of any of the foregoing claims, wherein an amount of the powdered fire
retardant chemical or chemical composition is added to the cellulose fiber material to meet at
least one of ASTM C-739, EP Category B, and EP Category C.

8. A method of producing a fire-retarded cellulose fiber material, comprising:
   applying a liquid fire retardant composition to a cellulose source material, the liquid
   fire retardant composition comprising water, phosphoric acid, and optionally one or more
   additional inorganic acids other than phosphoric acid;
   allowing the liquid fire retardant composition to permeate into the cellulose source
   material to form a liquid-permeated cellulose material;
   drying the liquid-permeated cellulose material to remove the solvent, wherein an
effective amount of the fire-retardant material remains in the cellulose source material, to
form a dried, fire-retarded cellulose material;
   fiberizing the dried, fire-retarded cellulose material to produce a fiberized
   fire-retarded cellulose material comprising cellulosic dust material comprising the fire
   retardant absorbed in and adhered to the dust material;
   removing a major amount of said cellulosic dust material from the fiberized
   fire-retarded cellulose material by screening the cellulose fiber material to produce a low-
dust, liquid-fire-retarded chemical-treated cellulose fiber material; and
   adding a powdered fire retardant chemical or chemical composition to the low-dust,
   liquid-fire-retarded chemical-treated cellulose fiber material to produce the fire-retarded
   cellulose fiber material.
9. The method of Claim 8, wherein the liquid fire retardant chemical composition consists essentially of water, phosphoric acid, optionally one or more additional inorganic acids other than phosphoric acid, and optionally one or more additives.

10. The method of any of the foregoing claims, wherein the liquid fire retardant chemical composition consists essentially of water, phosphoric acid, sulfuric acid, and optionally one or more additives.

11. The method of any of the foregoing claims, wherein the powdered fire retardant chemical is selected from the group consisting of gypsum (dihydrate), Epsom salts (magnesium sulfate heptahydrate), and a combination thereof.

12. The method of any of the foregoing claims, wherein the reduced size, fiberized fire-retarded cellulose material comprises, based on the total weight of the material, 80 to 95 wt% cellulose fibers comprising a liquid fire retardant chemical absorbed therein, 2 to 20 wt% powdered fire retardant chemical; and 0 to 0.2 wt% surfactant.

13. The method of any of the foregoing claims, wherein an amount of the powdered fire retardant chemical or chemical composition is added to the cellulose fiber material to meet at least one of ASTM C-739, EP Category B, and EP Category C.
A. CLASSIFICATION OF SUBJECT MATTER

According to International Patent Classification (IPC) and to both national classification and IPC:

INV. D06M11/70  D06M11/55  D06M23/08  D06M11/56

ADD.

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols):
D06M E04B  D21H  C09K

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched:

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used):

EPO-Internal, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

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<td>US 4 595 414 A (SHUTT THOMAS C [US]) 17 June 1986 (1986-06-17) cited in the application example 1</td>
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Further documents are listed in the continuation of Box C.

See patent family annex.

Date of the actual completion of the international search: 14 December 2016

Date of mailing of the international search report: 02/01/2017

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Authorized officer: Fiocco, Marco
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