June 26, 1951 R. R. OLIVER 2,557,993

PROCESS OF REMOVING MINERAL OIL FROM VEGETABLE FIBERS

Filed July 30, 1948

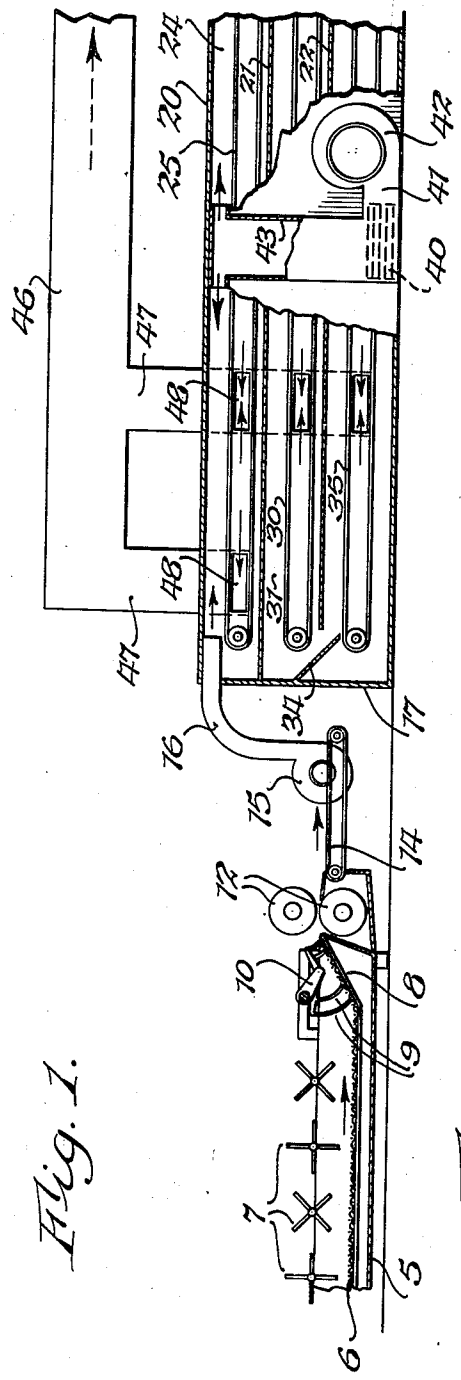


Fig. 1.

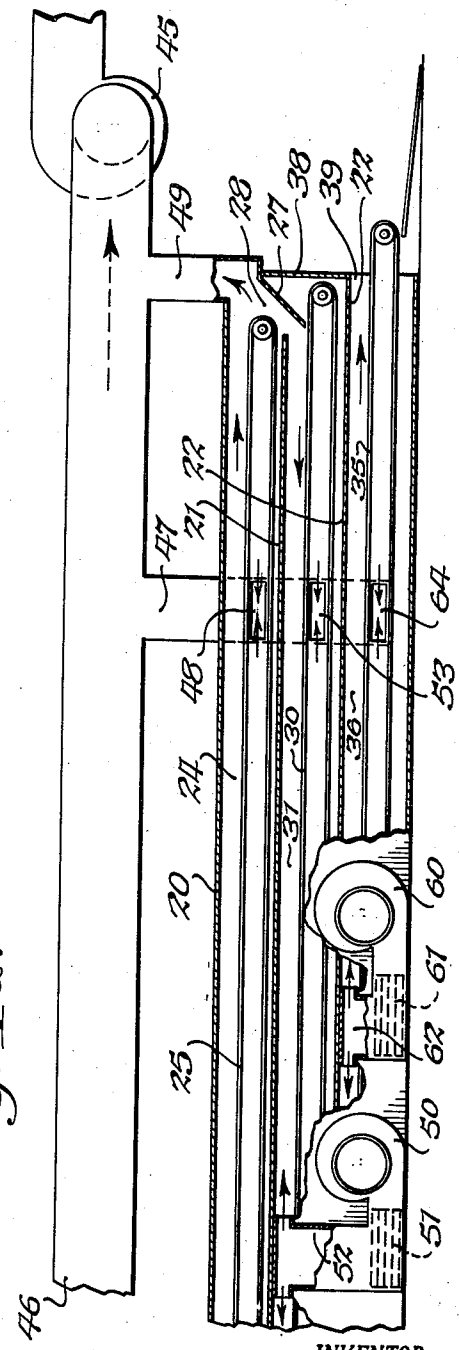


Fig. 1a.

INVENTOR.
Ralph R. Oliver,
BY
Parker, Prochnow & Farmer,
Attorneys.

Patented June 26, 1951

2,557,993

UNITED STATES PATENT OFFICE

2,557,993

PROCESS OF REMOVING MINERAL OIL FROM VEGETABLE FIBERS

Ralph R. Oliver, Lockport, N. Y., assignor to Lockport Cotton Batting Co., Lockport, N. Y.

Application July 30, 1948, Serial No. 41,654

10 Claims. (Cl. 117—138)

1

This invention relates to processes of removing from fibrous materials of vegetable origin, such as cotton, linen, hemp, jute, kapok and the like, mineral oils with which these materials have been contaminated.

In cotton mills, for example, a certain amount of cotton becomes contaminated with oil or lubricants during the spinning, weaving and other processing of the same, and this cotton must be discarded because it cannot be properly bleached, cleaned, dyed or flame-proofed. This cotton is commonly referred to as "cotton mill sweeps" or "oily card." Such mineral hydrocarbons that commonly contaminate fibers during the various operations on the same, are the usual spindle oils, lubricating oils and oils picked up by the vegetable fibers through processing or use, and are mostly unsaponifiable and hard to remove by any of the well known washing operations.

These cotton mill sweeps or oily card are ordinarily sold at a low price for the reason that heretofore it has been impossible to remove the mineral oils or greases from these cotton fibers, except by volatile solvent extraction, which is a hazardous operation, and the cost of which is prohibitive, or by a dry absorption process of incorporating finely ground china clay, talc or similar material, with the cotton in a tumbler mill, and then dusting out the excess of this material from the cotton, together with variable amounts of oil which has been absorbed by the material. This latter method is not sufficiently effective in removing mineral oils and has, therefore, not been found commercially practicable.

The tonnage available of this oily vegetable fibrous material, composed as it is of long and short vegetable fibers, is of such magnitude as to be of commercial interest providing the mineral hydrocarbon can be partly or wholly economically removed, leaving the fibers in a state suitable as a base fibrous material for other manufacturing purposes.

One of the objects of this invention is to provide an improved process for the removal of mineral oil contamination from vegetable fibers or fibrous materials, by means of which mineral oil can be very effectively and economicaly removed by volatilizing the same at high temperatures without damaging the fibrous materials.

Another object of this invention is to provide a process by means of which mineral oil can be removed by volatilization from the fibers, and in which water is used to prevent damage to the vegetable fibers while they are exposed to temperatures at which the mineral oil can be volatilized.

2

A further object of this invention is to provide a process by means of which mineral oils can be removed from vegetable fibers during the process of rendering the fibers flame-resistant.

Other objects and advantages of this invention will appear from the following description and claims.

According to my invention, the vegetable fibers contaminated with mineral oil are first thoroughly soaked in water which may, if desired, contain in solution materials which will render the fibers flame-resistant. After this soaking, excess moisture is removed from the fibers and they are then subjected to currents of air heated to temperatures considerably above the boiling point of water, which temperatures are sufficiently high to volatilize the mineral oil deposits on the fibers. I have found that when such high temperature air is passed into contact with the vegetable fibers, most of the mineral oils contaminating the fibers are volatilized and can thus be carried away with the currents of air used for drying. The temperature of this drying air, which is high enough to volatilize mineral oils is also high enough, so that it would when acting on relatively dry fibers, result in serious damage to the fibers by scorching, or charring the same, but when the fibers are wet with water, I have found that no damage results, even if the air used for volatilizing the mineral oils is as high as 160 degrees C. The fibers must, of course, be subjected to such high temperature only when they are wet and before sufficient moisture is removed from them to permit damage of the same by the high temperature air or other gas.

My process may be carried out by means of any suitable or desired apparatus and I have illustrated, by way of example, one type of apparatus which may be employed for this purpose.

In the accompanying drayings:

Fig. 1 is a sectional elevation of part of the apparatus which may be used in connection with the carrying out of the process, embodying this invention, and

Fig. 1*a* shows the other part of the apparatus.

In Fig. 1, there is shown a portion of a tank or elongated trough 5 for water or aqueous solution, and this trough includes a stationary screen 6 arranged above the bottom thereof.

The vegetable fibers contaminated with oil are usually received in bales, and the compacted mass of the fibers may be broken up or loosened in any suitable or usual manner (not shown) before they are supplied to the receiving end of the trough 5 containing water or a water solution of a material which when applied to or absorbed by

the films, makes them flame-resistant. The aqueous bath in the tank 5 may also contain a suitable surface depressant and is preferably held at temperatures between 45 degrees C. and 65 degrees C. to ensure a thorough wetting out of the fibers.

The loosened mass of fibrous material is fed lengthwise of the trough by means of a plurality of paddles 7 which also submerge the material in the water or aqueous solution in the tank. These paddle wheels 7 rotate in such a direction as to feed the fibrous material to the right in the drawing, as indicated by the arrow, toward the discharge end of the tank 5. At the discharge end the screen 6 slopes upwardly, as indicated at 8, and suitable means are provided for removing the saturated fibrous material from the tank, such for example, as a rake 9 mounted on a crank 10 which rotates in a counterclockwise direction so as to move the rake while in contact with the fibrous material to the right in Fig. 1.

The rake feeds the fibrous material between a pair of squeezing rollers 12 which remove excess moisture from the same. These rollers 12 are preferably adjusted so that the fibers discharged from the same will contain approximately from 45% to 55% of water, based on the dry weight of the fibrous material. The pressing rollers 12 discharge the fibrous material to a conveyor 14 which conducts the same to a fan or blower 15 or other suitable means for conveying the wet fibrous material to an evaporator. The fan, in the construction shown, discharges the fibrous material through the tube 16 which enters an end wall 17 of a drier or evaporator. A dryer or evaporator of any suitable or desired type may be employed, that shown in the construction illustrated being entirely enclosed in a housing 20 which includes the end wall 17.

The evaporation of water and solvent preferably takes place in a number of stages, three stages being shown in the construction illustrated, in which the evaporator or dryer is divided by means of substantially horizontally extending partitions 21 and 22 into three different compartments arranged one above the other in which the three stages of evaporation take place, and each of these compartments contains suitable conveying means for passing the fibrous material through the same. In the first stage of evaporation which is carried on in the upper compartment 24, a conveyor 25 is provided which conducts the material from the left end of the housing to the right end thereof, and discharges the same on an inclined plate or chute 27, Fig. 1*a* which conducts the material through an opening 28 in the partition 21. The material then passes on a conveyor 30 located in a second compartment 31 between the partitions 21 and 22, and this conveyor drops the fibrous material on another inclined plate 34 at the left end of the housing, Fig. 1, which carries the material past the end of the partition 22 to a third conveyor 35 arranged in the lowest compartment 36. The housing 20 has an end wall 38 with an opening 39 therein through which the conveyor 35 passes to discharge the material from the apparatus.

The apparatus shown is provided with three heaters. The first heater 40 is arranged in an air passage 41. This heater may comprise a series of steam pipes or any other means for elevating the temperature of the air flowing past the same to the desired degree. This passage 41 receives air from a fan or blower 42 and the heated air is discharged through an upright duct 43 leading to the upper compartment or stage 24 of the evaporator. The air in this compartment may vary between 120 degrees C. to 160° C. and this air is passed through the layer or mass of fibrous material carried by the upper conveyor 25. All of the conveyors 25, 30 and 35 are in the form of belts through which air may readily be passed, for example, by the provision of the large number of perforations in the conveyor belts, or if desired, the belts may be made of suitable metal screening or woven material with large interstices between them through which the drying or evaporating air may pass.

In the construction shown for this purpose, the duct 43 discharges the air into the upper portion of the compartment 24, and the heated air is drawn through the layer of the fibrous material on the conveyor 25 in any suitable manner. For example, an air exhaust system may be provided including an exhaust fan or blower 45, Fig. 1*a*, which discharges air from the evaporator or dryer and which receives air from the evaporator or dryer through a main duct 46 having a plurality of branch ducts 47 which have their air receiving openings arranged between the runs on the conveyor belts. For example, air is withdrawn from the first stage of the evaporating process in the compartment 24 by means of intake ducts 48 arranged between the runs of the belt 25 and, if desired, an additional duct 49 may be provided for withdrawing air from the discharge end of the compartment 24. Consequently, the drying air at a relatively high temperature above the boiling point of water is admitted to the compartment 24 above the upper run of the conveyor 25 and passes through the mass of fibrous material deposited on the upper run of this conveyor, and then enters the air receiving openings 48 of the suction or exhaust system and the additional duct 49, from where it is discharged through the main duct 46 to the exhaust fan 45. Any other means for withdrawing air and vapors from the evaporator housing and for supplying hot air thereto may, of course, be employed, if desired.

The heated air which is discharged into the second compartment 31 of the evaporator is at a materially lower temperature, preferably between 90 degrees C. and 120 degrees C. This air is supplied to the second compartment by means of a blower 50, Fig. 1*a*, and passes a heater 51 arranged in a duct 52 which terminates in the upper portion of the second compartment 31. The exhaust system which removes air from the first stage may be used for removing air and vapor from the second compartment or stage, and for this purpose, the ducts 46 may be provided with additional inlet openings 53, which extend between the two runs of the second conveyor 30. The drying operation in the second compartment is consequently very similar to that in the first compartment except that it carried on at a lower temperature, due to the fact that considerable moisture has been withdrawn from the fibrous material in the first stage or compartment.

The third compartment 36 receives air from the blower 60, Fig. 1*a*, which discharges air to a heater 61 and then into a duct 62. This duct discharges the heated air into the upper portion of the third compartment 36, and the air is withdrawn from the third compartment by the suction or exhaust system through air intakes 64 located between the runs of the con-

veyor 36. Since the fibrous material in the third compartment is relatively dry, the temperature of the air therein should not be materially above 90 degrees C.

Any other apparatus may, of course, be employed for subjecting the fibrous material while wet to temperatures sufficient to cause volatilization of the hydrocarbon oils which contaminate the fibrous material and, if desired, air discharged from any zone or stage of the apparatus may be reheated and re-circulated to absorb more water and oil vapors.

In the carrying out of my process in the apparatus illustrated, fibrous material, while containing a relatively large quantity of moisture is subjected to high temperatures in the first stage, which temperatures, if acting on dry, vegetable fibrous material would produce charring or scorching of the same, and render the same of little value. However, because of the presence of water in the fibrous material, these high temperatures have been found not to cause any deterioration or damage to the fibrous material, and the mineral oil in or on the material becomes volatilized and the vapors, together with water vapor, is discharged through the exhaust system. In the second stage, some additional mineral oil is volatilized and a large amount of water is removed from the fibrous material, and in the third stage, the fibrous material is dried to remove additional moisture therefrom. The fibrous material may be discharged from the third stage containing any desired amount of moisture, about 10% based on the bone-dry weight of the vegetable fibrous material usually being desired.

It is generally desirable to treat vegetable fibers for the purpose of rendering them flame-proof or fire-resistant, and the process of flame-proofing material may be carried on simultaneously with the removal of the mineral oils therefrom. Consequently, the water in the tank 5 in which the fibrous material is wetted may have, in solution, the materials desired to flame-proof the fibrous material. Boric acid and borax, or any of the other water-soluble, inorganic flame-proofing materials, such as borax and ammonium phosphate, ammonium sulphate, ammonium sulphamine, or others, may be used for this purpose. These flame-proofing materials dissolved in the water also increase to a considerable extent the ability of the fibrous material to withstand the high temperatures to which they are subjected in stages 1 and 2 of the apparatus to volatilize the mineral oils. Consequently, by dissolving flame-proofing materials in the water in the tank 5, the cotton or other fibrous material may be flame-proofed as well as freed of mineral oil contamination in the same process. When cotton contaminated with mineral oil is passed through the usual hot solution of borax and boric acid, or any of the other soluble, inorganic, flame-proofing materials, the mineral oil on the cotton is emulsified into a water-in-oil colloid. This fine dispersion of the oil exposes more oil surface to the heat during the resulting drying operation and thus facilitates the separation of oil in volatile form.

It is, of course, necessary that the fibrous material be subjected to temperatures above the boiling point of water only during the time that the fibers have sufficient moisture adhering thereto to prevent damage to the fibers by the hot air or gas. When my improved process is carried out in connection with the apparatus described, the fibrous material enters the first stage or zone containing approximately from 45% to 55% water, based on the dry weight of the fibers. When exposed to temperatures of between 120 degrees C. to 160 degrees C., the fibers remain in the first zone for approximately ten minutes so that when the fibers are discharged from the first zone, they will contain approximately 30% water. Since the temperature in the first zone is above the volatile temperature of the mineral hydrocarbons with which such fibers are commonly contaminated, as well as above the boiling point of water, volatile oils pass off as vapor and in a steam vapor phase, and the vapors are swept out of the drying chamber by the air currents. In the second zone, the fibers while exposed to temperatures of between 90 degrees C. to 120 degrees C. remain in this zone for about ten minutes and their moisture content is reduced to approximately 15%. In this second zone, more oil is volatilized and this vapor together with water vapor is discharged from this zone. In this stage, any aqueous inorganic flame-proofing compound in the solution is concentrated to a point of crystallization, the crystals forming within and around the vegetable fibers, and in or on the fibrous space formerly occupied by the volatilized oil. The final drying of the fibers in the third zone also takes place in about ten minutes, and the fibers leave the apparatus at the end of the third zone with the moisture content of about 7% to 10%.

The time required for each zone may, of course, be varied in accordance with variations in temperature and if the process is carried on in an apparatus having only two zones, one for volatilization of mineral oils and water, and the other for drying the fibrous material, the time of exposure of the fibrous material to the high temperatures may be varied considerably. When using apparatus of different types from the one shown in the drawings, the time during which the fibrous material can be safely exposed to high temperatures can be readily determined by passing samples of the fibrous material through the apparatus to determine the maximum time during which the fibrous material can be exposed to high temperatures without scorching or damaging the same.

My process is particularly desirable for use in connection with the simultaneous flame-proofing of the fibers and the removal of hydrocarbon oils therefrom, since both operations are carried on simultaneously, it being merely necessary to raise the temperatures in the first two zones sufficiently to volatilize hydrocarbon oils.

It will be understood that various changes in the details, materials, and arrangements of parts which have been herein described and illustrated in order to explain the nature of the invention, may be made by those skilled in the art within the principle and scope of the invention, as expressed in the appended claims.

I claim as my invention:

1. A process of removing mineral oil from vegetable fibers contaminated therewith which includes the steps of subjecting the fibrous material while the same contains at least 45% of water based on the dry weight of the fibers, to currents of air heated to temperatures between 120 degrees C. and 160 degrees C. to volatilize mineral oil and some of the water of the fibrous material, said treatment being continued until the water content of the fibrous material is approximately 30%, then subjecting the fibrous

material to air currents heated to approximately 90 degrees C. to 120 degrees C. until the water content of the fibrous material is reduced to about 15%, and then further drying the fibrous material at temperatures well below the boiling point of water to reduce the moisture content thereof.

2. A process of removing mineral oil from vegetable fibers contaminated therewith which includes the steps of soaking the fibers in an aqueous medium, removing from the fibers excess aqueous medium until the fibrous material contains not more than 55% of water based on the dry weight of the fibers, subjecting the wet fibers to currents of air heated to temperatures between 120 degrees C. to 160 degrees C. for a period of approximately ten minutes, to volatilize mineral oil and to evaporate some of the water in the fibrous material, then subjecting the fibrous material to temperatures between 90 degrees C. and 120 degrees C. for a period of ten minutes to remove further mineral oil and water therefrom, and then subjecting the fibers to lower temperatures to reduce the water content thereof to the point desired.

3. A process of removing mineral oil from vegetable fibers contaminated therewith, which includes the steps of soaking the fibers in an aqueous medium, removing from the fibers excess aqueous medium so that the fibers retain approximately from 45% to 55% of said medium based on the dry weight of said fibers, volatilizing most of said mineral oil by subjecting the fibers to currents of air heated to a temperature in excess of 120° C. and not exceeding 160° C., and then evaporating additional mineral oil and water from the fibers by subjecting them to currents of air heated to temperatures between 90° C. and 120° C.

4. A process of simultaneously removing mineral oil from vegetable fibers and rendering the fibers flame resistant by precipitating a flame-resistant material upon them, which method includes subjecting the fibers to an aqueous solution of said flame-resistant material, removing excess water from the fibrous material so that the water retained by the fibers is approximately 45% to 55% by weight of the dry weight of the fibers, subjecting the fibers to moving currents of air heated to a temperature of from 120° C. to 160° C. to vaporize and remove the oil and water until the moisture content of the fibrous material is reduced to about 30%, and thereafter subjecting the fibers to moving currents of air at a lower temperature to further reduce the moisture content of the fibers to approximately 7%–10%.

5. A process according to claim 4 in which the flame-resistant material in said aqueous solution is borax and boric acid.

6. A process of simultaneously removing mineral oil from vegetable fibers and rendering the fibers flame resistant by precipitating a flame-resistant material upon them, which method includes the steps of subjecting the

fibrous material to an aqueous solution of said flame-resistant material, subjecting the fibrous material, while it contains at least 45% of water based on the dry weight of the fibers, to currents of air heated to a temperature between 120° C. and 160° C. to vaporize and remove the oil and water until the moisture content of the fibrous material is reduced to approximately 30%, then subjecting the fibrous material to air currents heated to approximately 90° C. to 120° C. until the water content of the fibrous material is reduced to about 15% and then further drying the fibrous material at temperatures well below the boiling point of water to reduce the moisture content thereof to the final value desired.

7. A process according to claim 6 in which the flame-resistant material in said aqueous solution is borax and boric-acid.

8. A process of removing mineral oil from vegetable fibers contaminated therewith, which includes the steps of soaking the fibers in aqueous medium, removing from the fibers excess aqueous solution so that the fibers retain approximately from 45% to 55% of said solution based on the dry weight of said fibers, subjecting the fibers to moving currents of air heated to a temperature from approximately 120° C. to 160° C. for a period of approximately ten minutes, and continuously withdrawing from the fibers the vapors of said mineral oil and water.

9. A process of removing mineral oil from vegetable fibers contaminated therewith, which includes the steps of soaking the fibers in an aqueous medium, removing from the fibers excess aqueous solution so that the fibers retain approximately from 45% to 55% of said solution based on the dry weight of said fibers, subjecting the fibers to moving currents of air heated to a temperature from approximately 120° C. to 160° C. until the water in the fibrous mass is reduced to about 30%, and continuously withdrawing from said fibers the vapors of said mineral oil and water.

10. A process according to claim 9 and in which the fibers are then subjected to air at a lower temperature to remove additional moisture from the fibers.

RALPH R. OLIVER.

REFERENCES CITED

The following references are of record in the file of this patent:

UNITED STATES PATENTS

Number	Name	Date
372,987	Robeson	Nov. 8, 1887
1,569,484	Hall	Jan. 12, 1926
2,072,253	Ellis et al.	Mar. 2, 1937
2,178,625	Clayton et al.	Nov. 7, 1939
2,281,560	Dew et al.	May 5, 1942

FOREIGN PATENTS

Number	Country	Date
347,444	Great Britain	Apr. 30, 1931