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PROCESS RELATING TO THE PRODUCTION
OF A MATERIAL FROM VEGETABLE MAT-
TER OF HIGH PENTOSAN CONTAINING
PENTO-CELLULOSE MATERIALSOrland R. Sweeney, Ames, and Charles
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The principal object of our invention is to provide a product from vegetable matter that is tough, hard, dense and possessing a high dielectric strength.

5 A further object of our invention is to provide a product of hard uniform structure that is economical in manufacture and durable in use.

These and other objects will be apparent to those familiar with the art.

10 The product which we will herein describe is easily distinguishable from such products as wood, which is not free from grain, knots, heart wood, and the constituents of sap. Our material is uniform throughout its entire structure, tougher
15 than the toughest wood, harder than the hardest wood, stronger in tension, compression, and transverse strength, and being free of texture is free from many blemishes which universally occur in wood.

20 Our product is particularly valuable to the mechanical, electrical and chemical industries, and because of its great strength and toughness, it is satisfactory for gears. Because of its high elasticity it is suited to the production of sport-
25 ing goods which at the present time are made of wood or plastic material. Excellent shuffle boards, dice, dominoes, and checkers have been made from this material. For buffer parts in mechanical devices this material seems almost
30 ideal being non-corrosive and not easily abraded. Its great strength renders it suitable for use as a substitute for metals in machine parts.

Our starting materials are the high pentosan containing pento-cellulose materials. By this
35 classification we mean such materials as corn-cobs, cornstalks, peanut shells, cotton stalks, cotton burrs, cotton seed hulls, oat hulls and the like. Also less satisfactorily cereal straws may be used. These materials are all characterized
40 by being high pentosan containing materials as distinct from such materials as spruce and other woods which are much lower in pentosan content. Such woods, however, are very high in
45 cellulose. Cotton fibers are also low in pentosan content, while on the other hand, they are very high in cellulose. Generally speaking, woods which are known to be low in pentosan materials and are thought to be low in the combination of
50 the pentosan and cellulose, which are combined in the chemical term and called "pento-cellulose" would be classed as low pentosan containing materials and naturally low pento-cellulose materials. Pure cellulose materials such as cotton or
55 like are easily distinguished from materials hav-

ing a high pentosan content. In carrying out our process we proceed as follows:

The high pento-cellulose material is charged into a large vessel after which it is covered with water and the vessel closed. We now introduce
60 steam and develop heat and pressure within the vessel or, if preferred, steam or other heating mediums such as oil, can be introduced into a jacket surrounding the vessel or in coils within the vessel. When the cooking is brought about in this
65 manner, it is obvious that the higher the pressure and corresponding temperature within the vessel, the quicker the cooking will be carried out.

Thus for example, if we maintain a steam pressure within the vessel of one hundred
70 pounds, the cooking is completed within approximately ninety minutes. Whereas, if the steam pressure is maintained at only forty pounds, three hours are required for the cooking. Such
75 steam pressure, the cooking, and the rapidity with which it is accomplished, also aid in destroying undesirable bacteria and fermentation.

We have found that the cooking is facilitated by stirring the mass, circulating the cooking li-
80 quor, or otherwise introducing a mild agitation within the vessel. In practice it would probably be simpler to design the cooking vessel in the form of a large steel ball which could slowly be rotated during the cooking period. This prac-
85 tice is standard for cooking other types of material such as the raw materials for wallboard manufacture. After cooking the material for the prescribed time under the prescribed pres-
90 sure, and resulting temperature, the product is dumped from the autoclave into a suitable receiving pit.

We have found it somewhat advantageous to use instead of a straight water cook, a cook con-
95 taining a certain percentage of alkali, such as sodium hydroxide or sodium carbonate. Obviously the other alkalies could be used, but since the sodium alkalies are cheaper, they will be preferred. An example of the cooking process is as follows:

Digest the cornstalks or like for ninety minutes
100 at one hundred pounds steam pressure, with ten percent of their bone dry weight of caustic soda diluted to a one percent solution. After having completed the digesting, the stalks or like are discharged and the cook liquors are drained off.
105 It is obvious that this process can be tied up with the well known process of recovering the alkali from the cook liquors by evaporating, treating with lime and filtering.

We have successfully practiced this procedure 110

for recovering the alkali with a corresponding reduction in the cost of the finished product.

The cooked material is now transferred to the second stage of the operation. This consists in
5 charging the pulp into a beating engine similar to the engines used in the paper industry. Attached to this beating engine is a Jordan machine similar to the machine used for Jordaning paper stock. Generally speaking, however, the
10 beating engine is run slower than in standard papermill practice, and the Jordan machine is run at a higher speed. It is difficult to actually specify the speeds of the machines respectively because such speeds will depend upon the size of the machines, but it is generally recognized that
15 for any given machine there is a rated speed as recommended by its manufacturer. The Jordan machine is recommended to run at the speed of 300 R. P. M. In the making of our product, however, we run this machine at 600 R. P. M., and
20 recommend even a higher speed if possible. In the case of the beater we run it slower than the speed for which it is specified. Generally speaking, the speed of the beater roll is approximately
25 50 R. P. M. The reason for this is that the cutting action in the Jordan is greater and the drawing out of the fiber in the beater is better and less air is likely to be incorporated into the product by this procedure.

The machinery is so set up that the pulp circulates under the roll of the beater, then through the Jordan machine and is discharged back in front of the beater roll. The product must be
30 drawn from the beater to the Jordan from underneath the surface of the pulp, and must be discharged back into the beating engine under the surface of the pulp to prevent the inclusion of air. After the pulp is charged into the beating
35 engine, the roll is set high, and a washer of standard design but quite fine mesh screen, is lowered into the pulp and operated until the pulp is washed sufficiently free from dirt and adherent gums contained in the cook liquor and from the caustic alkali where such is used. We
40 have found the optimum washing point is easily controlled by washing until the wash water shows neutrality to litmus.

At this point the washing is stopped. The washer is removed from the beating engine, the
50 addition of fresh water, which was previously added to complete the washing, is stopped and acid is added until the pH value of the water is 5. The beater machine is now put in connection with the Jordan.

At this stage the consistency of the pulp in the beater should be approximately six percent. The circulation is now begun under the beater roll and through the Jordan. The beater roll is lowered as rapidly as possible on the bed plate and the refining is continued until the whole mass
60 takes on a jelly-like consistency, and until the fibers are cut down to a point where microscopic measurements show the size of the maximum fibers to be approximately one-tenth millimeter
65 in length. This point is of great importance because it has been found that if the beating is not sufficiently great, the strength and other valuable properties of the material are greatly lowered. Whereas if this point is exceeded, the
70 strength and other properties likewise fall off. In other words, for the most valuable material the beating time should be held at the optimum. When the beating has been carried to this point, which point is not difficult to obtain in practice
75 due to the appearance of the material and its

microscopic examination, the beating should be stopped.

The theory of this operation is that the strength of the finished material depends upon the number of molecular contacts which are made between each particle of the material and its contiguous particle. We are aware of the fact that
80 papermill men are of the opinion that long fibers produce strong paper.

Theoretically, if fibers which are cut very short
85 or instead, fibers which are reduced to colloidal proportion are produced, the material should be much stronger, because when the film of water which surrounds the particle is evaporated away, the chance for molecular contacts is greater. Of
90 course, if great care is not used to prevent the formation of gas film around many of the particles, then obviously when the water film evaporates away, the gas film will prevent the molecular contacts. Regardless of the theory or papermill experience our experiences have shown
95 that this finely comminuted material has the greatest strength when the conditions prescribed above are reached.

While some of the fibers show the microscopic
100 length as described above, many of the fibers have been reduced to colloidal particles and the size of particles in the mass range from colloiddally dispersed particles to the maximum size described
105 above.

Just why continued grinding again weakens the mass is not altogether clear. The theory is that when the amount of colloidal material becomes
110 excessively large its tendency to absorb air is greatly increased and therefore a sort of dehydration sets in. Experimental evidence is at hand to show that oxycellulose is formed in increasing amounts as the fine grinding is continued. This indicates not only the absorption of air, but may
115 also indicate that oxycellulose has a weaker molecular structure or is less susceptible to hydration.

The advantage of the combination of a fast moving Jordan machine and a slow moving beater is that on the pass under the beating roll the
120 fibers are drawn into greater length than they had when they entered the beating roll, being longer and thinner, they are more readily cut on their next passage through the Jordan machine. However, we do not care to limit ourselves to this
125 type of machine for comminuting and hydrating the fibers. We have used successfully a number of other devices. A high speed modified burrstone machine in which the burrs were replaced by hardened steel discs has given successful
130 production. Certain modifications of the "colloidal mills" now on the open market have given satisfactory results. We have also found the first stages of the grinding can be advantageously
135 done in a rod mill and many other mechanical devices for arriving at the proper point of comminuting of this material can be devised.

After the material has been reduced to the proper degree of hydration and fineness, it is next
140 run into steam jacketed kettles or other devices for heating the material, where it is subjected to heat until sufficient water is evaporated to leave the material in a rather stiff plastic condition. In the handling of this very finely comminuted
145 material in the beater and in stock chest contiguous to the evaporators, we have found that the material readily ferments; this fermentation being brought about by bacteria, molds, or yeasts always floating around in the air. If fermentation sets
150 in, gas films are formed through the mass and

destroy the hydration of the material and greatly impair its desirable properties. To prevent this we have used a variety of materials such as sodium-silico-fluoride, sodium fluoride, furfural, formaldehyde, and phenols.

Our preferred materials are cresol, furfural, or formaldehyde because it has been found better not to introduce soluble salts into the material.

Accordingly, to all material stored in stock chests and preferably during the beating, a small amount, usually a tenth of one percent or less, of preservative should be added. The preservative not only assures a stronger product but prevents large fermentation blisters from forming in the product and thus gives a greater yield of usable material.

After the water is evaporated out of the mass and while the material is still hot, it is transferred to suitable molds. For example, if a block of this material is desired it will be charged into a rectangular mold. If a sample resembling a doughnut be desired, it will be poured into a mold of such shape. If the finished product is to be a disc, the product will be poured into a cylindrical mold. If the final product to be desired is a tube, the product will be poured or pressed into the annular space between two elongated concentric cylinders composing the mold. It is impossible, however, to mold this material accurately as is done with hard rubber or phenol condensation products. This material shrinks to about one-fifth the volume which it has when it is poured into the mold.

After the material is placed in the mold, in a short time, due to drying and water evaporation, the mass becomes stiff enough to remove from the mold so that the mold can be used over again, thereby saving in the cost of molds. The material, however, has no very great strength at this time but must be aged for a considerably longer period. This aging time depends roughly on the size of the sample.

For example, a disc which when completely seasoned is six inches in diameter and one-half inch thick, requires approximately fourteen days drying at seventy degrees centigrade, but if the material is to yield an absolutely accurate machined surface this aging should be continued for six months before it is machined, to prevent the possibility of a slight warpage of the machined surface.

After the article has dried and seasoned thoroughly it is now a rough product which, if the mold has been properly designed, has roughly the shape of the desired product. If this rough piece is now machined, sawed or otherwise shaped by suitable tools, a product with very accurate dimensions results.

In drying or seasoning this product, it is necessary to keep the temperature below one hundred degrees centigrade, otherwise the product is likely to check or crack. The optimum temperature is seventy degrees centigrade. A humid air seems to be a better medium in which to dry, than an air containing a low relative humidity. Generally speaking, usual atmospheric conditions are satisfactory in so far as humidity is concerned if the temperature of seventy degrees centigrade be not exceeded. After the piece has assumed considerable strength, that is, after a few days of seasoning, the material can be advantageously stored in any warehouse and allowed to age without any temperature or moisture control.

In the process of manufacturing this material into its finished products, it will as has been shown, be necessary to shape the piece to accurate dimensions after it is seasoned.

There results from this procedure a mass of turnings which may amount to two percent or more of the total material. Due to its great density, we have found this material suited to a great variety of processes. For example, if it is destructively distilled, it, like other dense vegetable materials, yields an activated charcoal. In the event no more profitable outlet for this material is available, it would be advantageous that it be used back in the process. By grinding this material finely and incorporating it back in the beater, preferably after soaking it for a time in water, we have found that the material can be worked up without seriously impairing the product. The incorporation of a percentage of this material back into the process also tends to give a finished product which shrinks less in seasoning.

We claim as our invention:

1. The process of manufacturing and producing a hard rigid product from vegetable matter of high pentosan containing pento-cellulose materials, such as corncobs, cornstalks, peanut shells, cotton burrs and stalks, consisting in the cooking of the pento-cellulose material in a liquid, comminuting the mass in a suitable machine, removing a portion of the liquid from said mass, and lastly the molding of the mass into a suitable shape.

2. The process of manufacturing and producing a hard rigid product from vegetable matter of high pentosan containing pento-cellulose materials, such as corncobs, cornstalks, peanut shells, cotton burrs and stalks, which consists in the cooking of the pento-cellulose material with water, breaking the mass into fine particles, heating the comminuted mass to remove a portion of the water, molding the material into a suitable shape, and lastly the aging of the said material.

3. The process of manufacturing and producing a hard rigid product from vegetable matter of high pentosan containing pento-cellulose materials, consisting in cooking the pento-cellulose material with alkali and water, breaking the mass into fine particles, heating the comminuted mass to remove a portion of the water, molding the material into a suitable shape, and lastly the aging of the said material.

4. The process of manufacturing and producing a hard rigid product from vegetable matter of high pentosan containing pento-cellulose materials, which consists in cooking the pento-cellulose material with water and under a pressure greater than the atmospheric pressures, breaking the mass into fine particles, heating the comminuted mass to remove a portion of the water, molding the material into a suitable shape, and lastly the aging of the said material.

5. The process of manufacturing and producing a hard rigid product from vegetable matter of high pentosan containing pento-cellulose materials, which consists in the cooking of the pento-cellulose material with water, injecting an antiseptic into the mass, breaking the mass into fine particles, heating the comminuted mass to remove a portion of the water, molding the material into a suitable shape, and lastly the aging of the said material.

6. The process of manufacturing and producing a product from vegetable matter of high pentosan containing pento-cellulose materials,

which consists in the cooking of the pento-cellulose material with water, subjecting the mass to a washing, adjusting the pH of the mass to approximately five, breaking the mass into fine particles, heating the comminuted mass to remove a portion of the water, molding the material into a suitable shape, and lastly the aging of the said material.

7. The process of manufacturing and producing a hard rigid product from vegetable matter of high pentosan containing pento-cellulose materials, which consists in cooking the pento-cellulose material in pure water, washing the mass after the cooking, the injecting of a preservative into the mass, comminuting the mass in suitable grinding and cutting machines, heating the comminuted mass to remove water, forming the resultant hot material into a suitable shape, and lastly allowing the material to dry and age.

8. The process of manufacturing and producing a product from vegetable matter of high pentosan containing pento-cellulose materials, such as cornstalks, corncobs, peanut shells, cotton stalks, cotton burrs, cotton seed hulls, oat hulls, cereal straws, consisting in the cooking of the material with alkali and water, washing the material until it is neutral to litmus, adjusting the pH of the mass to approximately 5, the grinding of the mass into minute particles, the injecting of formaldehyde into the mass, heating the comminuted mass to remove water, molding the material into a desirable shape, and lastly aging the material to produce the desired product.

9. The process of manufacturing and producing a hard rigid product from vegetable matter of high pentosan containing pento-cellulose materials, such as corncobs, cornstalks, peanut shells, cotton burrs and stalks, which consists in the cooking of the pento-cellulose material with water, the agitating of the mass during cooking, the breaking of the material into fine particles, heating the comminuted mass to remove water, forming the material into a suitable shape, and lastly aging the material at a temperature below one hundred degrees centigrade.

10. The process of manufacturing and producing a hard rigid product from vegetable matter of high pentosan containing pento-cellulose materials, consisting in the cooking of the pento-cellulose material in water for approximately ninety minutes at one hundred pounds steam pressure, approximately one percent solution of alkali in said cook, the discharging of the material from said cook, draining off of the liquors from the material, the recovering of the alkali from the drained-off liquors, the comminuting of the material into fine particles, heating the comminuted mass to remove a portion of the water, molding the material into a suitable shape, and lastly the aging of the said material.

11. The process of manufacturing and producing a hard rigid product from vegetable matter of high pentosan containing pento-cellulose materials, consisting in cooking the pento-cellulose material with water, discharging the material from the cook, subjecting the material to a suitable beating machine and a suitable cutting machine, the running of the said beating machine at a relatively low speed and the running of the said cutting machine at a relatively high speed for producing a maximum fibre length of 0.1 m. m., removing a portion of the water from the mass, heating the mass, forming the mass, and lastly permitting it to age.

12. The process of manufacturing and producing a hard rigid product from vegetable matter of high pentosan containing pento-cellulose materials, which consists in cooking the pento-cellulose material with alkali and water, the comminuting of the mass in a suitable machine until the longest particles are approximately one-tenth millimeter in length, draining of the water and alkali from the mass, heating the comminuted mass to remove additional water, forming the material into a suitable shape, and lastly the aging of the material at a temperature below one hundred degrees centigrade.

13. The process of manufacturing and producing a hard rigid product from vegetable matter of high pentosan containing pento-cellulose materials, which consists in cooking the pento-cellulose material with water, subjecting the same to washing, comminuting the mass in a suitable machine, heating the comminuted mass to remove water, forming the material into a suitable shape, aging the material to produce a solid member, shaping the member into the desired product by trimming, and returning the trimmed material to the process.

14. The process of manufacturing and producing a hard rigid product from vegetable matter of high pentosan containing pento-cellulose materials such as cornstalks, corncobs, peanut shells, cotton stalks, burrs, consisting in the applying of water to the mass and the subjecting of the mass to heat, breaking the mass into fine particles, draining off the water from the mass, further heating of the mass to remove an additional amount of water therefrom, molding the material into a suitable shape, and lastly the aging of the material.

15. The process of manufacturing and producing a hard rigid product from vegetable matter of high pentosan containing pento-cellulose materials, consisting of applying water to the mass and subjecting the mass to pressures higher than atmospheric pressures, breaking the mass into particles of not greater than one-tenth millimeter, removing a portion of the water from the mass, and lastly permitting the mass to harden into a rigid product.

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