June 28, 1955 I. J. NOVAK 2,711,822

PURIFICATION OF FIBROUS ASBESTOS

Filed July 24, 1953 2 Sheets-Sheet 1

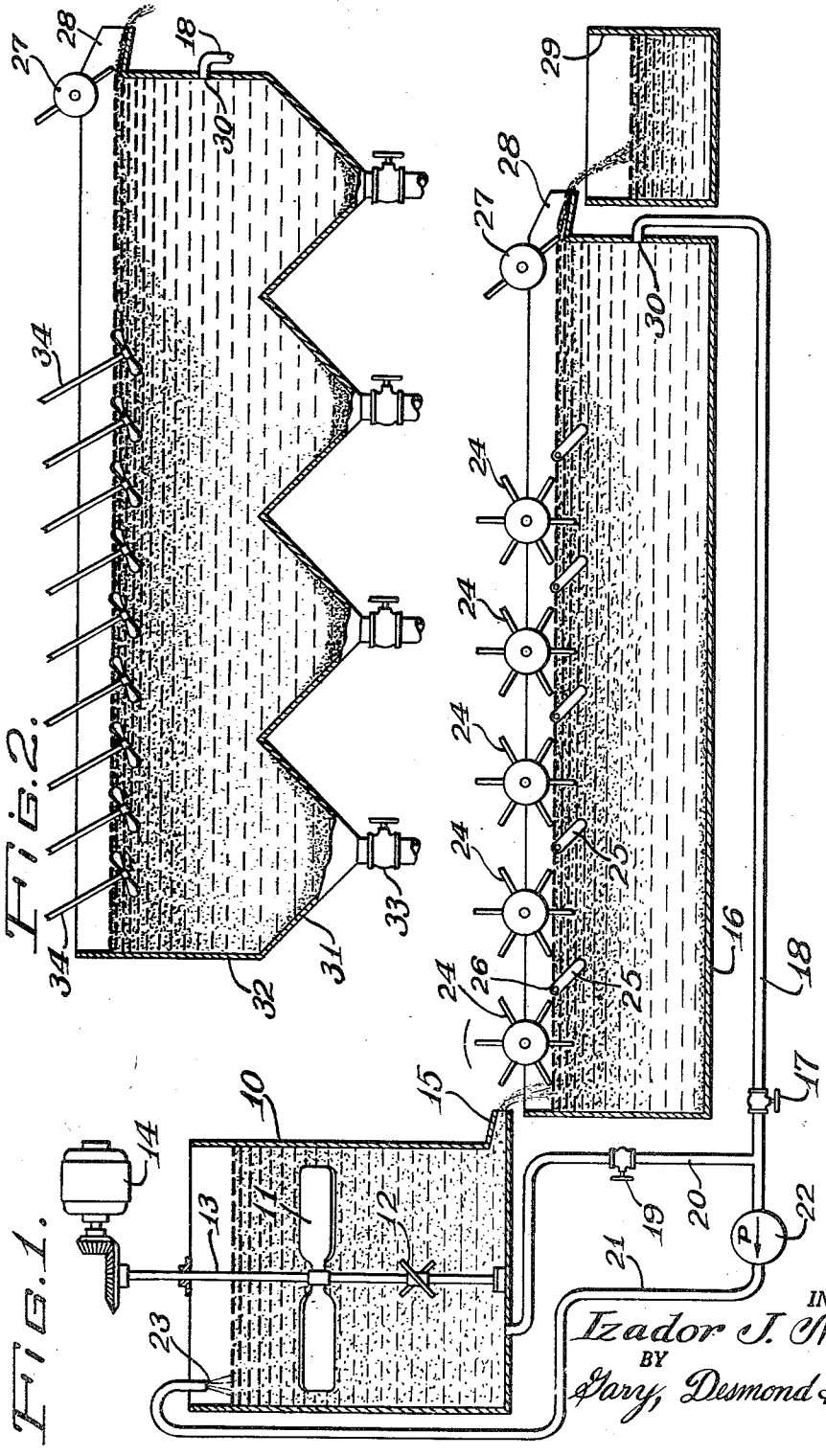


Fig. 2.

Fig. 1.

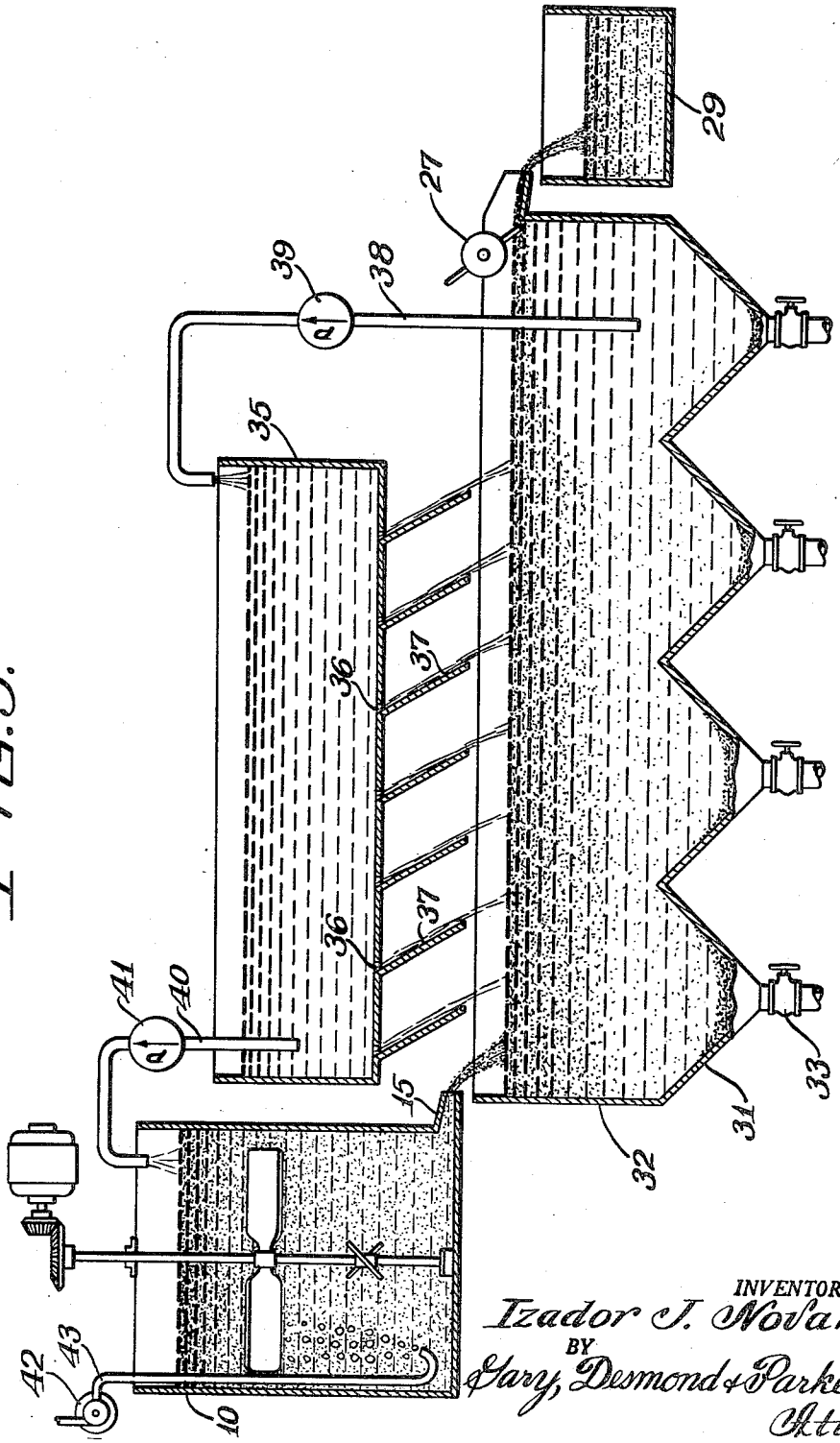
INVENTOR.
Izador J. Novak
BY
Gary, Desmond & Parker
Attys.

June 28, 1955 I. J. NOVAK 2,711,822

PURIFICATION OF FIBROUS ASBESTOS

Filed July 24, 1953 2 Sheets-Sheet 2

FIG. 3.



INVENTOR.
Izador J. Novak
BY
Gary, Desmond & Parker
Attys.

United States Patent Office 2,711,822 Patented June 28, 1955

2,711,822

PURIFICATION OF FIBROUS ASBESTOS

Izador J. Novak, Trumbull, Conn., assignor to Raybestos-Manhattan, Inc., Passaic, N. J., a corporation of New Jersey

Application July 24, 1953, Serial No. 370,023

6 Claims. (Cl. 209—166)

This invention relates to the cleaning of milled chrysotile asbestos fiber, and more specifically to the separation and removal of normally occluded residual undesirable particle material content from masses of said fibers.

As will hereinafter be more particularly described, the objects and advantages of the present invention are in general accomplished by novel froth flotation method and means.

It is well known to the asbestos industry that the milled, bagged, fibrous asbestos of commerce contains considerable proportions of non-asbestos mineral in granular form remaining from imperfect removal of the gangue, such as magnetite and serpentine sand, as well as splints of varying diameter comprising pseudo-asbestos called picrolite, which have been incompletely separated in the conventional crushing, winnowing and screening processes. It is therefore frequently necessary for the user to resort to additional purifying in order to remove enough of these impurities to allow him to use the asbestos for his own end products. This is particularly true where high grade yarns are produced wherein splints and granular material are very objectionable and in the case of electrical insulation products where the electrical qualities are impaired by the presence of even minor proportions of magnetite. Furthermore, wherever thin products are produced, oversize splints are considered serious defects because they form irregularities on the surface or body of the thin structure, and if the structure is flexed or surface ground they produce holes. The mechanical methods for removing splints and granular materials are quite destructive or fiber length and cause considerable expense and fiber loss. For example, losses in spinning fiber for yarn production ordinarily run from 15 to 25% of the original weight. Also, certain types of fiber, for example, single grade asbestos fiber, carry such large proportions of splinty material and magnetite that they are entirely unsuitable for electrical uses. Again, asbestos floats may contain only 75% of asbestos, the rest being finely ground sand and magnetite, thereby unfitting it for uses where clean asbestos is required.

As previously indicated, I have found, in accordance with the present invention, that masses of milled chrysotile asbestos fibers of commerce may be further cleaned by subjecting them to froth flotation. This is accomplished by aerating the fibers in the form of a dilute aqueous slurry containing a flotation agent selective to asbestos and adsorbable thereon which causes air bubbles to adhere to the surfaces of the asbestos fibers and thereby cause them to float while permitting the insensitive and nonfloatable granular and splinty impurity particles to sink and separate out. However, while this set of conditions establishes differential and opposite effects to the directions of the fiber and the impurities in the aqueous medium, somewhat akin to the ordinary ore flotation process, there are important and characterizing distinctions. Thus while in the ordinary ore flotation process separation of floated from unfloated material is relatively rapid, such is not the case with asbestos fibers.

By their very nature, the fine asbestos fibers have a tendency to tangle, which tendency increases with increase in fiber length, and is a primary reason for original retention or entanglement of the described impurities. Therefore only relatively imperfect separation can occur by froth flotation in the absence of resorting to added methods or means.

I have thus further found, in accordance with the present invention, that the asbestos fibers may be more perfectly cleaned by a forcing down of the aerated floating asbestos to temporarily reverse their floating tendency while moving forwardly in a body of aqueous liquid, by agitation in a zone adjacent to the surface of said liquid body, and while preventing the accumulation of asbestos froth at the surface until after the desired cleaning has been effected.

This successive forcing down and agitation of the floating fibrous material diffuses them in the form of a zone or cloud of relatively lowered concentration adjacent to the surface of the liquid body, with its bottom outlines fairly well defined from a lowermost relatively clear and quiescent zone. From the bottom of this cloud of fibers in the upper agitated zone, and into the lower relatively quiescent zone, fall the undesirable particles of granular and splinty material.

It is further desirable to maintain the fibers in such agitated condition for a period considerably in excess of that normally required for the fibers to freely float to the surface and sufficient to allow the maximum possibility for particles of undesirable material to reach the interface of the cloud and the quiet zone below to get the best removal. In the present continuous process where an impure aerated asbestos slurry is fed into one end of a tank, at or adjacent to the upper surface of the liquid therein, and the purified asbestos fibers are removed at the other end, it is a requirement that a forward movement of the asbestos in the agitated zone be provided, so that the rate of passage from the intake to the exit is controllable and a long enough period in the diffuse agitated condition is allowed to offer the maximum opportunity for separation of impurities consistent with a reasonable throughput. As will appear hereinafter, this successive forcing down and agitation of the aerated asbestos froth and continuous forward movement thereof may be accomplished by mechanical or hydraulic means.

As previously indicated, as a first step in the process, impure asbestos fiber, flotation agent and water are premixed in a suitable vessel, in a relatively dilute condition, for example from about ¼% to about 1% asbestos concentration. The matter of flotation agents and the technique for providing a froth flotation agent system in this first step are well known in the ore dressing art, and the same general principles are applicable here.

Thus, for example, with the flotation agent present, the aeration may be caused by recirculation of the slurry with entrainment of air during the recirculation, or by the type of mixing equipment employed, or by the introduction of air below the surface, all of which means are well known.

It will be understood that the flotation agent employed should not be one which might deteriorate the asbestos such as would be the case if a strong caustic or acid were used, nor should it be of a kind which might form sticky asbestos surfaces tending to cause fiber clotting, since fiber separation is necessary for good cleaning. Also too great a tendency to open the fibers and increase their number, as in a colloidal asbestos dispersion or under severe mixing or beating conditions, is also undesirable, since it decreases the chances of impurities dropping out by increasing the

number of interfering fibers. As examples of highly suitable chrysotile asbestos flotation agents, among others, are the following: dioctyl sodium sulfosuccinate (Aerosol O. T.); kerylbenzene sulfonate (Nacconal N. R.); hexadecyl benzene sulfonate (Ultrawet K); hydrocarbon sulfonate (Du Pont MP 189); alkyl aryl sulfonate (Santomerse D); alcohol sulfate (Wetanol); alcohol sulfate (Duponol WA); sodium oleate; ammonium oleate, and others. They are adsorbable on the surfaces of the asbestos fibers and provide both selectivity to the asbestos and the requisite firm attachment of small air bubbles to the asbestos surface of a sufficiently desirable nature to float the fibers safely for the period necessary for passage through the flotation operation. Soft water is desirable if the flotation agent is sensitive to magnesium or calcium salts. Based on the asbestos weight, from about ¼% to about 2% of the flotation agent is adequate. Below and above the optimum range, good flotation is hindered or prevented.

The process of the invention and means for carrying same out will be further explained in connection with the accompanying diagrammatic drawings, wherein:

Fig. 1 illustrates in vertical section one suitable arrangement of apparatus.

Fig. 2 illustrates in vertical section a modified form of the flotation cell of Fig. 1.

Fig. 3 illustrates in vertical section another modified arrangement of apparatus.

Referring to the drawings, and particularly to Fig. 1, the reference numeral **10** indicates a premix vessel provided with the mixing means **11** and **12** on the axial shaft **13** driven through motor **14**, to keep the mixture of water, asbestos fibers and flotation agent introduced thereto, in a mildly agitated condition, to prevent flotation of the fibers, and to provide a uniform feed of a substantially homogeneous slurry from the outlet spout **15** to the flotation cell **16**. Aeration of the mixture in vessel **10** may be accomplished by closing valve **17** in line **18** and opening valve **19** in line **20**, and recirculating the contents of vessel **10**, through line **21** by means of pump **22**. Aeration occurs by discharge from line **21** at its terminal end or nozzle portion **23** spaced above the level of liquid maintained in vessel **10**.

The aerated mixture from vessel **10** is introduced through spout **15** by gravity to the body of aqueous liquid maintained in the flotation tank or cell **16** and thereat forced down under the surface by means of the series of horizontal paddle wheels **24**. These wheels in addition to causing a current to move the frothed asbestos in a forward direction to the discharge end of the tank, provide agitation of the asbestos fibers and disseminate them in the form of diffuse cloud beneath the surface of the liquid level. The position of paddle wheels **24** or the level of liquid in the tank with respect to them, and their speed of rotation, are regulated so that the diffused cloud of fibers extends to a fairly well defined zone of limited depth, leaving a relatively quiescent zone adjacent the bottom of the tank. The baffles **25** extending transversely of tank **16** aid in controlling the desired depth of the fibrous cloud and forward movement of the fiber. They may be mounted at a fixed angular inclination, or may be adjustably mounted on the pivots **26** so that they may be fixed at an altered inclination. The asbestos fibers in their forward movement are forced below the liquid level and agitated by the successive paddle wheels in the series so as to provide an extended diffused cloud of fibers beneath the liquid level while retaining therebelow a continuous relatively quiescent zone of liquid, and thus permitting the non-floated undesirable impurities to settle out to the bottom of tank **16**.

After passing the last paddle wheel **24**, the frothed asbestos is permitted to collect and float to the top and be skimmed off in relatively clean condition by the rotating skimmer **27** through the spout **28** and the discharge end of tank **16**, and into the collecting vessel **29**. For example, in a tank four feet deep, twenty feet long, and five feet wide, the paddle wheels and baffles are suitably regulated to produce an asbestos cloud in the agitated zone of from about one to about two feet in depth, for fifteen feet of the tank length. The remaining five feet is the flotation area where agitation is stopped and the asbestos cloud "settles" to the surface as an asbestos froth where it is skimmed off. At this point the fiber may be used wet or dried out as needed for further processing. The froth disappears rapidly on dilution with new water.

Since the purified asbestos is at a concentration usually considerably higher than when it entered the tank, the excess of water must also be removed in order to keep the level constant. It is taken out at the end of the tank at any convenient place below the collected frothy asbestos on the surface and above the settled impurities level, as at the point **30**, and returned to the premix system through lines **18** and **21** by means of pump **22** by closing valve **19** and opening valve **17**. In operation the concentration of the asbestos in the agitated zone may be of the order of 1/10%, depending on the rate of throughput and the degree of cleaning required. Its period in the agitated zone may run variously from 30 seconds to 10 minutes, again depending on the degree of cleaning required. The longer periods naturally provide more opportunity for removal of impurities. It should be emphasized that this is a single stage flotation cell with artificially and intentionally prolonged duration of the agitated fiber condition and thereby differs materially from conventionally multi-stage flotation cells where the cycle of complete mixing and floating is repeated and rapid in each cell. Essentially I have horizontal superimposed turbulent and quiet zones, the turbulent zone at the top containing the asbestos fibers, separated and agitated, and below it a quiet settling zone for the slowly falling impurities. At the bottom of the turbulent zone is an intermediate layer of relatively low turbulence into which impurities which are carried around by the turbulent liquid lose their rapid random direction and velocity and begin to fall into the quiet zone, then continue on to the settling area at the bottom, which is undisturbed by the upper currents. A horizontal screen or sloping baffles may be used to reduce the possibility of disturbing the settled matter but ordinarily are not necessary, as the zones can be kept quite well defined.

The capacity or throughput is proportional to the width of the agitated zone, and the feed rate and the degree of cleaning to the length in the direction of motion and time of residence in the agitated zone.

As shown in Fig. 2, I may, for greater convenience in cleaning continuously, supply a series of inverted cones or spitzkasten **31** in the bottom of the modified flotation tank **32**, whereby the settled impurities can be drawn off through valves **33** while the equipment is in operation instead of remaining on a flat bottom until the whole tank can be emptied and the impurities washed or shoveled out.

Other means of providing turbulence in the upper zone may be various types of vertical bar or propeller stirrers arranged to cover the desired amount of surface and agitate for the desired depth as well as provide the forward impetus to the fiber and prevent accumulation of asbestos froth on the surface until the flotation area is reached, such as the inclined propeller stirrers **34** shown in Fig. 2.

As a further alternative, sprays or flat streams of water drawn from the clarified liquid at the exit end of the flotation tank may be recirculated to give the desired upper layer agitation and flow. Thus Fig. 3, which shows a premix vessel **10** as in Fig. 1, and a flotation tank or cell **32** as in Fig. 2, further illustrates hydraulic means for forcing the aerated asbestos fibers under the surface of the liquid body and to thereby agitate them at and adjacent thereto in the form of a cloud zone, while at the same time providing forward impetus. This is accomplished in the illustrated means by disposing a trough **35** having a

plurality of spaced slots **36** in its bottom opening to the forwardly inclined aprons **37** extending transversely of trough **35**, above the level of the liquid in tank **32**. A suitable head of liquid is maintained in trough **35** by recirculating excess clarified liquid from the exit end of tank **32** by means of line **38** and pump **39**, and excess liquid in trough **35** is recirculated to the premix vessel **10** by means of line **40** and pump **41**. Thus the liquid issuing from trough **35** in the form of a plurality of successive flat streams provides forward impetus to the aerated asbestos in tank **32** while at the same time providing cloud agitation as previously described. Aeration may be provided by a separate source of air from the air pump **42** to the line **43** projecting into tank **10**.

Although not shown, as a further alternative, where it is desired to process the cleaned fiber in dilute condition, the cleaned fiber may be removed at the exit end before it is allowed to concentrate as froth at the surface as in the previous examples, and at once be transferred in this condition to further process equipment. Also the froth concentration may be accomplished in a succeeding step away from the main flotation tank.

By proceeding in accordance with the present invention I have cleaned various grades of Canadian Thetford asbestos in one pass to provide electrical quality fiber having fewer conductive particles than standard electrical grade Rhodesian Chrysotile when made into similar sheet products. As a comparison, a three mil thick paper sheet of uncleaned Canadian Thetford spinning grade fiber (3R) showed 125 conductive particles per square foot, Rhodesian Chrysotile (Shabanie) showed 90, Canadian Cassiar (surface ore) showed 100, flotation cleaned 3R showed 40, flotation cleaned Cassiar showed 20, flotation cleaned Thetford shingle grade showed 20. Asbestos paper of this thickness is used satisfactorily in magnet wire insulation at a conductive particle count of 30 to 50 per square foot. In addition to removal of magnetite particles as demonstrated by the above marked improvement, the fiber was free of oversize splints and sand, as well as considerably reduced in respect to water soluble salts. The weight of impurities varied between 5 and 8% for the above fibers and the impurities themselves consisted of asbestos and picrolite splints, splinty asbestos impregnated with magnetite, as well as granular magnetite and grains of serpentine rock. The cleaned asbestos, when dried and conventionally opened, shows an improved degree of fluffiness, whiteness and softness to the feel. It may be used directly in standard asbestos processes wherever its qualities are required. Although it carries a small percentage of organic matter, this is immaterial in any of its common uses. The percentage is usually not over ½ to 1%. There is no deterioration of asbestos quality, since no chemical deterioration occurs as would be the case if strong caustic or acid were used, since the flotation reagents in the proportion indicated have practically no effect on the pH of the asbestos slurry, and the fibers are not substantially shortened.

It should be noted that the high degree of cleaning accomplished by this process was not and could not have been foreseen or predicted, since both wet and dry cleaning and classification methods have been in use in the asbestos mining industry for long periods, and the asbestos of commerce today still contains the high proportions of undesirable impurities mentioned and is bought as is, nor is there any class of asbestos fiber sold with high purity specifications except what is the run of a naturally high purity asbestos deposit. For the first time, therefore, undamaged and undegraded asbestos of high purity from impure sources is available by the use of the present process.

I claim as my invention:

1. The method of refining a mass of milled asbestos fibers to remove therefrom residual particles of naturally associated impurities, which comprises introducing said fibers in an aerated frothy floatable condition to one end of and adjacent to the surface of a body of aqueous liquid and repeatedly forcing said floatable fibers under the surface of said liquid body while subjecting them to a forward impetus to the opposite end to successively agitate and disseminate them in the form of a diffuse turbulent cloud extended over a major portion of the liquid body and of limited depth in a zone beneath and adjacent to the surface of said body while retaining therein a lower relatively quiescent zone, whereby to permit separation and settling by gravity of said impurities through the disseminated agitated fibers and into said lower relatively quiescent zone, thereafter permitting the resulting cleaned fibers to float to the surface at the other end of said body and collecting them thereat, and collecting the separated impurities from the bottom of the liquid body.

2. The continuous method of refining a mass of milled asbestos fibers to remove therefrom residual particles of naturally associated impurities, which comprises introducing a dilute substantially homogeneous aqueous slurry of said fibers in an aerated frothy flotable condition to one end of and adjacent to the surface of a body of aqueous liquid, agitating said floatable fibers at a plurality of points spaced to successively force said fibers under the surface of said liquid body while subjecting them to a forward impetus towards the opposed end, to disseminate them in the form of a diffuse turbulent cloud of limited depth in a zone beneath and adjacent to the surface of said body while retaining therein a lower relatively quiescent zone, whereby to permit separation and settling by gravity of said impurities through the disseminated agitated fibers and into said lower relatively quiescent zone, thereafter permitting the resulting cleaned fibers to freely float to the surface at the other end of said body and collecting them thereat, removing excess aqueous liquid therebelow, and collecting the separated impurities from the bottom of the liquid body.

3. The method of refining a mass of milled asbestos fibers to remove therefrom residual particles of naturally associated impurities, which comprises aerating a dilute aqueous slurry of said asbestos in the presence of a flotation agent therefor in a premixing zone, introducing said slurry therefrom to one end of and adjacent to the surface of a body of aqueous liquid and agitating and repeatedly forcing said floatable fibers under the surface of said liquid body while imparting forward movement thereto towards the opposed end, to disseminate them in the form of a diffuse turbulent cloud of limited depth in a zone beneath and adjacent to the surface of said body while retaining therein a lower relatively quiescent zone, whereby to permit separation and settling by gravity of said impurities through the disseminated agitated fibers and into said lower relatively quiescent zone, thereafter collecting the resulting cleaned fibers at the other end of said body, and collecting the separated impurities from the bottom of the liquid body.

4. A continuous method of refining a mass of milled chrysotile asbestos fibers to remove therefrom residual particles of naturally associated impurities, which comprises aerating a dilute aqueous slurry of said asbestos in the presence of a chrysotile asbestos flotation agent in a premixing zone, continuously introducing said slurry in a substantially homogeneous condition, with said fibers in an aerated frothy floatable state and said impurities in a nonfloating state, to one end of the surface of a body of aqueous liquid and successively forcing said floatable fibers under the surface of said liquid body at spaced areas while subjecting them to a forward impetus to the opposed end, to repeatedly agitate and disseminate them in the form of a diffuse turbulent cloud of limited depth in a zone beneath and adjacent to the surface of said body while retaining therein a lower relatively quiescent zone, whereby to permit separation and settling by gravity of said impurities through the disseminated agitated fibers and into said lower relatively quiescent zone, thereafter permitting the resulting cleaned fibers to freely

float to the surface of said body in the form of a matted stratified frothy layer and collecting them thereat, removing excess aqueous liquid therebelow and returning it to said premixing zone, and collecting the separated impurities from the bottom of the liquid body.

5. Apparatus for refining a mass of matted asbestos fibers to remove therefrom residual particles of naturally associated impurities, comprising a premixing vessel for forming a substantially homogeneous aerated aqueous slurry of said asbestos in the presence of a flotation agent therefor, a flotation tank adapted to contain a body of aqueous liquid, means for transferring said homogeneous slurry from the bottom of said premixing vessel to one end of a body of liquid in said flotation tank, means disposed adjacent the top of said flotation tank and extending over a major portion thereof from its inlet end towards its opposed outlet end adapted to agitate and impart forward impetus to frothed asbestos introduced to said liquid body and to force it down a limited depth in the form of a disseminated cloud over a corresponding major portion of the liquid body while retaining therein a lower relatively quiescent zone, overflow means at the opposed end of said flotation tank for separating therefrom cleaned asbestos, and means for recirculating excess liquid from the relatively quiescent lower zone of said tank to said premixing vessel.

6. Apparatus for refining a mass of matted asbestos

fibers to remove therefrom naturally associated impurities, comprising a flotation tank adapted to contain a body of flotation liquid therein, means for introducing to one end thereof an aqueous slurry of said fibers in aerated frothy floatable condition, means disposed adjacent the top of said tank and extending over a major portion thereof from its inlet end and spaced from its opposed outlet end a sufficient distance to give the fibers an opportunity to rise adapted to impart a forward impetus to and to successively force frothed asbestos down beneath the surface of said liquid body for a limited depth in the form of a disseminated cloud while retaining therein a lower relatively quiescent zone, and means at the opposed end of said tank for separating from the body of liquid therein cleaned asbestos.

References Cited in the file of this patent

UNITED STATES PATENTS

1,282,730	Arzinger	Oct. 29, 1918
1,323,373	Myers	Dec. 2, 1919
1,624,163	Dolbear et al.	Apr. 12, 1927
2,606,660	Klepetko et al.	Aug. 12, 1952

FOREIGN PATENTS

121,991	Sweden	June 22, 1948