June 18, 1946. C. C. HERITAGE 2,402,160

MANUFACTURE OF BITUMINOUS FIBER AND FIBER PRODUCTS

Filed May 13, 1940

Fig. 1

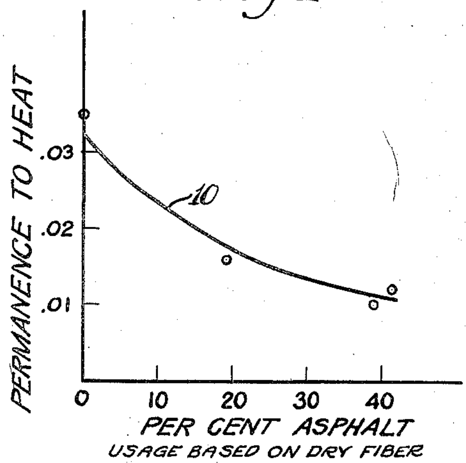


Fig. 2

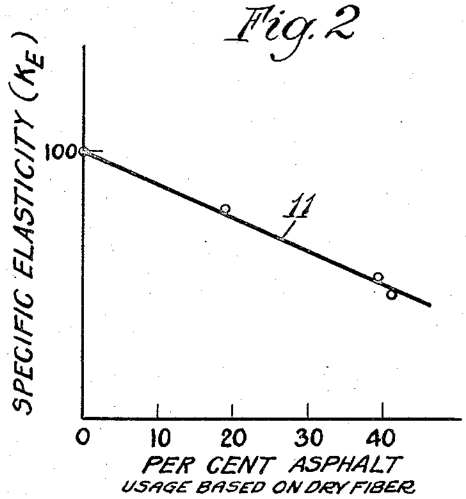


Fig. 3

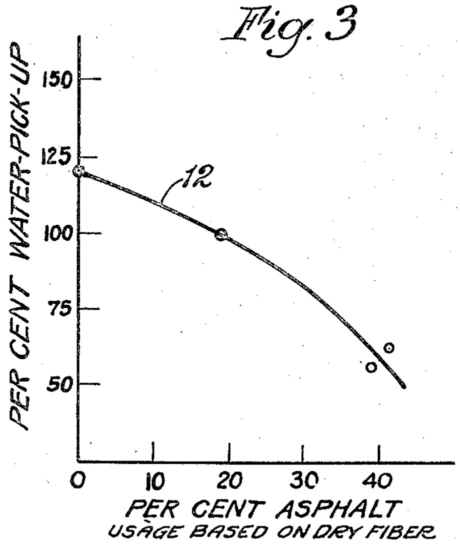
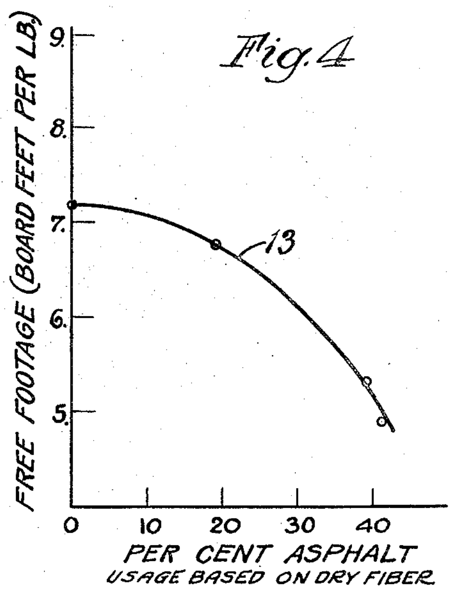


Fig. 4



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2,402,160

MANUFACTURE OF BITUMINOUS FIBER AND FIBER PRODUCTS

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17 Claims. (Cl. 92—6)

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The present invention relates to the manufacture of fiber and fiber products from vegetable matter. In particular it relates to the treatment of the vegetable matter with bituminous heat-plastic materials simultaneously with the formation of fiber, whereby a treated fiber is formed.

Heretofore, it has been proposed in Asplund U. S. Patent No. 2,047,170 to add sizing materials to wood or the like, which is subjected to disintegration at elevated temperatures in a gaseous atmosphere such as steam (as distinguished from suspension in water) by a rotary device of a hammer-mill type, wherein he may add up to 5% of fusible size. In his Patent No. 2,008,892, he describes a different type of machine which he says may employ the said process of his Patent No. 2,047,170. The present invention is directed to the use of heat-plastic and fusible bituminous materials with or without other agents to treat the fibers as the fibers are formed. It also relates to the subsequent treatments of the so-treated fiber to make products in which the bitumen and the treated fibers have a distinctive function.

An object of the invention is to introduce heat-plastic bitumens to the grinding means along with the raw material to be defibered.

Another object is to utilize the moist treated fiber directly to form useful products.

Another object of the invention is to dry the treated fiber so produced, directly, whereby all the substance of the treating agent is preserved and all the substance of the original vegetable matter is retained.

Still another object of the invention is to impregnate fiber from wood with a bitumen by a process which preserves all the content or all the water-insoluble content of the fiber resulting from defibering the wood in the absence of suspending water.

Another object of the invention is to provide a fiber having all the content of the wood providing it, which carries a heat-plastic bitumen, whereby the fiber may be thermoplastically treated or moulded to form an integrated product not lacking in solid constituents of wood.

Still another object of the invention is to provide an original fiber carrying bitumen, for use in a furnish for felting from water to form paper, boards and forms of high or low density, with increased water-resistance, increased strength and other improved and new properties.

Still another object of the invention is to use bitumen to alter certain characteristics of the fiber for felting and other uses.

A specific object of the invention is to use mixtures of bituminous material and a cutting agent therefor to facilitate the application of the bitumen to the fibers being formed.

The invention is illustrated only in part by reference to the accompanying drawing in which graphs show certain relationships later described in detail.

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In the drawing:

Fig. 1 is a graph relating permanence to heat and usage of asphalt.

Fig. 2 is a graph relating specific elasticity of a mass of fibers to usage of asphalt in producing said fibers.

Fig. 3 is a graph relating percent water pick-up to usage of asphalt.

Fig. 4 is a graph relating free-footage to usage of asphalt.

Among the common bituminous heat-plastic materials are the petroleum residues, referred to herein as asphalt, and the coal-tar residues, referred to herein as pitch. Of these asphalt is preferred for economy and for the lack of both odor and suspected harmful ingredients. Accordingly, the invention is explained with reference to the use of asphalt, without intent to limit the invention.

Reference is first made to the U. S. Asplund Patents No. 2,145,851, No. 2,008,892, and No. 2,047,170, relating respectively to a machine, the process of said machine, and the use of a size in said process and machine. The said patents are more particularly directed to the preparation of fiber from wood for the purpose of making board. To these ends the fiber is discharged from the machine, directly into water. Such operation subjects the material to agitation in the presence of water-soluble material, derived from the wood or added, tending to emulsify some of the bitumen, if present, whereby it is lost in the suspending water. The water also dissolves an appreciable content of the fiber.

According to one phase of the present invention the bituminous treated fiber is dried directly from the defibering machine, whereby all the content of the wood remains in the fiber, and none of the bituminous agent is lost. Such a fiber may be highly heat-plastic with large content of the bitumen. Such fiber has been made with as much as 80 parts of asphalt to 100 parts of wood (oven dry basis). It is readily subject to being moulded by heat and pressure, while originally moist, or drier, or dried, into forms of varied shapes, but more particularly into flat boards. Such boards are characterized by a content like wood plus asphalt, without loss of wood content, and have high strength, high water resistance, and other valuable properties.

According to another phase of the invention the so treated fibers are ready, immediately after formation, to be used as raw material for making bituminous papers and boards in the conventional processes of suspending in water, and then felting. Without necessarily hydrating the stock, as is conventional for paper and board formation, or without adding special binder, such as a bitumen or a bituminous emulsion, the bituminous-treated fibers are per se as formed, have a binding action when felted from water and dried with the aid of heat. Nevertheless, such fibers may be treated to the action of machines which normally hydrate untreated fibers, for the purpose of producing a more uniform stock, that is, to reduce fiber bundles or coarser elements of the stock to a smaller size. Incidentally, hydration may be effected, and may be controlled, to add to and to control the total binding effects in forming felts, paper and board.

According to another phase of the invention the fibers, dried directly from the machine, or wetted and dried, or washed with water and dried, may be used for bound or unbound felted masses, such as fibrous insulation. It has been determined that certain properties of fiber pertinent to use of it for bound or unbound loosely felted masses, are variable in a systematic order by increasing the content of asphalt or the like. It has also been determined that these variations follow specific rules, and do not for all properties parallel the changes made by substituting other materials. Two such other materials are herein indicated as B and C, for the purpose of showing the said specific influence, the change in properties having been compared in parallel series with B and C.

Another aspect of the invention concerns the use of the bitumen alone, and the bitumen mixed with an agent acting to cut it. For example, paraffin is a well known agent to smooth asphalt, to lower its melting point, and to lessen its tackiness, or to plasticize it. Such effects are comprehended within the term "cutting." This effect is well illustrated by parallel treatments with asphalt, with and without the addition of paraffin. Where fiber is produced by adding asphalt to the wood, the discharged fiber is grey to black. Where a small amount of paraffin is added, not necessarily combined with the asphalt for adding the mixture to the wood, the discharged fiber is blacker. The paraffin aids in the dispersion of the bitumen on the fiber as it is formed, and the impregnating action of it on the fiber.

With a content as high as 40 parts by weight of asphalt melting at 170° F. to 100 parts of oven dry wood, the moist fiber from the machine, when dried in the loose condition of discharge, or even with slight compression, does not have a self-binding action. At this 40% usage, which is 28.6% content, binding action is not exhibited until higher densities, dependent on temperature, are attained. For effective insulating felt densities, 40% usage gives fiber no self-binding property resulting from the bitumen. Thus, for loose rigid or flexible insulation felts (not as dense as rigid insulation board) bitumen may be used as a modifier for properties of the fiber, and not as a binder.

The addition of asphalt to the defibering machine may be done in any suitable manner. The important point is to introduce both the wood and the asphalt to the grinding disks simultaneously in the desired proportion. Considering that wood passes through the Asplund machine in about two minutes, more or less, during which it is heated and fiberized, it is important mechanically to control the introduction of bitumen to effect the result. In the particular machine, chips are rammed into the form of a plug by intermittent action, the end of which plug breaks off intermittently to fall into a preheating chamber from which fallen material is conveyed to the grinding disks. Use of finely powdered asphalt distributed throughout the wood causes the asphalt to melt too early and drain from the wood before it reaches the disks. This is remedied by using larger particles of asphalt mixed with the wood. Desired results are obtained by feeding with the wood, chunks of 170° F. melting point asphalt, about ¾ inch in diameter. However, it is preferred to inject molten asphalt into the preheater of the machine onto the chips already therein.

The bitumen may be introduced to the material before or after it enters the grinding elements, so long as it is introduced to the material prior to completion of the defibering action. With a stationary grinding plate, it may be introduced therethrough as a liquid, within the discharging periphery of the rotary plate.

The use of the asphalt of itself is of course preferred. However, the use of dispersions of asphalt, such as solutions or emulsions, is not excluded. Solutions in volatile solvents easily lose the solvent in the defibering process. Emulsions in water are permissible in low usage of asphalt, the amount of water added in the emulsion being the limiting factor. In high usage, so much water could be introduced that the discharged fiber is wetted and in clots. For many purposes this is undesirable. Also, many asphalt emulsions contain foreign agents such as clay, the introduction of which must be considered. For making board from water, the clay may be largely lost.

Felting Fiber

In order to explain first the easiest acquisition of fiber from the process, the following example of a felting fiber is given, in part purpose, to designate properties of felting fiber which may be influenced by the treatment of the present invention.

The fiber specimen of which the analysis and properties are set forth in Table 1, is made from jack pine wood and asphalt by the present invention. In my U. S. Patent No. 2,325,055, issued July 27, 1943, on my application Ser. No. 336,495, filed May 22, 1940, the method and means for determining the "coarseness modulus" (found in Table 1) are fully described.

Table 1

Kind of wood	Jack pine
Percent asphalt (M. P. 170° F.) (determined by extraction)	19.2
Particle size:	
Percent by weight 1st fraction	34.0
Percent by weight 2d fraction	19.6
Percent by weight 3d fraction	21.0
Percent by weight 4th fraction	16.6
Percent by weight past 4th fraction	8.8
Coarseness modulus (weighted summation of four fractions)	253.4
Compressive properties:	
Coefficient sliding friction	0.985
Elasticity at 3.15 board feet per lb. in lbs. per sq. ft	66.0
Absolute elasticity, an index	0.495
Felting at 3.15 board feet per lb. in lbs. per sq. ft	15.6
Absolute felting, an index	.326
Free footage, board feet per lb	6.78
Thermal conductivity B. t. u. per hr. per sq. ft. per 1° F. per inch	0.249
Friability	0.0036
Free dust, percent	4.0

The "compressive properties" above referred to are pertinent to making insulation or other loose felts, and are determined by the procedure set forth under the title "Practical procedure" in the copending application of Anway, Serial No. 313,920, filed January 15, 1940, now U. S. Patent 2,325,026, issued July 27, 1943.

The "impact resistance" is the density, expressed as its reciprocal value in board-feet per pound of fiber, to which it is finally compressed by repeated impacts of 1 foot-pound, effected by dropping a column of fiber to produce such impact. This is more particularly explained in another copending application of Anway, Serial No. 313,919, filed January 15, 1940, now U. S. Patent No. 2,375,182, issued May 8, 1945.

The free dust content, friability and heat-permanence are determined by the methods of still another copending application of Anway, Serial No. 290,999, filed August 19, 1939, now U. S. Patent No. 2,324,126, issued July 13, 1943.

Referring generally to the results of much work involving adding numerous agents in increasing proportion to the wood fed to the Asplund defibrator, and the tedious evaluation of the products, some generalizations have been drawn, to show the specific influence of asphalt, compared to the two materials B and C above referred to. The materials B and C are not named specifically herein as they are subject matter for other applications for patent. However, these specific influence of increasing quantities of asphalt on some properties is shown in the drawing, by way of illustration.

In the following series comparing materials: asphalt, B and C, it is explained that the statements of effects attributed to the materials are not absolute for all conditions. The properties compared are affected in part by variations of particle size distribution of the mass tested, and in part by other factors, for all the masses compared. However, conditions of setting a given machine and of feeding, were held constant. The presence of treating materials has some effect in changing the action of the machine so that a change in particle size distribution is one result of the treatment. The effects described are given merely to illustrate that the general effect of asphalt is specific when compared to the general effect of non-bituminous materials B and C.

Coefficient of Sliding Friction

Increasing asphalt increases the value. Material B also increases the value. Material C decreases the value.

Specific Elasticity

Increasing asphalt lowers the value. Material B decreases the value but is not very effective below 4% usage. Material C increases the value.

Absolute Elasticity

Increasing asphalt has little effect up to 40% usage tested in the series. Material B increases the value gradually up to 8% usage, and a usage above 8% gives a more rapid drop in the value. Material C gradually increases the value.

Free Footage

Increase in asphalt lowers the value. Material B lowers the value. Material C increases the value.

In this way other properties may be compared with the materials: asphalt, material B, and material C. A sufficient number of comparisons

have been given to illustrate the fact that the results in changing properties are dependent upon the character of the agent added. The materials B and C are not bituminous nor heat-plastic, although one melts and the other does not. Other effects are:

Felting (at 3.15 Board-Feet per Pound)

Asphalt increases the value up to about 20% usage, after which the value decreases sharply.

Absolute Felting

Up to 20% usage the asphalt increases the value. Beyond 20% usage the particular way in which the asphalt is present in the mat, affects the felting.

Water-Pick-Up

Increase of asphalt makes the fiber more water-repellent, and lowers the water content which it may pick up.

Hygroscopicity

Asphalt lowers the hygroscopicity.

Thermal Conductivity

Increased usage of asphalt raises the thermal conductivity.

Friability

Increased usage of asphalt lowers the friability.

Free Dust

Increased usage lowers the amount of free dust.

Some of the properties in the series above discussed are illustrated by graphs, merely to show the effect of bitumen upon measurable properties. In the drawing, Figs. 1 to 4 inclusive, respectively show the changes in heat permanence, specific elasticity, percent water pick-up, and free-footage, with increasing content of asphalt.

In all of the figures the horizontal axis shows the percentage of asphalt used on the basis of dry wood fed to the machine with asphalt, to be defibered. Line **10** in Fig. 1 shows permanence to heat, plotted vertically. This is a value defined in terms of friability before heating divided by the friability after heating for 24 hours at 150° C. The friability is determined by subjecting the fiber to a standardized mechanical pounding to break it into dust particles, according to the method set forth in said Anway Patent No. 2,324,126. It is shown that as the asphalt increases, the fiber is less permanent to heat.

In Fig. 2, line **11** indicates the specific elasticity (K_E) defined above. It appears that as the asphalt increases the fiber mass is less resilient.

In Fig. 3 the percent water pick-up when in contact with liquid water, is shown by line **12**. The fibers are less absorptive of liquid water as the asphalt content increases.

In Fig. 4, the free-footage, which is a property indicative of the bulk of the fibers, as defined in Anway U. S. Patent No. 2,325,026 referred to above, is shown by line **13** to decrease as the asphalt increases.

Hard Board

Hard board may be made in several ways from the asphalt treated fiber. Dry fiber may be matted and heated under pressure to make a board. Asphalt tends to squeeze out at the higher densities, so that the amount must be limited by the final density. At a density of about 65 lbs. per cu. ft. 20% usage of asphalt based on oven dry wood, shows exudation at forming pressures of about 215 lbs. per sq. inch and at a temperature of about 155° C. The amount of asphalt, therefore, is not critical to secure the binding action, but dependent upon conditions.

Another way to make board is to heat and press directly the moist fiber discharged from the machine. One difficulty is uniformity in formation due to unequal distribution of the moist fiber. This may be overcome by the third method.

A third method is to suspend the fiber in water, filter to form a mat, and then heat and press the formed mat. The disadvantage of this process is that water soluble material is extracted from the fiber, which serves otherwise as part of the binder. Boards made by such suspension procedure are weaker.

Combinations of these procedures are possible, such for example as suspending the asphalted fiber in water, with or without hydration, forming a lower-density mat, drying, and applying heat and pressure to the dry mat to make a higher-density mat, or by omitting the drying of the mat and pressing with heat.

In the following Tables 2 and 3 are given boards so made, which are identified by sample number. Table 2 gives the methods, and Table 3 gives the characteristics. S indicates a water-felt, dried, heated and compressed. M indicates moist fiber directly compressed with heat. The given percentages of asphalt are in terms of usage based on 100 parts by weight of dry fiber.

ods give fairly equivalent results as far as strength is concerned, but in general the water penetration is less by the moist-forming methods. It is clear that the use of asphalt greatly reduces the water penetration with increased usage, as well as reducing the swelling from water.

Other experiments have shown that higher temperature or higher pressures, or both, improve the board. Pressures up to 3000 pounds per sq. in. have been used, and temperatures up to 184° C. The temperature is not critical so long as it is above 212° F. to boil out the moisture in the board, and so long as it does not injure the board.

The press had a smooth metal plate on one side and a screen on the other side. In all the asphalt board samples above described, asphalt exuded on the screen side to leave a spotty asphalt film thereon. The water penetration tests were made on the smooth side, which is a dull black surface, with visual markings indicating a fibrous content.

Wet Board Process

The fiber-formation may be made uniform and be controlled by forming the mat for the board from a water suspension. The following detailed procedure will illustrate one method of doing this.

Wood such as jack pine or aspen or a mixture, in chip form is fed to the Asplund defibrator with asphalt in amount equal to 30% of the oven dry wood. The machine is adjusted to give a coarseness modulus of about 190 with about 15% content of the first fraction.

Table 2

Sample	Wood	Special treatment	S-Suspension M-Moist	Temp., ° C., in	Temp., ° C., out	Pressure lbs./sq. in.	Minutes in press
206-3	Jack pine	None	S	160	147	215	20
206-4	do	do	M	160	147	215	20
206-17	do	20% asphalt	S	161	145	215	20
206-18	do	do	M	161	145	215	20
206-19	do	40% asphalt	S	161	152	215	20
206-20	do	do	M	159	153	215	20
206-7	Aspen	None	S	160	150	215	20
206-8	do	do	M	159	153	215	20
206-35	do	20% asphalt	S	161	153	215	20
206-36	do	do	M	159	151	215	20
206-37	do	40% asphalt	S	161	153	215	20
206-38	do	do	M	161	155	215	20

Table 3

Sample	Treatment	Thick, in.	Lbs./cu. ft.	Hardness Shore C scale	Modulus of rupture	Swelling 24 hours per cent increase in thickness	Water penetration, gm/1000 sq. cm. per 24 hrs. smooth side
JACK PINE							
206-3	Untreated	.180	65	95	2,200	83.1	514
206-4	do	.225	69.7	95	3,300	46.8	215
206-17	20% asphalt	.177	69.8	94	2,400	18.8	81
206-18	do	.244	63.5	96	3,400	24.2	130
206-19	40% asphalt	.188	71.5	96	5,400	32.8	180
206-20	do	.218	72.2	96	4,200	25.4	81
ASPEN							
206-7	Untreated	.187	62.5	92	2,400	151.0	970
206-8	do	.228	69	93	2,000	88.6	754
206-35	20% asphalt	.180	71.2	95	1,800	109.0	454
206-36	do	.220	71.7	95	1,800	54.3	376
206-37	40% asphalt	.163	69.5	95	2,200	54.5	105
206-38	do	.177	66.9	90	2,800	66.6	178

Because the fiber-formation by the moist procedure is not readily controllable, the results do not correlate well to draw conclusions from the data above presented. The two formation meth-

The coarseness modulus relied upon is determined by use of a set of fractionating screens or perforated plates into which the fiber suspended in water is placed for dividing it into fractions.

The divisions and sizes are arbitrary for control purposes, and the one herein referred to as giving 190 coarseness modulus, is as follows:

1st fraction retained by ⅛ in. holes v%
2nd fraction retained by $\frac{3}{64}$ in. holes w%
3rd fraction retained by 40 mesh wire x%
4th fraction retained by 80 mesh wire y%
5th (past 4th) passes 80 mesh wire z%
Coarseness modulus $=4v+3w+2x+y$

See my said Patent No. 2,325,045.

The defibrator machine is supplied with steam at about 150 lbs. per sq. in. pressure giving an operating temperature of about 365° F. The asphalt (M. P. 170° F.) is melted at 350° F. and as a liquid is forced into the preheater where it mixes with the wood being fed to the grinding disks. The fiber is discharged into a small amount of water for feeding to a Bauer refiner operated to give a stock testing about 1200 cc. freeness (Greene freeness tester). This is a 10% consistency and approximately 900 parts water to 100 parts oven dry wood employed. The stock from the Bauer may be diluted, and is screw-pressed to remove water. This removes some water-solubles of the stock and may remove some asphalt in the Bauerizing process. The fiber is then conveyed in a minimum of water to one or more finely set Bauer machines to refine it to a freeness of 800 cc. (Greene). Thereafter the refined stock is diluted with water to a consistency of 3.5% in stock chests.

From the stock chests it may be made into board by any well known method. For the particular board being described, the stock in the chest is sized with a wax to the extent of 1.5 parts of wax to 100 parts of oven dry wood used. This may be done by any of the known methods such as precipitating a wax emulsion with alum.

The stock is then run onto a wire in a board-making machine, such as that combining the devices of Ziska in Patent No. 1,690,152, and Streeter No. 1,712,852, or onto a Fourdrinier type of machine. In the latter case the process as described in Frost No. 2,154,201 may be used, preferably omitting the surface roughening. A dry board of about ½ inch thickness may thus be obtained which contains about 28% to 30% of asphalt based on oven dry fiber content.

The formation is uniform and by the process it is controllable. The board is useful in providing a dry uniform felt for making a more dense board. By applying heat and pressure, the board softens and compresses to form a more dense asphalt-containing board of desired higher density. Compression may go so far as to cause asphalt to equal or exceed the volume of voids in the board. Thus, when exudation of asphalt is evidenced, the useful maximum density for the particular stock has been attained.

The ½ inch board described may also be used as a waterproof board for many purposes. A ½ inch board has been made as described, having a density of about 23 lbs. per cu. ft., weighs about 1000 lbs. per M sq. ft. It has a moisture content of 8% based on dry weight of board. Its properties are represented by the following Table 4.

The board may be further waterproofed superficially by impregnation. Materials which fuse readily penetrate and combine with the asphalt content of the board. For example, more of the same or special asphalts may be coated on. By applying a melted mixture at 300° F. of 90 parts of wood rosin and 10 parts of paraffin, at 30 pounds of the mixture per 1000 sq. ft., the surface is improved in its water-resistant properties. The material soaks in and does not appear on the surface. One use for such a surface-treated board is as a base for shallow metal cups in imitation of brick or tile. The board is grooved to a depth of about $\frac{1}{16}$ inch to define squares, rectangles or other shapes. Into the grooves the flanges of the metal cups or tile are fitted, and cemented, with or without cement on the surface of the board between the grooves.

The effect of asphalt in the board may be shown by comparing an asphalt board made as generally described above, which operation was continued with omission of the asphalt, merely for producing a board in which the operating variable was as far as possible the inclusion and omission of the asphalt. The following Table 4 shows two such boards in which the wood is jack pine, and in which specimen S 192–211 contains the asphalt used in the defibrator at 35% based on the dry weight of wood, while specimen S 192–208 omits the asphalt. In the table, columns 2 and 4 show the propertiy of the board of columns 1 and 3 respectively, after soaking in water for 24 hours, freezing for 24 hours, thawing, oven-drying, and standing to recover equilibrium with normal surroundings.

Table 4

Property or condition (units)	S 192 211 no asphalt		S 192–208 asphalt	
	1	2	3	4
Columns 1 and 3 (board as made)	x	------	x	------
Columns 2 and 4 (board after "wet-dry" cycle)	------	x	------	x
Thickness, inches	0.536	------	0.488	------
Density—air dry, lbs./cu. ft.	17.9	------	24.4	------
Density—bone dry, lbs./cu. ft.	17.0	------	23.7	------
Hygroscopicity (per cent water at equilibrium):				
93% relative humidity—75° F	16.8	------	13.3	------
93% relative humidity—55° F	15.6	------	12.1	------
55% relative humidity—75° F	10.6	------	7.6	------
55% relative humidity—55° F	9.22	------	8.1	------
Wet expansion:				
Machine direction, percent	0.0035	------	0.0055	------
Across machine direction, per cent	0.0035	------	0.0047	------
Wet swelling (per cent increase in caliper)	11.4	------	6.68	------
Transverse breaking load, pounds	29	24.5	41	48
Load at elastic limit, do	16	14.9	19	23
Modulus of rupture, lbs./sq. in.	308	243	518	577
Modulus of transverse strength, lbs./sq. in.	170	148	240	276
Modulus of transverse elasticity, lbs./sq. in.	9,610	7,290	15,620	15,025
Transverse deflection at rupture, inches	0.63	0.56	0.92	.085
Transverse deflection at elastic limit, inches	0.20	0.22	0.19	0.22
Compressive strength (lbs./sq. in. at elastic limit)	128.0	120	182	118
Compressive deformation (inches at elastic limit)	.123	0.151	0.100	0.081
Compressive modulus, lbs./sq. in.	557	447	887	709
Thermal conductivity (B. t. u./hr./sq. ft./1″/1° F.)	.313	------	.394	------
Thermal conductance (B. t. u./hr./sq. ft./1° F.)	.564	------	.809	------
Surface harndess (Shore durometer A/B/C)	69/55/48	------	78/59/50	------
Water penetration—Surface (gms./M sq. cm./24 hrs.)	36	40	22	44
Fire resistance (wt. loss in per cent	38.2	------	5.0	------
Minutes to burn thru	31.8	------	34.3	------

It has been found that when freshly made board is evaluated, and then aged and again evaluated, the desirable properties improve. Its strength properties in particular improve. For example, in one case where the board was aged for 38 days, the breaking load increased 34.5%; the modulus of rupture increased 34.5%; board-moisture content increased 149%; the moisture absorption decreased 67.6%.

This indicates that the freshly made board is slow to reach an equilibrium condition. The changes in moisture properties indicate that the moisture equilibrium may play an important part in the strength characteristics of the board.

Other Uses and Products

The moist treated fiber from the defibering machine may be baled for shipment for subjecting it to hot pressing, or for taking it up in water for forming felts, or for the purpose of further refining. The moist treated fiber from the defibering machine may be supplemented by sufficient water to permit refining it, that is, to break up the bundles with or without hydration, and the refined fiber may be provided as wet laps for shipment. Such wet laps may be hot pressed directly, shredded and reformed, and variously treated as herein described for the product direct from the machine or from the refiners.

The invention is not limited to use of the Asplund machine. That machine is, however, highly efficient and economical, with a rapid rate of production, increasing with the increased temperature. Other devices of equivalent action which grind wood or the like at elevated temperatures are useful for the present invention, where water to suspend the fiber is absent during the grinding or defibering and wherein the machine has a mixing action to disperse the bitumen on the fibers as formed.

The invention is not limited to use of raw wood in chip form, although that is the preferred economic raw material. Chemicals, such as normal sodium sulfite, or caustic soda may be added with wood chips to effect a degree of chemical softening or other action in the process.

Chips which have been preliminarily cooked, as in hot water, or in normal sodium sulfite, or other materials, to soften or alter them, without destroying the lignocellulose character of the material, may be used. Mixtures of these forms may be used, and pulp-mill screenings added to make economic utilization of the fiber content. Various straws and grasses, or lignocellulose of annual growth, such as cane, flax straw, or corn stalks, may be used alone or in admixture with wood or other lignocellulose forms. In the accompanying claims, the term "lignocellulose" comprehends these various materials as well as wood, but wood is specifically preferred and specified.

It is well known that wood and other lignocellulose, dry or moist, at elevated temperatures exhibits a plastic effect due to a thermoplasticity of lignin content. It is a feature of the present process that when the fiber has plastic lignin, the bitumen is fused to it. The resulting fiber is therefore a fiber with a bitumen coat fused to a thermoplastic constituent of the fiber.

Many omissions and changes may be made in the above detailed operations, where there are different objectives. For example, the bitumen-containing fibers derived from the fiber-forming process, may be mixed with other fibers or fibrous material not carrying bitumen. Such fibers need not be wood fibers, but may be paper pulp, ground wood, cotton fibers, rag stock, waste-paper such as newsprint or other paper, or any kind of fiber or fibrous material, to produce mixtures wherein the bitumen introduced with the fiber, may exert a function. Where wastepaper is used, it need not be as fiber, where the mixture is to be refined, as in a beater. Where all the stock is present as fiber, the beating or other refining may be dispensed with.

For example, a slurry of the bitumen-carrying fibers, alone, or including other fibers such as paper pulp, or rag stock, or both, may be felted to provide a saturating felt for the manufacturing of roofing and like products. Such dry saturating felts are impregnated with asphalt and other compositions to produce roofing material, shingles, flooring and the like. The presence of asphalt in the felt, and of already-asphalted-fibers in the felt, renders the saturating felt more easily penetrated by and absorbent of the numerous impregnating compositions.

Heretofore, attempts to make saturating felts entirely or largely of wood fiber or lignocellulose in substantially undegraded form, have not been successful because of the brittleness of the fiber, and resistance to impregnation. It has been accepted heretofore that such wood fiber cannot be used in excess of 50% to 60%, and that softer and more absorbent fibers such as rag stock or paper pulp are necessary as diluents for successful saturating felts.

By using the bitumenized-formed-fiber of the present invention for saturating felts, a much greater percentage than 60% of wood fiber may be utilized in saturating felts. For example, 60% of non-bitumenized wood fiber may be employed, and then beyond this point the bitumenized-formed-fiber may be used with or without paper pulp or rag stock up to 100%. And of course some or all of the said 60% may be the bitumenized-formed fiber. Such fiber is already impregnated with asphalt, and it offers no resistance to the impregnating treatment. Such fiber is also less brittle, especially as the temperature is raised, so that varying degrees of flexibility of the saturating felt may be obtained by controlling the temperature and the content of the bitumenized-formed-fiber.

While it is possible as far as fiber quality is concerned to make a felt which is substantially all lignocellulose or wood fiber, for practical purposes it is desirable to use some hydrated fibers to provide a bond for the felt to facilitate handling the same. Up to 20% of hydrated fibers may be present for this purpose, and where this amount is not exceeded, the felt is considered to be substantially all wood fiber or lignocellulose fiber. The fiber for hydration may be wood fiber, other lignocellulose fiber, the bitumenized-formed-fiber, cotton fiber, rag stock, or the various forms of cellulose or semi-chemical pulp which is commonly hydrated in making a paper furnish. The necessity for such paper-maker's bond may be avoided by sufficient bitumen content to provide bond where the felt is heated to activate it. As shown above, the lower ranges of bitumen in the treated fiber do not present a bond at low density felts even when heated.

Although the invention is illustrated by reference to the preferred machine for producing and treating the fibers, namely the Asplund machine, it is to be understood that the invention is not limited to such machine, or to the process thereof whereby the reduction to fiber is effected in a steam pressure chamber. That is preferred for the reason that the process as to fiber is rapid and economical, and because the higher temperature permits use of higher melting-point substances. Thus may be used special asphalts melting well above 212° F., and for example in the range from 256° to 316° F., which soften in the range from 266° to 298° F., and which are substantially non-tacky at about 140° F. Such asphalts are very valuable for saturating felts for roofing, or board for siding on buildings, where there is exposure to sun. The British Patent No. 15,105 (1911), shows a similar process where atmospheric steam pressure is used. Schouten U. S. No. 1,367,895, shows a similar process. Both of these may operate upon raw wood. Also, there are other machines and processes which are suitable. There are also the machines and processes of "Respats, Inc." wherein wood chips are steamed to soften them, then mechanically ground by a heavy roller operating upon them in a suitable dished receptacle. Asphalt or other bitumen may be added to the softened chips and as the fibers are formed, be distributed on the resulting fibrous product. The Respess U. S. Patent No. 1,976,279 illustrates such a machine.

The utility of the originally moist bitumenized fibers is far more extensive than above set forth. It has utility in many directions adding insufficient water to produce a continuous liquid phase, and without adding any water at all. It has other uses and forms resulting from adding water to produce a continuous liquid phase. The following will illustrate:

Moist-Bitumenized Fiber

The fiber may be marketed moist or dry either in bulk or after baling. It may be subjected while moist to well known refining machines, such as rolls or rod mills to further reduce the coarser sizes of the mass, and the product may be marketed moist or dry, in bulk or bales.

The original or the moist-refined fiber may be felted from air suspension, by gravity dropping or conveyance in air currents. So felted it may have a density from less than 1 pound per cu. ft. to 10 or more pounds, according to the process and to the form of the fiber. Adhesive may be used by spraying a liquid into the carried or depositing fibers. The liquid may be such as to cause the bitumen content to become adhesive, or it may carry dissolved adhesive. Mats deposited without adhesion, may be heated, or heated and pressed, to activate bitumen to adhesiveness. Bulky mats may be retained in light flexible form as blankets, as for insulation. More densely formed mats, or compressed mats so formed may be made into paper or boards or moulded forms not of sheet form.

Water-Bitumenized-Fiber

Bitumenized fiber to which water has been added to form a continuous liquid phase, permits of other ways of treating the fiber, and of forming masses therefrom. Some water soluble material may enter the liquid phase, and be removed in removing some of the liquid phase. Thus, fibers heavy with water, or suspended, may be screw pressed or formed into laps. The product, wet or dry, may be marketed. Suspensions may be filtered to paper, board, or special forms, such as dishes or containers, like slurries of other pulps, and the products used or sold, wet or dry. Such forms wet, may be dried with heat, or heat and pressure, to activate the bitumen into binding or impregnating, or filling relation to the fibers. Likewise, the dry felted forms may be so treated. Such dry forms may be merely heated without pressure.

Bitumenized fiber heavy with water, or suspended in water, may be refined in the presence of such water with more or less hydration as desired. This refined fiber may be treated as described in the preceding paragraph, with the additional benefits from hydration, and with the additional uniformity resulting from the refining.

In the foregoing, it is to be understood that the disclosures contemplate working with fibers all of which have passed through the bituminizing process, as well as working with such fibers which are diluted with fibers not so treated, for example as referred to above in making saturating felts.

The present invention is a continuation in part of my copending application Serial No. 227,338, filed August 29, 1938, wherein the use of Gilsonite, the most pure natural bitumen, is disclosed in accordance with the invention herein described and claimed.

I claim:

1. The method of producing fibers of lignocellulose which comprises heating lignocellulose in undefibered form in a gaseous environment containing water vapor at an elevated temperature above 212° F. and at which the lignocellulose becomes soft and plastic, while rubbing and pressing said plastic lignocellulose in said gaseous environment to reduce it substantially all to individualized ultimate fibers, while distributing into the materials being so defibered a heat-softened composition predominating in bituminous heat-plastic material selected from the group consistinging of bitumens, petroleum asphalts and coal-tar pitches, while feeding said undefibered lignocellulose substance to a space between and within the periphery of relatively rotating facially spaced elements including at least one circular element operating at high speed, and while centrifugally discharging peripherally from said space between said elements a loose moist mass of coated fibers carrying said composition, there being employed and carried by said fibers by weight from a minimum of about 20 to a maximum of about 80 parts of such heat-softened composition to 100 parts of oven-dry lignocellulose.

2. The method of producing fibers of lignocellulose which comprises heating wood in undefibered form in a gaseous environment containing water vapor at a temperature elevated above 212° F. and at which the lignocellulose of the wood becomes soft and plastic, while rubbing and pressing said plastic lignocellulose in said environment to reduce it substantially all to individualized ultimate fibers, while distributing into the material being so defibered a heat-softened composition predominating in bituminous heat-plastic material selected from the group consisting of bitumens, petroleum asphalts and coal-tar pitches, while feeding said undefibered lignocellulose substance to a space between and within the periphery of relatively rotating facially spaced elements including at least one circular element operating at high speed, and while centrifugally discharging peripherally from said space between said elements a moist mass of fibers carrying said composition. there being employed and carried by said fibers by weight from a minimum of about 20 to a maximum of about 80 parts of such heat-softened composition to 100 parts of oven-dry lignocellulose.

3. The method of producing fibers of lignocellulose which comprises heating lignocellulose in undefibered form in a gaseous environment containing water vapor at a temperature elevated above 212° F. and at which the lignocellulose becomes soft and plastic, while rubbing and press-

ing said plastic lignocellulose to reduce it substantially all to individualized ultimate fibers, while distributing into the material being so defibered a heat-softened composition predominating in bituminous heat-plastic material selected from the group consisting of bitumens, petroleum asphalts and coal-tar pitches, while feeding said lignocellulose substance to a space between and within the periphery of relatively rotating facially spaced elements including at least one circular element operating at high speed, and while centrifugally discharged peripherally from said space between said elements a loose moist mass of fibers carrying said composition, there being employed and carried by said fibers by weight from a minimum of about 20 to a maximum of about 80 parts of such heat-softened composition to 100 parts of oven-dry lignocellulose, and drying the moist fibers to provide dry treated fibers while maintaining the fibers in a loose mass.

4. The process of claim 1 followed by refining the resulting fiber thereof in the presence of water, utilizing the refined fiber in a felt-making slurry, and felting fibers from said slurry, the heat-bonded union of the applied composition to the fibers resulting from the process of claim 1 minimizing the tendency of the composition to be emulsified and removed from the fiber into attendant water, whereby said applied composition is largely preserved in the resulting felt.

5. The process of claim 1 followed by refining the fiber as a slurry in water, and felting fibers from said slurry, the heat-bonded union of the applied composition to the fibers resulting from the process of claim 1 minimiizng the tendency of the composition to be emulsified and removed from the fiber into attendant water, whereby said applied composition is largely preserved in the resulting felt.

6. The process of claim 1 followed by subjecting the resulting fiber thereof to heat and pressure while softening the applied composition of adhesiveness and while uniting the compressed heated fibers by said adhesiveness.

7. The process of claim 3 followed by subjecting the resulting dry treated fiber thereof to heat and pressure while softening the applied composition to adhesiveness and while uniting the compressed heated fibers by said adhesiveness.

8. The method of producing fibers of lignocellulose which comprises heating wood in undefibered form in a gaseous environment containing water vapor at a temperature elevated above 212° F. and at which the lignocellulose of the wood becomes soft and plastic, while rubbing and pressing said plastic lignnocellulose to reduce it substantially all to individualized ultimate fibers, while distributing into the material being so defibered a heat-softened composition comprising a predominant quantity of petroleum asphalt and a small quantity of paraffin as a cutting agent therefor, while feeding said undefibered lignocellulose substance to a space between and within the periphery of relatively rotating facially spaced elements including at least one circular element operating at high speed, and while centrifugally discharging peripherally from the space between said elements a loose moist mass of fibers carrying said composition, there being employed and carried by said fibers by weight from a minimum of about 20 to a maximum of about 80 parts of such heat-softened composition to 100 parts of oven-dry lignocellulose.

9. The process of claim 1 followed by refining the resulting fiber thereof as a slurry in water, felting fibers from said slurry, the heat-bonded union of the applied composition to the fibers resulting from the process of claim 1 minimizing the tendency of the composition to be emulsified and removed from the fiber into attendant water, whereby said applied composition is largely preserved in the resulting felt, and drying the resulting felt while heating to soften the composition to adhesiveness to bind the resulting dry felt.

10. Treated fiber resulting from the process of claim 1.

11. Treated wood fiber resulting from the process of claim 2.

12. Dry treated fiber resulting from the process of claim 3.

13. The product obtained by molding under adhesive-activating heat and mechanical pressure, dry fibers produced by the process of claim 3.

14. The method of producing fibers of lignocellulose which comprises heating lignocellulose in undefibered form in a gaseous environment containing water vapor at an elevated temperature above 212° F. and at which the lignocellulose becomes soft and plastic, while rubbing and pressing said plastic lignocellulose in said gaseous environment to reduce it substantially all to individualized ultimate fibers, while distributing into the material being so defibered a heat-softened composition predominating in bituminous heat-plastic material selected from the group consisting of bitumens, petroleum asphalts and coal-tar pitches, while feeding said undefibered lignocellulose substance to a space between and within the periphery of relatively rotating facially spaced elements including at least one circular element operating at high speed, and while centrifugally discharging peripherally from said space between said elements a loose moist mass of fibers carrying said composition, there being employed and carried by said fibers by weight from a minimum which is in excess of 5 to a maximum of about 80 parts of such heat-softened composition to 100 parts of oven-dry lignocellulose.

15. The method of producing fibers of lignocellulose which comprises heating lignocellulose in undefibered form in a gaseous environment containing water vapor at a temperature elevated above 212° F. and at which the lignocellulose becomes soft and plastic, while rubbing and pressing said plastic lignocellulose to reduce it substantially all to individualized ultimate fibers, while distributing into the material being so defibered a heat-softened composition predominating in bituminous heat-plastic material selected from the group consisting of bitumens, petroleum asphalts and coal-tar pitches, while feeding said lignocellulose substance to a space between and within the periphery of relatively rotating facially spaced elements including at least one circular element operating at high speed, and while centrifugally discharged peripherally from said space between said elements a loose moist mass of fibers carrying said composition, there being employed and carried by said fibers by weight from a minimum which is in excess of 5 to a maximum of about 80 parts of such heat-softened composition to 100 parts of oven-dry lignocellulose, and drying the moist fibers to provide dry coated fibers while maintaining the fibers in a loose mass.

16. Treated fiber resulting from the process of claim 14.

17. Dry treated fiber resulting from the process of claim 15.

CLARK C. HERITAGE.

Certificate of Correction

Patent No. 2,402,160. June 18, 1946.

CLARK C. HERITAGE

It is hereby certified that errors appear in the printed specification of the above numbered patent requiring correction as follows: Column 3, line 3, after the word "fibers" strike out "are"; column 10, line 24, for "propertiy" read *property*; same column, lines 30 to 35, Table 4, for that portion of the heading reading

S 192 211 no asphalt		S 192-208 asphalt	
1	2	3	4

read

S 192-211 No asphalt		S 192-208 Asphalt	
1	2	3	4

column 12, lines 10 and 11, for "manufacturing" read *manufacture*; column 15, line 35, claim 5, for "minimiizng" read *minimizing*; column 15, line 42, claim 6, for "composition of" read *composition to*; and that the said Letters Patent should be read with these corrections therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 3rd day of September, A. D. 1946.

[SEAL]

LESLIE FRAZER,
First Assistant Commissioner of Patents.