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MOLDABLE LIGNOCELLULOSIC MATERIAL AND METHOD FOR MAKING THE SAME

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This invention relates to improvements in the method of preparing moldable products from natural lignocellulose, and to the products thereof. In particular, it relates to the treatment of molding compositions composed of such lignocellulose to reduce the volume of a given weight thereof whereby it is better adapted for handling, shipping and storage.

Thermoplastic compositions which are adapted to be hot-pressed into hard, water-resistant, resin-like products may be prepared by cooking a natural lignocellulosic material, such as wood, with water at an elevated temperature and pressure whereby a portion of the lignocellulosic material, particularly the hemicelluloses, are rendered water-soluble, washing the cooked product with water, removing moisture and disintegrating the product to a powder. The cellulose and lignin of the lignocellulosic material are left substantially as they were originally, and the product is capable of being molded by hot-pressing into a hard, resin-like object, the cellulose acting as the filler and the lignin as the binder. Such molding powders are finely divided and bulky, occupying a large volume for their weight, and for this reason it is desirable that the volume of a given weight be materially reduced.

It is the object of this invention to provide a method by which the volume of a given weight of such lignocellulosic molding compositions may be very substantially reduced. In accordance with the invention it has been discovered that this may be done by subjecting the composition, preferably containing a moderate amount of moisture, to moderate heat and mechanical pressure, insufficient to cause it to coalesce by fusion into a resin-like product, as it does in the hot-molding operation.

The raw material for the molding composition may be any natural lignocellulosic material, such as wood, corn cobs, straw, bagasse, cornstalks, etc. Newsprint paper containing, for example, 70% to 90% of coniferous ground wood is also suitable, and when a natural lignocellulosic material is referred to herein, it includes manufactured products such as newsprint of this character.

In the preparation of the molding powder, the natural lignocellulosic material is first comminuted to a fine state of subdivision and is then cooked with water at an elevated temperature, temperatures between 160° and 200° C. being suitable, for a period sufficient to cause solution of the water-solubles, one hour or less usually being satisfactory. During the cooking operation acids are formed or released from the lignocellulose and it is advisable to neutralize these acids by the introduction, prior to the cook, of a small amount, from 2 to 5% of the air dry weight of the natural lignocellulose, depending upon the kind of lignocellulose used, of sodium carbonate or sodium hydroxide.

The alkali serves to neutralize the acids progressively as they are produced. If these acids are not neutralized, the strength of the finished resin-like product is impaired, although its plasticity is increased somewhat. Care should be exercised to avoid the use of too much alkali or the lignin may be attacked by the excess.

The molding properties, and the properties of the finished molded product may be varied by adding other materials to the cook. For example, the lignin, and hence the binder content, may be increased by adding to the cook lignin which has been obtained from other sources. An example of such source is the "black liquor" obtained in certain cellulose pulp cooking operations, and also the cooking liquor from alcohol cooks, such as butanol cook, used for the preparation of paper pulp. Aniline and similar aromatic amines may be added in limited quantities. Organic solvents for lignin, such as methyl, ethyl and other alcohols, methylene glycol, acetone, acetaldehyde, ethylpropyl ether and dioxan, also may be present during tahe cook. The strength of the molded product is improved thereby. Such solvents are removed as by distillation or evaporation after the cook is completed.

After the completion of the cook the lignocellulose is washed with water whereby the water solubles are removed, and it is then dried to a moisture content preferably less than 1%. The dried product, called the "primary material" is disintegrated to a powder by a suitable disintegrating mechanism. The properties of the final product may be improved by adding lignin to the primary material at this point. This may be done by a mechanical mixing operation or by adding lignin in solution in an organic solvent and then removing the solvent by evaporation.

The properties of the molding powder may be modified by adding other materials after the cooking operation and before molding. Phenols or organic amines may be mixed into the powder to increase the water resistance, plasticity and strength of the product during the final molding operation. Waxes, such as paraffin and

montan wax may be added and also drying oils, such as linseed and tung oil. In the same way a small amount of zinc stearate, for example, 1%, may be mixed with the molding powder to secure better release in the mold. The zinc stearate acts as a lubricant and also increases the water resistance of the final product. These materials may be added either before or after the pressure-densifying operation of this invention.

The resulting thermoplastic powder is very bulky, having an apparent density of about 0.2 grams per cubic centimeter. For handling, shipping and storage, such a large volume per unit of weight obviously is an inconvenience.

In accordance with this invention, a method has been provided by means of which this volume may be reduced to one-half or less. This may be accomplished by subjecting the primary material, either before or after it has been disintegrated to a powder, and while it contains a moderate amount of water, to an elevated temperature and mechanical pressure, such temperature and pressure being lower than that required to cause the powder to coalesce by fusion into a resin-like body.

In carrying out the method, the moisture content of the primary material is first adjusted to that which is suitable for the purpose. From approximately 5% to 50%, based on the air dry weight of the powder, is satisfactory. A higher proportion of moisture may be present, but the advantage of maximum densification is not obtained. The moist powder is then heated to a moderately elevated temperature, preferably between 50° and 100° C., and is subjected to a mechanical pressure of approximately from 1500 to 5000 pounds per square inch. The above temperatures are insufficient to cause the powder to coalesce by fusion into the resin-like form.

The compression may be applied by any suitable means such as a pair of cooperating heated platens, or a pair of heated rolls. The material may then be in the form of dense lumps or flakes, and may be dried and disintegrated again to a powder. If the moisture content before the compressing operation is substantially above 50%, maximum densification is not obtained. Such excess of moisture may be evaporated during the compressing operation, when satisfactory densification is realized.

In an example of a satisfactory method for carrying out the invention, the primary material resulting from the cook is dried to a moisture content of approximately 30%, and without disintegrating is fed upon the surface of a relatively large heated revolving roll, against the surface of which a number of smaller pressure rolls are arranged at spaced intervals. The temperature of the heated roll is such as to heat the material to approximately 85° C., and the material is carried by the roll beneath the pressure rolls in succession. It undergoes a crushing action, and in one revolution of the heated roll it undergoes the compression required to produce densification. It is then scraped from the surface of the roll by means of a suitable blade or doctor. It is obtained in the form of dense flakes or powder, and this may be dried and disintegrated further if desired to produce the final molding powder. Resins, or resin-forming materials, may be added to the primary material prior to the pressure-densifying operation. For example, the primary material may be moistened with a solution of a water-soluble phenol-formaldehyde resin, and then subjected to the compressing operation. In the same way the primary material may be moistened with an aqueous emulsion of a resin.

A resin emulsion may be prepared as follows. A mixture of 80 parts of cumar resin and 9 parts of stearic acid is heated until it becomes molten and is then added with agitation to a boiling solution of 3.5 parts of triethanolamine in 250 parts of water, and the agitation continued until the emulsion has cooled. Sufficient of this emulsion is added to the primary material to incorporate 5% of cumar, based on the dry primary material. After thorough mixing, sufficient aluminum sulphate is added to convert the stearic acid to aluminum stearate. The mass is then washed with water, dried to a moisture content of about 30% and is subjected to moderate heat and pressure as described heretofore.

As a further example, an emulsion is made by pouring a hot solution of Manila copal in ethyl alcohol with agitation into a hot solution of sodium stearate. This emulsion is added to the primary material with mixing, the subsequent steps being the same as in the preceding example.

The apparent density of lignocellulosic molding powders of the character described herein may be increased from approximately 0.2 gram to approximately 0.45 gram per cubic centimeter by the pressure-densifying method of this invention. Since the density of the molded product is approximately 1.40 to 1.45 grams per cubic centimeter, it is apparent that the pressure-densifying contributes a valuable advantage in reducing the volume of the powder. The properties for hot-molding purposes are not impaired by the densification.

The molding powder is plastic under hot-molding conditions. A small amount of moisture, usually 1.0% or somewhat more, present in the powder is advantageous since the powder is rendered more plastic and the moisture resistance is increased. The primary product may be molded at a pressure of 1600 to 5000 pounds per square inch, the pressure of 3000 pounds being a favorable one, while it is maintained at an elevated temperature, usually above 100° C. The favorable temperature is 185° C., though temperatures of over 200° C. may be used. The molding time should be sufficient to produce the desired hard and resinous properties, 2 to 15 minutes usually sufficing for small objects. After being subjected to the desired temperature and pressure as hereinbefore specified for the necessary time, the mold may be cooled while maintaining some pressure. Hot or cold ejection is used. The resultant product is hard, has a resinous appearance, remains thermoplastic and has many of the properties of products made by molding mixtures of a resin, such as the phenolformaldehyde resin known as Bakelite, and a filler such as wood flour.

I claim:

1. The method of treating a lignocellulosic molding composition to reduce the volume of a given weight thereof which comprises subjecting a moist mass of said composition to heat and mechanical pressure between opposed surfaces, said heat and pressure being insufficient to cause said composition to coalesce by fusion, and disintegrating said compressed material to a powder.

2. The method of treating a thermoplastic lignocellulosic molding composition to reduce the volume of a given weight thereof, which comprises moistening said composition with water containing a resin in solution or sus-

pension therein and subjecting said moistened composition to a temperature of approximately 50° C. to 100° C. and mechanical pressure between opposed surfaces, said heat and pressure being insufficient to cause said composition to fuse and form a hard resin-like body.

3. The method of treating a lignocellulosic molding composition formed by cooking a natural lignocellulosic material with water at a temperature and pressure sufficiently elevated to render said material thermoplastic and thereafter washing with water, which comprises adjusting the moisture content of said composition to approximately between 5% and 50%, subjecting said moist composition to mechanical pressure of from 1500 to 5000 pounds per square inch while maintaining the temperature thereof approximately between 50° C. and 100° C.

4. In the preparation of a thermoplastic lignocellulosic material capable of being hot-molded under pressure into a hard, water-resistant and resin-like product by cooking with water at a temperature and pressure sufficiently elevated to render it thermosplastic and thereafter washing with water and removing moisture, the step which comprises subjecting the moist thermoplastic product to a temperature of approximately 50° C. to 100° C. and mechanical pressure between opposed surfaces, said heat and pressure being insufficient to cause said product to coalesce by fusion.

5. In the preparation of a thermoplastic lignocellulosic material capable of being hot-molded under pressure into a hard, water-resistant and resin-like product by cooking with water at a temperature and pressure sufficiently elevated to render it thermoplastic and thereafter washing with water and removing moisture, the steps which comprise subjecting the moist thermoplastic product to heat and mechanical pressure between opposed surfaces, said heat and pressure being insufficient to cause it to coalesce by fusion, drying and disintegrating the compressed product to a powder.

6. A lignocellulosic molding composition comprising powdered thermoplastic lignocellulose having an apparent density of approximately 0.45 grams per cubic centimeter.

7. A lignocellulosic molding composition comprising a body of powdered thermoplastic lignocellulosic material having an apparent density substantially greater than 0.2 grams per cubic centimeter.

8. The method of treating a lignocellulose molding powder to reduce the volume of a given weight thereof which comprises subjecting a moist mass of said powder to a temperature of approximately 50° C. to 100° C. and mechanical pressure between opposed surfaces.

9. The method of treating a lignocellulose molding powder to reduce the volume of a given weight thereof which comprises subjecting said powder containing approximately 5% to 50% of its air dry weight of moisture to a temperature of approximately 50° C. to 100° C. and a mechanical pressure between opposed surfaces of from approximately 1500 to 5000 pounds per square inch.

10. The method of treating a lignocellulose molding powder to reduce the volume of a given weight thereof which comprises roll-pressing a moist mass of said powder while maintaining the temperture of said mass at approximately 50° C. to 100° C.

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