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PROCESS OF MAKING LIGNOCELLULOSE HARDBOARD

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This invention relates to a process of making fibrous lignocellulose products having improved physical properties, e. g. hardboards or sheets hereinafter designated as boards.

It is known in the art that impregnating fibrous lignocellulose boards with certain oils and baking the boards improves the properties of the boards, as measured for example by modulus of rupture. U. S. Patent #1,941,536 pertains to such subject matter. The present application is in the nature of an improvement of that patent.

It has been found according to the present invention in general that boards having very good properties are produced by first impregnating the boards with lignin, and then with siccative oil or other siccative material, followed by baking the boards.

While siccative materials in general are applicable, the invention will be described with special reference to the siccative material sold under the trade-mark Velsicol. Velsicol is comprised primarily of an admixture or copolymer of a mineral oil fraction (partially polymerized petroleum hydrocarbon having double bonds in conjugate arrangement) and a vegetable oil, e. g. linseed oil or soybean oil.

In practicing this invention according to a preferred embodiment thereof, lignocellulose material, e. g. wood chips from trees is explosively fibrated by discharge from a closed chamber, e. g. a gun as described in U. S. Patent 1,824,221. The gun process comprises charging the gun with relatively small wood chips or other woody material, subjecting the chips to pressure of about 200–1200 pounds per square inch and a temperature of about 200° C.–300° C. for approximately 30 minutes to 5 seconds, respectively, in the presence of steam, and then discharging the woody material from the gun through a constricted discharge opening therein, for example a valve, into a zone of lower pressure (preferably atmospheric pressure), thereby exploding the wood chips into comparatively finely divided elongated fibers and bundles of fibers, hereinafter called fiber. The gun conditions may be varied so as to be suited to changes in kind of wood, size of chips (desirably up to about one inch long), water content of the chips (preferably not exceeding about 30%), extent of desired steam treatment, and the like. Under the described treatment the wood is hydrolyzed and fibrated.

This hydrolyzed fiber is refined, screened and formed into a wet lap and hot pressed into a consolidated hardboard product according to conventional methods. Desirably there is a screen between the bottom face of the wet lap and the lower press platen during pressing to aid the escape of moisture from the wet lap.

According to the present invention, the resulting board is impregnated with lignin dissolved in an organic solvent. The amount of lignin may be up to about 5% or more by weight of the dry fiber. The solvent is evaporated leaving the lignin deposited throughout the board. Then the board is impregnated with siccative material, e. g. Velsicol. The amount of Velsicol used varies inversely with the amount of lignin used. Up to about 5% of the siccative material may be used; preferably about 2.5%–4.5% is used after impregnating with about 5%–2.5% of lignin. Finally the board is baked about 2–7 hours in the presence of air maintained at a temperature of about 200° C.–150° C., respectively.

Fibrous vegetable growths other than trees applicable to the present invention include bamboo, straw, cornstalks, grasses, canes, sedges, and the like. Methods of fibrating these materials other than by explosion may be employed. Preferably fibrating methods will be used

in which the non-cellulose constituents are largely retained in the fiber. For example fibration of these materials may be accomplished by grinding or by other mechanical means, by semi-chemical cooks, or by any combination of the above means and made into board products by conventional methods. Desirably the fiber will have been subjected to mild hydrolysis before hot pressing into boards. For this reason the gun process of fibration is preferred.

The following examples illustrate specific embodiments of this invention. All the starting boards were made under the same conditions from wood chips and according to the process disclosed in U. S. Patent 2,046,750. These boards were divided into three lots. Lot I boards were used as blanks and the properties determined without impregnating and baking. Lot II boards were impregnated with Velsicol alone, baked, and the properties determined on the baked boards. Lot III boards were impregnated with lignin in solution followed by impregnation with Velsicol after evaporation of the lignin solvent, baked, and the properties determined on the baked boards. All boards were allowed to stand at room conditions about twenty-four hours before testing. Impregnation was accomplished by immersing the boards in the impregnant. All boards were approximately ⅛" thick.

Example I

The lignin used in this example was extracted from gun fiber with methyl cellosolve and precipitated in water. This lignin was used in the form of a 25% solution in methyl cellosolve.

Lot No.	I	II	III
Impregnant	none	Velsicol	Lignin then Velsicol.
Percent impregnant uptake	do	5.3	8.3 (3.6 lignin, 4.7 Velsicol.
Baking time, hrs.	do	3	4.
Baking temp., ° C.		160	160.
Modulus of rupture (p. s. i.)	6,380	10,200	11,000.
Specific gravity	1.03	1.05	1.01.
Percent water uptake during 48 hrs.	19.8	13.4	16.4.

Example II

The lignin used in this example was extracted from gun fiber by washing the fiber with sodium hydroxide. On a dry weight basis, 100 parts of fiber per 66 parts of sodium hydroxide as a 3% solution was used. The extract was neutralized with acetic acid, centrifuged, the lignin washed with water and then dried. This lignin was used in the form of a 7.5% solution in methyl cellosolve.

Lot No.	I	II	III
Impregnant	none	Velsicol	Lignin then Velsicol.
Percent impregnant uptake	do	6.7	8.0 (3.1 lignin, 4.9 Velsicol).
Baking time, hrs.	do	3	3.
Baking temp., ° C.		160	160.
Modulus of rupture (p. s. i.)	5,400	8,280	7,690.
Specific gravity	.98	1.01	.96.
Percent water uptake during 48 hrs.	31.0	22.2	19.8.

Example III

The lignin used was obtained as that in Example II. It was used in the form of a 10% solution in dioxane.

Lot No.	I	II	III
Impregnant	none	Velsicol	Lignin then Velsicol.
Percent impregnant uptake	do	5.5	5.3 (2.6 lignin, 2.7 Velsicol).
Baking time, hrs.	do	3	3.
Baking temp., ° C.		160	160.
Modulus of rupture (p. s. i.)	5,000	9,500	9,000.
Specific gravity	1.01	1.04	1.03.
Percent water uptake during 48 hrs.	24.0	16.1	15.6.

Lignin applicable in this invention may best be described by the source of the lignin and the various methods of obtaining the lignin. In general lignin extracted from natural lignocellulose material, which material previously has been subjected to hydrolysis, or is subjected to hydrolysis during extraction is suitable.

In the manufacture of fiber boards from coarse lignocellulose fiber produced by exploding wood chips from a gun, solid fine material amounting to about 5%–8% of the total weight of fiber is produced as a result of the mild acid hydrolysis the fiber undergoes in the gun. This fine material adversely affects the properties of the boards and therefore is washed out of the fiber and dried. It is known in the trade as Benaloid. Benaloid has a very high lignin content. The lignin therein for use in this invention may be extracted with a substantially water-soluble organic solvent, followed by precipitating the lignin in water, or with an alkali (e. g. sodium hydroxide) and the like substances, followed by acidifying the alkaline extract until the lignin precipitates, and then washing the precipitated lignin with water. Suitable organic solvents include methyl cellosolve, dioxane; methyl, ethyl, propyl, and tertiary butyl alcohols; acetone; furfural. From an over-all standpoint methyl cellosolve and dioxane are preferred. Lignin extracted in like manner from the entire fiber product of the gun is substantially the same and equally applicable.

Other suitable lignins include lignin obtained by subjecting lignocellulose materials to an alkaline cook. Likewise lignin from the soda and sulfate paper making processes is well adapted for the purposes of this invention. The lignin can be extracted from the alkaline liquor by acidifying the liquor and washing the precipitated lignin with water.

Suitable solvents for preparing the lignin impregnating bath include alcohols: e. g. methyl, ethyl, propyl, and butyl alcohols; ketones: e. g. acetone, methyl ethyl ketone, diacetone alcohol; aldehydes: e. g. acetaldehyde, propionaldehyde, furfural; ethers: e. g. ethyl propyl ether, dioxane, and methyl cellosolve. From an overall standpoint dioxane and methyl cellosolve are preferred.

By the term "siccative material" as used herein is meant an oil or mixture of oils, a synthetic resin or resins dissolved in a solvent, or mixtures of said oils and resins which will bake into a hardened condition upon application of heat preferably in the presence of air. Suitable siccative oils include the conventional drying oils such as linseed, tung, oiticica, perilla, semi-drying oils such as soya bean oil, treated non-drying oils treated to improve drying properties such as treated castor oil, treated mineral oils such as petroleum polymers described in United States Patents 1,919,722 and 1,919,723 to Hyman. Suitable synthetic resins include cumarone, cumarone-indene and oil soluble phenolic resins or mixtures thereof in suitable solvents. The siccative oils used herein preferably are oils which contain a conjugated double bond structure (e. g. —c=c—c=c— or —c=c—c=c—c=c—) prior to impregnating the board or such oils which are converted to a conjugate structure during the baking process described herein. The oils may be treated to effect a partial shift to the conjugated structure before being used for impregnating the board and further shifted to such structure during the step of baking the oil-impregnated board. Conventional driers such as metallic salts, resinates or the like may be used with the oils to enhance the drying properties.

If the amount of siccative material used exceeds about 5%–6%, the possibility of encountering baking difficulties arises. Baking difficulties are particularly apt to occur if the siccative material used exceeds about 6%. These baking troubles show up by the siccative material forming a thin film or skin in the surface of the board during baking and excluding atmospheric oxygen from the inner portion of the board, thus preventing proper drying of the siccative material. Good boards under such conditions are difficult to obtain even under prolonged baking time. If the excess of siccative material is very large, baking may even decrease certain desirable properties of the board.

An outstanding advantage of this invention results from the discovery that such baking difficulties are eliminated and very good boards produced if lignin is substituted for a portion of the siccative material in the impregnant. Lignin is substituted for a portion of the siccative material to such extent that the amount of siccative material is below the amount which causes baking trouble, whereas the amount of total impregnant is much higher, as for example 8%–10%. A further advantage is that production of good boards with far less siccative material is made possible. An additional advantage is that lignin is much more economical than siccative materials. To a large extent lignin is somewhat of a waste material, particularly the lignin from the soda and sulfate papermaking processes.

As many apparently widely different embodiments of this invention may be made without departing from the spirit and scope thereof, it is to be understood that the invention is not limited to the specific embodiments thereof except as defined in the appended claims.

We claim:

1. Process of making an improved fibrous lignocellulose hardboard which comprises impregnating a fibrous lignocellulose hardboard with lignin dissolved in an organic solvent, evaporating the organic solvent from the board, impregnating the board with a siccative material consisting of a mixture of a vegetable oil and partially polymerized petroleum hydrocarbon containing conjugated double bonds, the combined weight of impregnants in the hardboard being not more than about 10% of the dry weight of the board, and then baking the impregnated board at an elevated temperature for a period of time not in excess of about 7 hours whereby the siccative material is completely polymerized and the modulus of rupture of the treated board is greatly increased.

2. Process of making an improved fibrous lignocellulose hardboard which comprises impregnating a fibrous lignocellulose hardboard with up to about 5% of lignin dissolved in an organic solvent, evaporating the organic solvent from the board, impregnating the board with about 5%–2.5% of a siccative material consisting of a mixture of a vegetable oil and partially polymerized petroleum hydrocarbon containing conjugated double bonds, both the lignin and the siccative material being based on the dry weight of the board and the quantity of each of said impregnants varying inversely with the other, and then baking the impregnated board at an elevated temperature for a period of time not in excess of about 7 hours whereby the siccative material is completely polymerized and the modulus of rupture of the treated board is greatly increased.

3. Process of making an improved fibrous lignocellulose hardboard which comprises impregnating a fibrous lignocellulose hardboard with about 2.5%–5% of lignin dissolved in an organic solvent, evaporating the organic solvent from the board, impregnating the board with about 4.5%–2.5% of a siccative material consisting of a mixture of a vegetable oil and partially polymerized petroleum hydrocarbon containing conjugated double bonds, both the lignin and the siccative material being based on the dry weight of the board and then baking the impregnated board at an elevated temperature for a period of time not in excess of about 7 hours whereby the siccative material is completely polymerized and the modulus of rupture of the treated board is greatly increased.

4. Process of making an improved fibrous lignocellulose hardboard which comprises impregnating a fibrous lignocellulose hardboard with about 2.5%–5% of lignin dissolved in an organic solvent, evaporating the organic solvent from the board, impregnating the board with about 4.5%–2.5% of a siccative material consisting of a mixture of a vegetable oil and partially polymerized petroleum hydrocarbon containing conjugated double bonds, both the lignin and the siccative material being based on the dry weight of the board, and then baking the board for a period of time within the range of about 2–7 hours in the presence of air maintained at a temperature of about 200° C.–150° C., respectively.

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