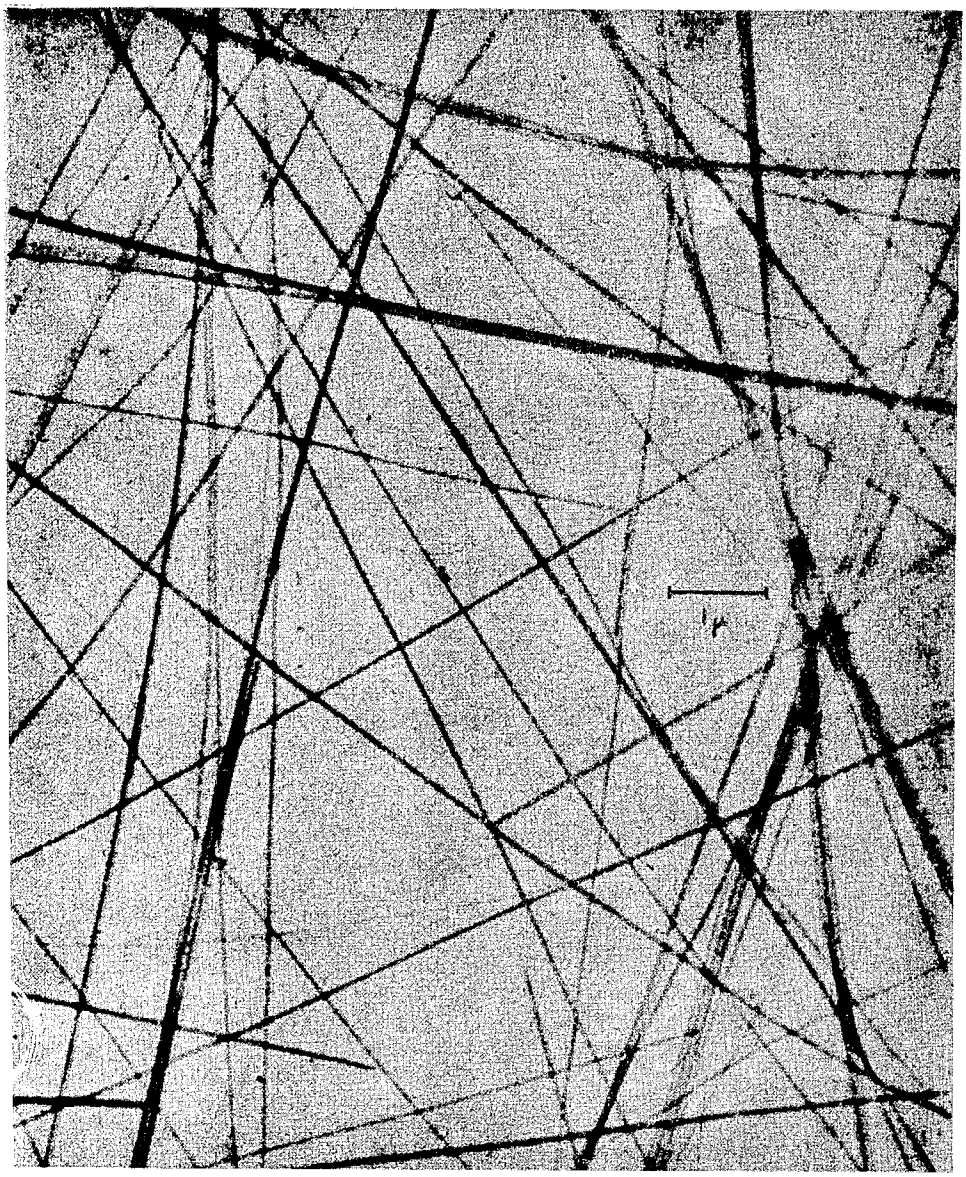
Jan. 20, 1953 I. J. NOVAK 2,626,213

ASBESTOS DISPERSIONS AND METHOD OF FORMING SAME

Filed Dec. 21, 1948 6 Sheets-Sheet 1

FIG. 1.

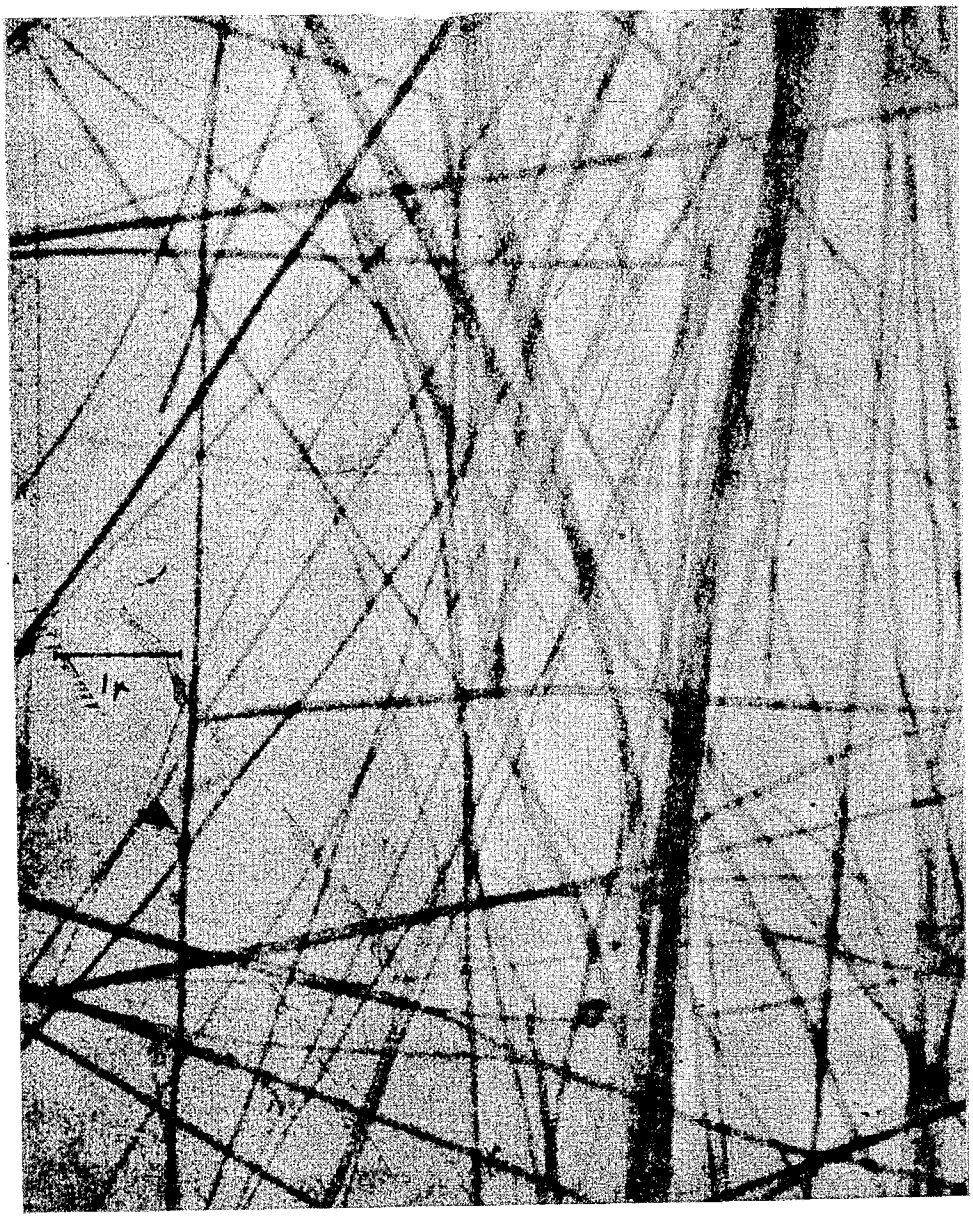


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ASBESTOS DISPERSIONS AND METHOD OF FORMING SAME

Filed Dec. 21, 1948 6 Sheets-Sheet 2

FIG. 2.



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ASBESTOS DISPERSIONS AND METHOD OF FORMING SAME

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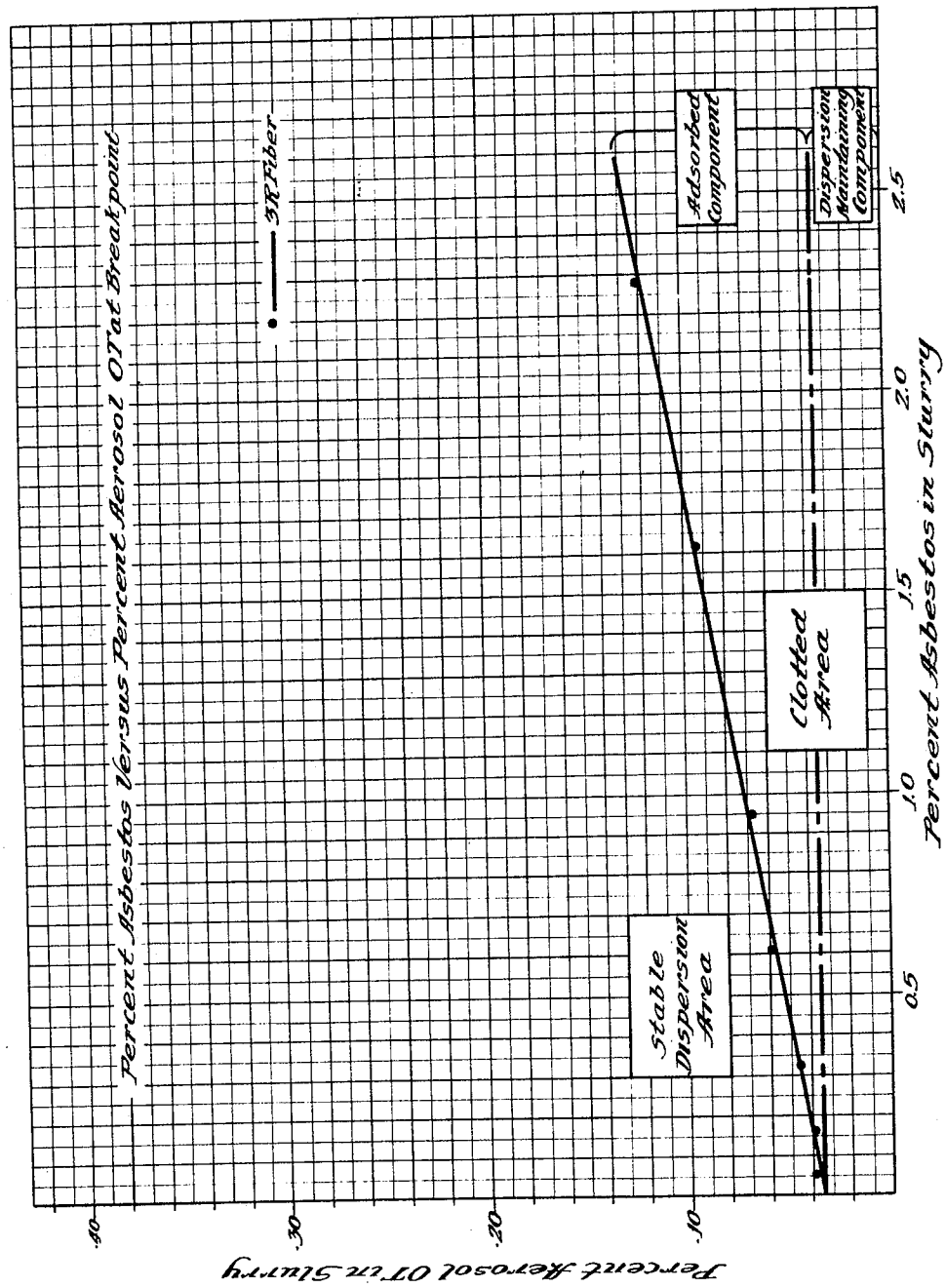


FIG. 3.

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ASBESTOS DISPERSIONS AND METHOD OF FORMING SAME

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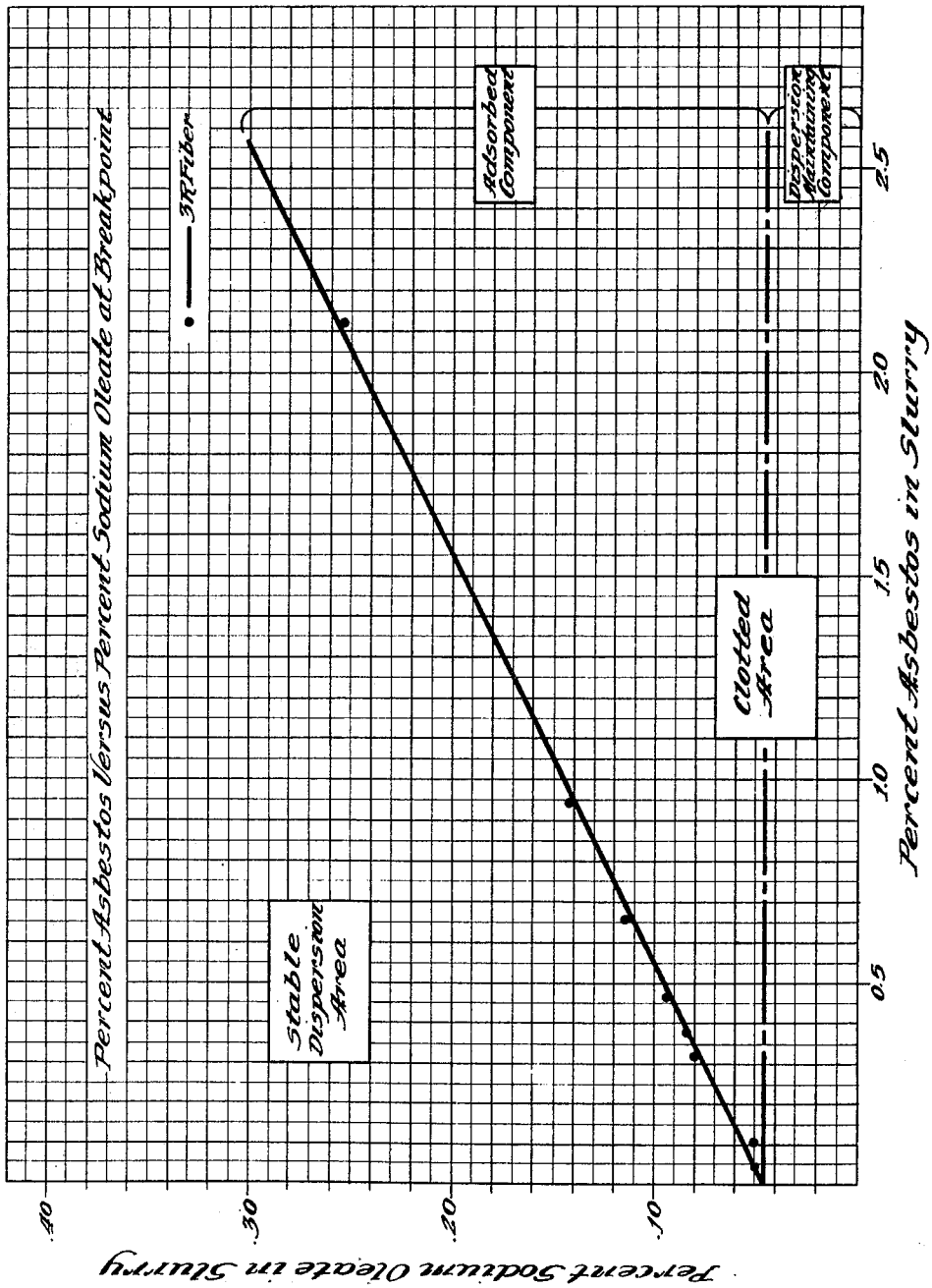


FIG. 4.

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ASBESTOS DISPERSIONS AND METHOD OF FORMING SAME

Filed Dec. 21, 1948 6 Sheets-Sheet 5

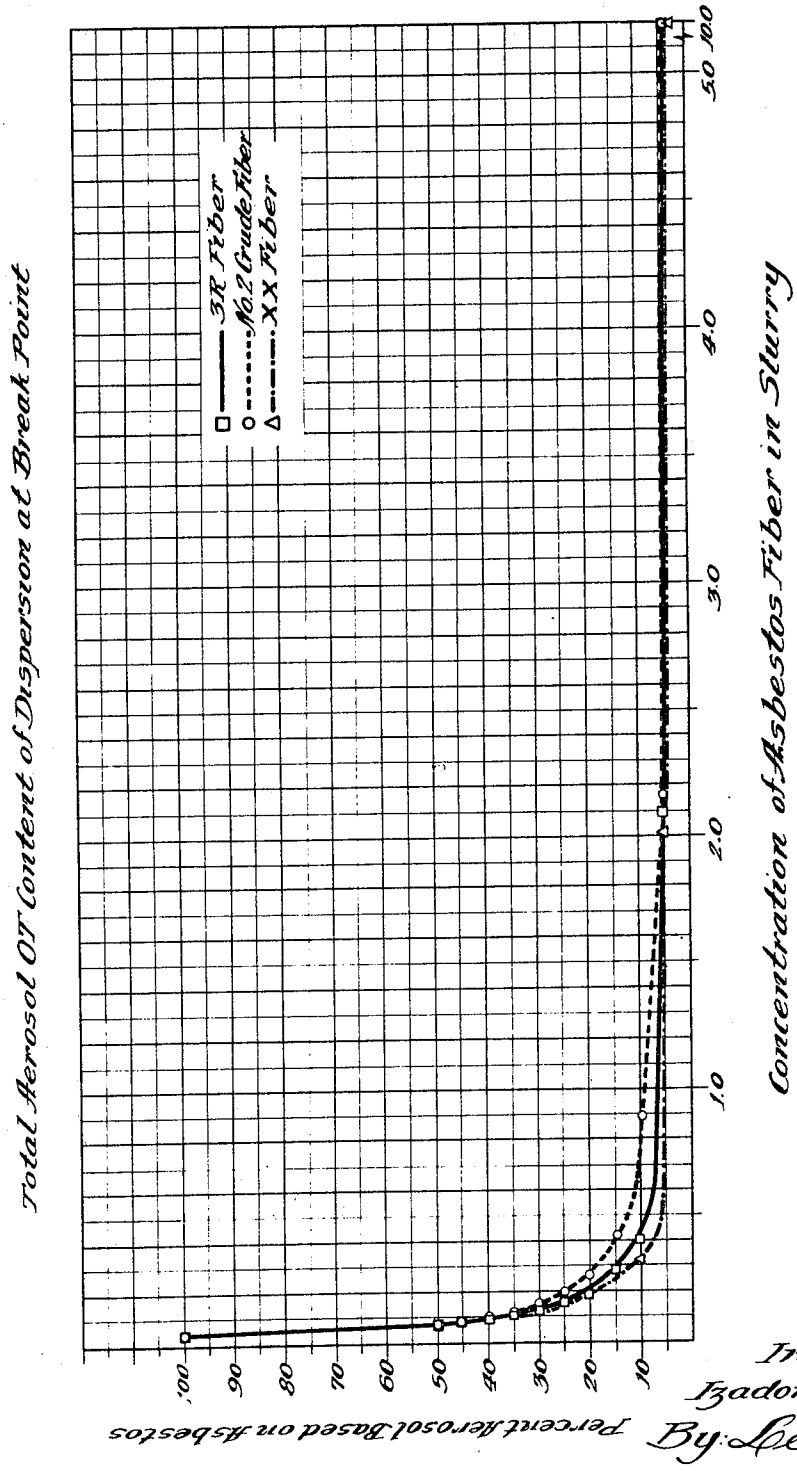


FIG. 5.

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ASBESTOS DISPERSIONS AND METHOD OF FORMING SAME

Filed Dec. 21, 1948 6 Sheets-Sheet 6

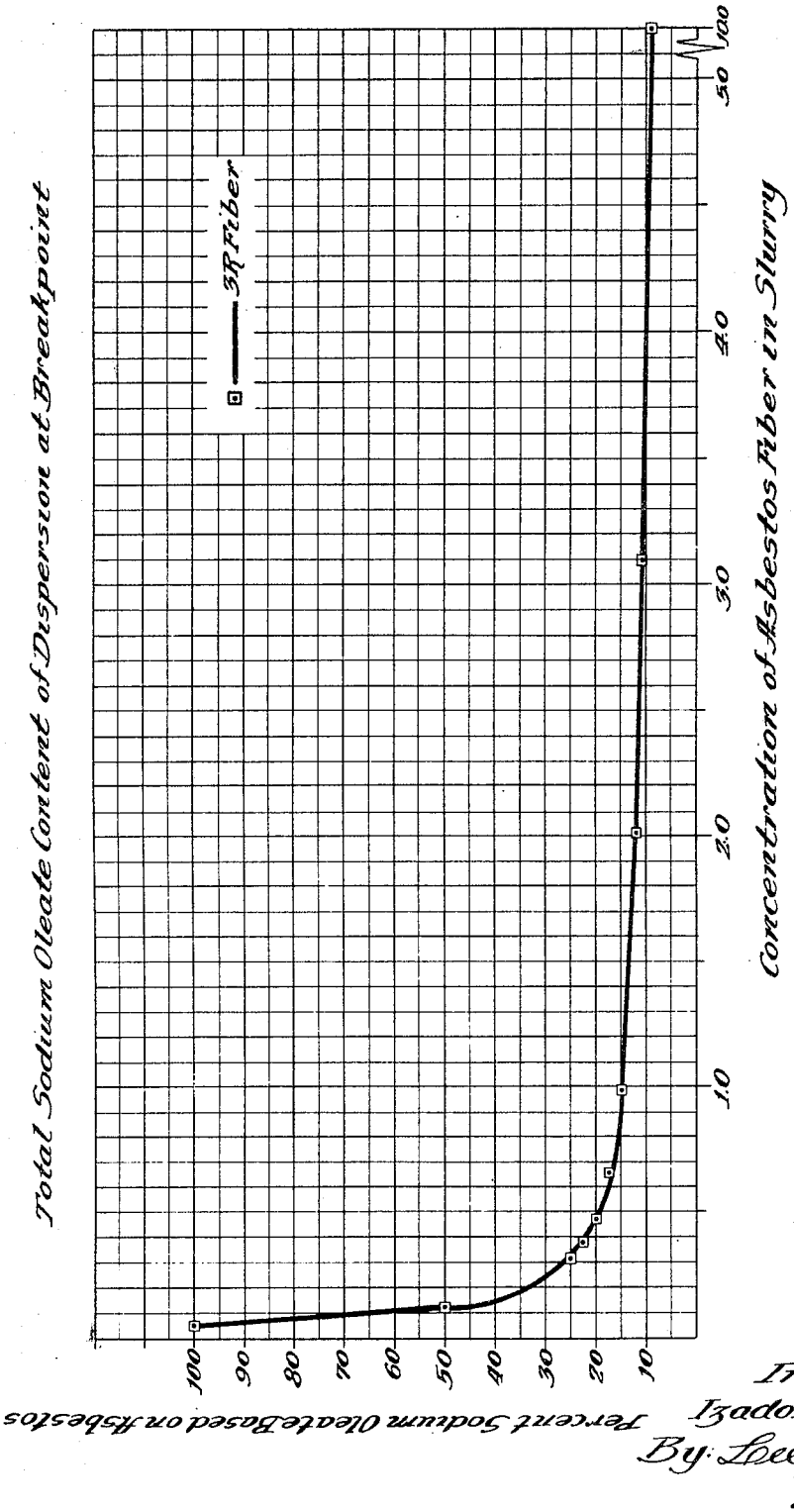


FIG. 6.

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UNITED STATES PATENT OFFICE

2,626,213

ASBESTOS DISPERSIONS AND METHOD OF FORMING SAME

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Application December 21, 1948, Serial No. 66,552

14 Claims. (Cl. 92—3)

1

This application is a continuation-in-part of my co-pending application, Serial No. 786,159, filed November 14, 1947, now abandoned.

This invention relates to a novel means for opening up or disintegrating either large or small tightly-bound macroscopic aggregates or bundles of asbestos to produce therefrom a high and hitherto commercially unattainable yield of fibers of novel and modified character and of extremely small cross-section or diameter while maintaining a high length-diameter ratio.

The invention further relates to the separating, individualizing, or disseminating fibers of asbestos to produce alkaline, gelatinous, stable, aqueous dispersions of asbestos of colloidal or "fibro-colloidal" character.

The invention further relates to the production of asbestos fiber of novel character and properties, and wherein the fiber surfaces are substantially modified by organic material adsorbed and/or fixed thereto.

The invention further relates to the production of bodies of fibrous asbestos wherein the majority of the fibers are of "unit-size" or "mono-crystalline" range, that is, individuals or single fibers of diameters between about 200 and 500 angstroms. At the same time these unit fibers may be of at least average paper-making fiber length, and if desired they may be of average spinning fiber length such as ⅛ inch in length and upwards, and are further characterized by their smooth, level character.

The process of my invention is particularly adapted to disintegrate and disperse the fibers of asbestos which is essentially hydrated magnesium silicate occurring in aggregates or bundles of fine, crystalline, silky fibers, such as asbestos of the chrysotile variety, and I may employ unfiberized or partially fiberized material capable of longitudinal or lateral subdivision, such as, natural rock masses, crushed rock, splints, spicules, or mechanically or disruptively-opened and fiberized asbestos material.

The value of asbestos lies primarily in the length of its fibers and the ability to produce therefrom various materials such as yarn, cloth, felt, tape, paper and many other specific products well known in the art. However, prior to the present invention, the ability to obtain not only increased production of uniformly long fibers of spinning grade, but fibers of uniformly fine cross-section and the ability to produce fine, level yarns and woven fabrics and felted fibrous materials of a relatively smooth and even surface character and of a relatively thin nature and high strength

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was a matter to be desired, but generally unattainable. The products heretofore produced of asbestos material were generally and of necessity of a coarse, uneven, relatively thick character because of the presence of a large proportion of unopened fiber masses and the agglomerated, jackstraw characteristic of asbestos, dry or in water.

The method of producing asbestos fibers from the asbestos ore generally comprises a series of crushing and winnowing operations whereby the fibrous rock is subjected to mechanical pressure and shock, and during these operations the fiber is carried away by air currents and the associated rock is discarded. It will be seen that during operations of this nature the asbestos fiber is also crushed and broken, being relatively much softer than the associated rock. The result is that, out of 100 parts of available fiber produced from Canadian chrysotile mines, only about 1% is removed by hand-cobbing to produce #1 and #2 grades of "crude" of from about ⅜ to 1 inch or more in length, i. e. highest spinning grades. The remainder is mill fiber, divided according to use, in accordance with standard Canadian screen tests into long spinning fiber (for example, 3R grade), medium spinning and compressed sheet fiber (as, for example, 3Z grade), pipe covering fiber, shingle stock (4T grade), paper and millboard stock (for example, XX grade or 5R), and cement stock (7D or "C" grade). Of this mill fiber, about 12% to 15% is made up of fibers of from ⅛ to 1 inch, generally termed "spinning grades"; from 50% to 75% is composed of fibers ranging from about ⅛ inch down, known as paper-making fiber, including the grades of medium and compressed sheet fiber, pipe covering fiber, shingle stock, and paper and millboard stock. The balance is impalpable dust which includes the grade of cement stock and shorts.

Furthermore, during the preparation of such fibrous material, for example, for spinning, the various operations concerned with the production of fibers more uniform in diameter than that produced at the mines require further mechanical operations, such as crushing between rolls, pulverizing in hammer-mills, and other tearing and disintegrating processes followed by eventual carding which, in itself, is actually a disintegrating operation, and all for the purpose of reducing macroscopic bundles to thinner fibers. Thus, losses of fiber quality as regards length have heretofore been concomitant with the effort to reduce the diameter of the unopened fiber below certain limits for the purpose of making smoother products.

It is recognized that the condition of the fiber in the original vein is mainly that of parallel columnar crystals or cells having extremely fine diameter and cross-section and great length compared to diameter, and that they are extremely close together, held in position by molecular forces, probably secondary valence forces. When these asbestos masses are subjected to mechanical or other disruptive force, they break into smaller masses having the same columnar or fibrous characteristics, but of diameter representing widely varying numbers of unit cells. The cross-section unit cell, according to recent research, is of the order of 9 to 15 angstroms or less than 1/500 of a micron. The smallest diameters revealed by electron microscopic photographs of mechanically subdivided fibers show fiber diameters of the order of 50 to 500 angstroms.

According to my method hereinafter described, I have been able to obtain asbestos containing a preponderate number of fibers in the colloidal diameter range, which is considered roughly from 5000 angstroms down to about 10 angstroms without using the prior attrition methods for disruption of the macro fiber bundles.

In the accompanying drawings, Figures 1 and 2 are electron microphotographs of spinning grades of asbestos fibers opened and dispersed in accordance with my invention.

Figures 3 and 4 are graphs illustrating the minimum percentages of treating agents required for preparing and maintaining asbestos fiber in dispersed condition in accordance with my invention, wherein the per cent of treating agent in a slurry is plotted against the per cent asbestos in the slurry.

Figures 5 and 6 are graphs illustrating the minimum percentages of treating agents required for preparing and maintaining asbestos fiber in dispersed condition in accordance with my invention, wherein the per cent treating agent based on asbestos is plotted against the concentration of asbestos fiber in the slurry.

In general, the method of my invention comprises the opening and dispersing of macroscopic bundles or agglomerates of chrysotile asbestos fibers of the clases hereinbefore described to separate, individualize and disseminate the fibers and to produce therefrom an alkaline, gelatinous, stable, aqueous dispersion composed predominantly of fibro-colloidal asbestos fibers by treating said macroscopic fiber bundles with an aqueous solution of organic detergent surface-active material adsorbable on and thereby effective to disrupt the molecular bond contained in the asbestos fibers, while employing an excess of the same or another such agent in a proportion adequate to maintain or preserve the opened fibers floating in an individualized state in a fluid dispersion. The agent employed reacts with, or is adsorbed on and substantively modifies the asbestos fibers and, in some manner which is not fully understood, at the same time opens up the macroscopic fiber bundles; and since it has been observed that this action is progressive, an adequate amount of the agent should be present in the liquid phase so as to satisfy the progressively increasing number of surfaces developed. The excess surface-active agent should be present in the remaining free liquid for the purpose of sustaining the resultant increased number of fibers in the aforesaid dispersion. As is evident from the foregoing, these surface active agents are actually colloidizing agents for asbestos in an aqueous vehicle since they reduce the asbestos to colloidal size particles and hold said particles in colloidal dispersion.

The amount of agent required by the asbestos material for the production of my novel and characteristic dispersion is further graphically illustrated by the accompanying drawings, whereof Figures 3 and 4 show the surface-active material requirement for a range of asbestos concentrations in the case of spinning grade fiber (3R) and indicate that the proportion of surface-active agent is related to the quantity of asbestos in the dispersion in a very definite manner as shown by a straight line relationship. The area above the solid line represents proportions characteristic of the dispersed condition, while that below this line represents proportions which do not provide dispersions but rather the coagulated or clotted condition.

From the fact that the line intersects the vertical axis at 0 asbestos concentration it is seen that a certain definite quantity of surface-active agent is required to be present in the liquid before dispersion (as distinguished from opening) can occur. The distance between the horizontal dotted line upwards to the solid line at any point horizontally represents the amount of surface-active agent which is required by that amount of asbestos and adsorbed thereon to place the fiber in condition for dispersion.

I have assumed for the purpose of simplicity that the two functions of the detergent are divided as shown by the broken horizontal line, but it may well be that the division should be represented by a line curving upward slightly. In any case, the two functions are certainly present, and the broken line as shown is intended to be diagrammatic.

The straight line curve is different for each detergent as exemplified by the difference between Figures 3 and 4. Figure 3 relates to Aerosol OT (dioctyl sodium sulfosuccinate), a very active agent, and Figure 4 relates to sodium oleate, a less active agent. However, the explanation above is similarly effective.

Inasmuch as the straight line curve shown represents the boundary between the coagulated and the dispersed condition and this boundary, because of the nature of fiber dispersions, is not a sharp one, it is always desirable, in producing dispersions for subsequent mechanical handling, to operate well above the boundary so that changes which affect the boundary may not damage the dispersion. Such changes may be temperature, ageing resulting in further opening of the fibers, air bubble entrapment which tends to clot fibers, and metallic or other contaminations; in all of which cases a substantial safety factor is desirable. This safety factor in practice may be of the order of 25% to 100% or even more.

From electron micrographs of my opened fiber, there seems to be a range of unit or single fiber diameters between about 200 and 500 angstroms, although there may be an occasional fiber as small as 50 angstroms in diameter. Some fiber masses of larger diameter which still remain incompletely subdivided in my processed fiber can usually be seen to be composed of unit fibers of the above stated diameter range.

Accompanying Figures 1 and 2 are electron microphotographs of Canadian chrysotile asbestos opened and dispersed and diluted to an .05% slurry in accordance with my invention, Figure 1 being of one specimen magnified 19,500 times

and Figure 2, another specimen, magnified 25,800 times.

The fiber length as shown in the electron micrographs of my opened fiber is at least 200 times the unit diameter within the field shown in the micrograph, and from external evidence, such as tearing or stretching felted fiber masses, is probably of the order of several thousand to several hundred thousand times the diameter, of course dependent on the original lengths of fiber present in the unopened asbestos, and also on the amount of mechanical working of the dispersion to homogenize it for processing. The reason for the definite diameter size range of the unit fibers found in these electron micrographs is not yet understood, since the unit cell determined by X-ray diffraction studies is very much smaller (a=14.66 Å., b=9.24 Å., c=5.33 Å., according to B. E. Warren), but this range is characteristic of Canadian crysotile asbestos both in my dispersions and in undispersed condition and it will therefore be understood that the terms "unit," "unit-size" or "monocrystalline," "individualized," "fibro-colloidal" diameter as employed in this specification and the following claims, refer to fibers which apparently cannot be further opened or laterally subdivided and which have diameters of from about 200 to 500 angstroms and appear as "singles" in the electron micrographs. As a comparison, the lower diameter specified is also close to that of rods of tobacco mosaic virus shown in electron micrographs.

My dispersion, as evidenced by these electron micrographs, shows that the unit fibers are individualized and separated from one another, that they are substantially clean and smooth which I believe is due to the repulsive effect of the similar charges which they carry, and they do not therefore attract small fiber fragments. To this fiber character I attribute the fact that they slip readily past each other and, in general, can act as individuals.

Of course, there may also be present bundles of fibers of smaller and larger numbers which have not been reduced to fibers of unit range, which will, of course, modify the flow characteristics and product qualities, but these bundles also have the repulsive charge and the general properties of the unit fiber. However, even though the individual fibers may be separated and repellent, their great length often will tend to keep some bundles from separating fully or easily because of some tangling and intertwining. Thus, there commonly exist both unit fibers and bundles in my dispersion, but both types are individualized and separated and because of their smooth surfaces show a minimal tendency to tangle and clot. However, there is always a majority by number of long, not fragmentary unit fibers in a well-made dispersion and which characterizes my treated asbestos mass, as distinguished from the much smaller content of such small diameter fibers which may be present in commercially known masses. For my purposes, I do not consider very short bits as fibers. As an evidence of the high degree of subdivision I obtain, I have made comparative freeness tests in the standard manner using 3 grams of asbestos in 1,000 c. c. of aqueous liquid on a Williams freeness tester.

Straight XX asbestos paper-making fiber shows a freeness of 415 seconds in water. Straight "C" asbestos paper-making fiber (a very short grade) shows a freeness of 265 seconds in water.

Using the same fibers dispersed in accordance with my invention with Aerosol OT the freenesses were, for XX, 69,600 seconds, and for "C" or 7D grade, 64,500 seconds.

With sodium oleate soap similarly, XX, 54,000 seconds, "C" or 7D grade, 22,800 seconds.

Similar comparison cannot be made in the case of spinning grade fibers since they do not disperse in water but form clots without my treatment.

This tremendous difference in freeness is due to the much greater number and much greater fineness of the fibers developed by this treatment, since the effect of the very dilute wetting agent content on the viscosity of water is negligible.

The viscosities of some of my dispersions were determined by employing a Bakelite viscosity tube. In this instrument viscosity is determined by timing the flow, at a temperature of 25° C., through a tube of a calibrated amount of liquid, in this case my aqueous asbestos dispersions. The results are given in the following table, XX grade or 5R fibers being employed in each case:

Wetting agent	Percent asbestos in slurry	Percent wetting agent on asbestos	Time, seconds	Viscosity in centipoises
Aerosol OT	5.0	35	170	965.0
Sodium oleate	5.0	50	9.3	52.8
Aerosol OT	.375	35	3.9	22.1
Sodium oleate	.375	50	3.8	21.6

Even with all the prior developments in opening asbestos fiber, there has been no practical way, or no way, which does not involve excessive losses, to produce unit asbestos fiber which is largely of colloidal diameters and of length adequate for spinning yarn for fine woven tape or cloth. Thus, in yarn there was heretofore considered to be a commercial limit at approximately 2500 yards per pound. In paper, anything thinner than .003 to .004 inch has been so weak (without large amounts of binder) that it is useless. In other words, the inability to produce uniform, fine, long asbestos fibers has sharply delimited progress towards fine fiber products such as exist in similar fashion in the case of silk or cellulose fibers, both of which are known to have fiber diameters considerably greater (10 to 15μ) than that of the unit asbestos fiber (.02 to .05μ). The finest glass fibers have a diameter of 0.5μ. By this present method there is now available a supply of very long staple uniform fiber, 10 to 25 times as fine as previously known, and thus products proportionately fine can now be produced therefrom. I have now been able, with the fine fiber of this invention, to make asbestos yarn running 10,000 yards per pound and smooth, strong, tough asbestos tissue as thin as one quarter thousandth inch. Still finer structures are indicated by the relative ease of production of the above specified dimensions.

As distinguished from mechanical or other disruptive fiberizing methods, I have thus found that it is possible to produce asbestos fiber of extremely great transverse subdivisions and fine structure, while at the same time retaining good fiber length of the material subjected to treatment, by exposing asbestos material of the class and character hereinbefore described to the action of dilute aqueous solutions or dispersions of certain detergent or surface-active organic colloidal materials which I have found to have wetting and dispersing action on the asbestos to produce therefrom fluid gelatinous dispersion of

fibers, largely of a colloidal character with the fibers largely of unit diameter.

These surface-active agents are generally of the classes which have been used elsewhere for detergents, wetting agents, foaming agents, penetrating agents, or emulsifying agents, and which I have found to be reactive with or adsorbable on the asbestos fibers. Those suitable for my purpose produce alkaline dispersions with asbestos usually in the pH range of 8 to 11 where no intentional change of pH is made by addition of other agents. In this pH range there is no corrosion of the fibers as the normal pH of asbestos fibers in water lies within the range.

Among the reagents which I have found to be useful for both opening asbestos fibers and maintaining them in stable dispersion are many compounds containing SO_3 groups, such as sulfated and sulfonated long chain carbon compounds, e. g., sulfated or sulfonated esters, alcohol sulfates, alkyl aryl sulfonates, fatty acid derivatives, and hydrocarbon sulfonates. Others are included in the phosphated organic compounds, amine derivatives, and fatty acid soaps. In the tables appearing hereinafter, those agents having the lowest breakpoint number are most effective for my purpose since the breakpoint is the minimum percentage concentration of asbestos at a constant detergent:asbestos proportion at which any given dispersion is stable.

While the use of acid reagents to produce acid dispersions of positively charged asbestos fibers has been suggested for deflocculation of short or paper-making asbestos fiber, such materials are not only ineffectual for opening up macroscopic bundles of both short and long fibers, but are corrosive of the fibers in that solution of Mg, Fe and SiO_2 takes place until the acid is neutralized by the basic asbestos. At best, such acidic reagents form non-gelatinous, transitory, short-lived suspensions of finely flocced fiber which settle rapidly on standing rather than true stable dispersions, and which revert to visibly clotted condition even before the acid is neutralized, and in rapidly deteriorating quality as the acid reacts. On the other hand, my dispersions on the alkaline side are relatively viscous, gelatinous, indefinitely stable, and may be stored for long periods before use without deterioration, clotting or coagulation. They form transparent films between the fingers on handling, even when dilute.

The following is a list of detergent or surface-active materials which I have found to be suitable for both opening and dispersion of asbestos, in accordance with my invention, although it will be understood thtat they are not all fully equivalent, that is, the effect of some may be more rapid than others, the concentrations required to produce equivalent effects may vary, and the like.

Supplier	Trade name	Active ingredient	Break-point	pH 2.5% slurry
American Cyanamid	Aerosol OT	Dioctyl ester of sodium sulfosuccinate	0.078	9.6
Gen. Dyestuff	Nekal N. S	Na trialkyl sulfotricarboxylate	.117	9.8
National Aniline	Nacconal L. A. L	Na lorolsulfoacetate	.17	9.5
Titan	Titazole A. E. Conc	Sulfonated aliphatic ester	.13	10.0
American Aniline	Featherwet	Sulfonated organic ester	.22	10.9
Alrose	Alrosene 31	Coconut fatty acid sulfate	.17	8.9
Carbide & Carbon	Tergitol 07	Na sulfate der. of 3,9 diethyltridecanol-6	.18	8.3
Du Pont	Duponol D	Mixed alcohol sulfate	.20	9.4
Do	Duponol G	Higher alcohol sulfate	.08	9.7
Do	Duponol L. S	Oleyl alcohol sulfate	.08	8.5
Do	Duponol W. A. T	Triethanolamine sulfate	.09	8.3
Emulsol	Emcol X-25	Alkanolamine of sulfated complex alcohol	.11	9.1
Laurel	Supersulphate F. S	Aryl alcohol sulfate	.18	9.6
Procter & Gamble	Orvus W. A	Sodium lauryl sulfate	.12	9.1
Rohm & Haas	Triton 770	Aryl polyether sulfate	.11	9.6
Armours	Synthetic Detergent 422	Alkylaryl sulfonate	.12	10.6
Atlantic	Ultrawet E	Dodecyl benzene sulfonate (Tot. C's=12)	.17	9.2
Do	Ultrawet K	Hexadecyl benzene sulfonate (Tot. C's=22)	.14	9.2
Burkart Scheir	Aromline PM	Alkylated aromatic sulfonate	.09	10.5
Commonwealth	Unitex	do	.185	9.3
Houghton	Cerfax	Alkylaryl sulfonate	.23	8.8
Jacques Wolf	Wetsit	Alkylated aromatic sulfonate	.16	9.5
Monsanto	Santomerse #3	Na dodecylbenzene sulfonate	.17	9.5
Do	Santomerse B	Na m-lauryl benzoate sulfonate	.21	8.7
National Aniline	Nacconal N. R	Kerylbenzene sulfonate	.15	9.2
Do	Nacconal E. P	Alkylarylsulfonate	.17	8.4
Do	Nacconal F. S. N. R	Kerylbenzene sulfonate	.24	8.9
Oronite	Detergent D-40	Alkyaryl sulfonate	.16	9.6
Rohm & Haas	Triton 720	Dibutylbenzene polyether sulfonate	.23	9.6
Titan	Titanole R. M. A	Alkylated aromatic sulfonate	.08	9.5
Ultra	Sulframine AB	Dodecylxylene sulfonate	.10	10.0
Alrose	Arogel	Fatty amide sulfate	.08	9.1
Armours	Neo-Fat D-142	40% Na oleate, 50% Na linoleate, 10% sat. acids	.15	9.8
Do	Neo-Fat #3	50% Na oleate, 40% Na linoleate, 4% Na linolenate, 6% Na rosinate.	.13	9.8
Colgate	Arctic Syntex A	Sulfated ethylene glycol ester of oleic acid	.15	8.2
Do	Arctic Syntex T	Fatty acid amide sulfonate	.14	8.9
Carlstadt	Caramide	Sulfonated fatty amide	.09	8.3
E. F. Drew	Quix	Sulfonated fatty acid amides	.09	8.2
Eimer & Amend	Sodium Oleate	Na oleate	.12	9.8
Hart	Bozetol	Sulfonated castor oil	.13	9.3
Do	Hytergon BM	Sulfated fatty acid amide	.10	9.4
General Dyestuff	Igepon A. P. Extra	Sulfonated oleyl ester	.10	8.1
Nopco	Nopco 2272 R	Sulfated fatty ester	.12	9.7
Onyx	Phil-O-Sol WA	Disulfonated fatty ester	.18	8.8
Do	Xynomine Paste	Sulfonated fatty acid condensate	.09	8.7
Ciba	Detergent M	Amine condensation product	.25	8.6
American Aniline	Orthoscour	Long chain alkyl sulfonate	.25	9.5
Commonwealth	Detanol A	Hydrocarbon sulfonate	.16	8.0
Du Pont	M. P. 189	do	.11	9.4
Alrose	Alrophil G	Synthetic detergent	.12	11.9
Riches-Nelson	Fluid Gel	Organic anionic agent	.11	12.1
Ultra	Ultrapone D. L	Amine condensate	.20	9.4
Do	Ultrapone S	do	.18	9.6
Warwick	Horn Kem 3G	Dialyzed lignin sulphonic acid	.20	8.7
Alrose	Alrosene N. C	Modified alcohol sulfate	.50	8.2
Do	Alropon	Sodium salt of secondary alcohol sulfate	.50	9.2
Du Pont	Avitex C	Modified alcohol sulfate	.50	10.0
Do	Duponol W. S	Long chain alcohol sulfate	.50	9.8
Do	Duponol W. A	Sodium lauryl sulfate	.31	9.7
Onyx	Mapro Degum A	Sodium cetyl sulfate	.32	10.0

Supplier	Trade name	Active ingredient	Breakpoint	pH 2.5% slurry
Riches-Nelson	R. N. 31	Modified alcohol sulfate	.50	8.9
Do	R. N. 600	Sodium salt of secondary alcohol sulfate	.42	9.5
Armours	Regal	Alkylaryl sulfonate	.31	9.5
Beacon	Beaconal S	Monoethylphenyl phenolaminomonosulfonate	.47	8.8
Du Pont	Alkanol W, X. N	Petroleum alkyl sodium sulfonate	.48	9.8
National Aniline	Nacconal F. S. N. O	Alkylaryl sulfonate	.34	9.8
Riches-Nelson	Spray Dried Beads	do	.33	10.0
Amalgamated	Alkamine W	Fatty Alkanolamide	.42	8.1
Arnold Hoffman	Ahcowet R S	Sulfonated fatty acid ester	.34	8.6
Commonwealth	Cominol	Sulfonated higher fatty amide	.31	8.1
Michel	Michelene DCA	Alkylamido sulfate	.55	8.6
Synthetic	Janusol	Ammonated and sulfated lauryl and myristyl esters	.46	8.5
Ultra	Sulframin DT	Alkyl amide sulfate	.60	8.9
Do	Sulframin LW	do	.40	9.0
American Cyanamid	Aerosol AY	Na diamyl sulfosuccinate	>2.5	9.0
Do	Aerosol MA	Na dimethylamyl sulfosuccinate	1.7	8.3
Do	Aerosol 18	N octadecyl disodium sulfosuccinamate	>2.5	9.7
Alrose	Alrosene P. D	Modified alcohol sulfate	1.25	8.3
Amalgamated	Dynesol D-50	Sulfated naphthalene ester	.70	10.2
Commonwealth	Lightning Penetrator XW	Sulfated higher alcohol	.77	9.3
Carbide & Carbon	Tergitol 04	Na der. 7-ethyl,2-methyl undecanol-4	.77	8.5
Du Pont	Avitex AD	Special sulfated oleyl alcohol	96	10.0
Do	Duponol ME Dry	Lauryl alcohol sulfate	2.0	10.2
Do	Avitex W	Partially sulfated stearyl alcohol	1.00	9.6
Do	Duponol 80	Octyl alcohol sulfate	>2.5	9.7
Laurel	Supersulphate NT	Sulfated alcohols and sulfonated oils	>2.5	9.2
Onyx	Maprofix Paste	Sulfated cetyl alcohol	>2.5	10.3
Do	Maprofix New Powder	Tech. Na lauryl sulfate	.83	9.8
Rohm & Haas	Triton W-30	Na alkylated aryl polyether sulfate	.79	9.9
Alrose	Rinsynol 50L	Na alkylnaphthalene sulfonate	>2.5	9.1
Commonwealth	Soapotol	Sulfonated fatty amide	.31	8.5
Arnold Hoffman	Ahcowet ANS	Alkylnaphthalene sulfonate	>2.5	9.3
Do	Detergent 240	do	>2.5	9.0
Du Pont	Merpentine	do	1.0	9.8
General Dyestuff	Nekal BX	Na diisobutyl naphtahalene sulfonate	>2.5	9.1
Do	Corikal B	Na alkylnaphthalene sulfonate	>2.5	9.3
J. Wolf	Sellogen A. S. D	do	1.25	9.4
American Aniline	Orthowet	Na alkyl aryl sulfonate	1.25	9.1
Burkart Schier	Penetrant M7K	do	.70	9.3
Charlotte Chem. Labs	Sontal S	Substituted aromatic sulfonic acid	.72	9.5
Emulsol	Midonhate	Complex Na aryl sulfonate	1.00	8.9
Monsanto	Aresklene 400	Dibutyl phenylphenol disodium disulfonate	>2.5	8.2
Do	Santomerse D	Na decyl benzene sulfonate	>2.5	9.0
Nopco	Nonco 1067	Sulfonated alkylated aromatic	1.0	8.0
Riches-Nelson	RN-35	Alkylaryl sulfonate	>2.5	10.0
Colgate	Arctic Syntex M	Sulfated coconut fatty acid ester	1.1	10.0
E. F. Drew	Quixite	Sulfonated fatty acid amide	1.0	9.0
Emery Industries	Twitchell Oil 7250	Sulfated fatty ester	.80	9.3
Eimer & Amend	Na Stearate	Na stearate	>2.5	9.5
General Dyestuff	Cyclonon A	Fatty acid dialkylamide monosulfonate	.84	8.7
Onyx	Stearyl Glyceryl Sul	Sulfated tallow	1.4	9.4
Procter & Gamble	Ivory	Na oleates, stearates, and palmitates	1.0	9.5
Ultra	Sulframin DH	Alkylamide sulfates	.8	9.8
Do	Sulframin DR	do	1.5	8.6
Amalgamated	Alkamine C-30	Fatty alkanolamide derivative	.08	10.7
Alrose	Alrosol	Fatty alkanolamide condensate	.10	9.4
Do	Alrosol O	do	.15	9.4
Riches-Nelson	RN-600	Fatty amide derivative	.26	10.0
Alrose	Alrosol C	Fatty acid amide type	1.25	9.9
Do	Arosol S	do	.95	10.0
Do	Nonisol 110	Polyoxyalkylene difatty ester	>2.5	9.2
Do	Nonisol 200	Polyoxyalkylene ester of oleic acid	>2.5	9.5
Do	Nonisol 300	Polyoxyalkylene ester of stearic acid	>2.5	9.4
Do	Span 40	Sorbitan monopalmitate	>2.5	9.2
Atlas	Atlas 1162	Modified stearic acid ester	>2.5	8.3
Do	Emargol	Mono and diglycerides and sulfoacetates	2.5	9.0
Emulsol	Emcol SPN	Hydrophilic fatty esters	2.5	10.0
Do	Emcol RDC-D	Diethylene glycol laurate	2.0	10.0
Do	Aldo 28	Glyceryl monostearate plus soap	2.5	10.0
Glyco	Carbowax Dioleate 1500	Dodecaethylene glycol dioleate	>2.5	9.4
Do	Diglycol Laurate S	Diethylene glycol laurate plus soap	2.0	8.7
Do	Diglycol Stearate	Diglycol stearate	>2.5	7.7
Do	Victamul 67	Long chain alkyl ether phosphate	>2.5	8.5
Victor	Victamul 89	do	1.8	8.5
Do	Charlab Condensate	Condensed amine laurate	.08	11.5
Charlotte Chem. Labs	Nopco 1179 R	Fatty amine	.10	9.2
Nopco	Janusol	Lauryl and myristic esters having amino and sulfate groups.	.46	8.5
Synthetic	Victamine C	Amino amido phosphate ester	.74	8.1
Victor				

The breakpoint shown above is obtained by diluting with water a homogeneous dispersion of asbestos with a detergent to the point where the slurry becomes non-homogeneous or clotted. For the above tests a 2.5% asbestos slurry was used containing 50% by weight of detergent on the asbestos. The magnitude of the figure is inversely proportional to the effectiveness of the detergent, the lower figures indicating the most active materials, and the higher figures the less active ones.

In general, my process comprises subjecting the asbestos material to be opened and dispersed to an aqueous solution or dispersion of a detergent organic surface-active agent having a low degree of water solubility which is of the character of the materials listed above. Depending on the temperature, concentration and type or class of agent, specific asbestos material and source and mixing means, the time of treatment may vary from, say, one-half hour to about a week. The action is progressive from the exterior to the interior of the asbestos fiber bundle with progressive lateral subdivision of the fiber bundles, and with molecules of the surface-active agent apparently becoming adsorbed by, oriented on and fixed to the surfaces of the fibers. I believe that the action is a combination of physical and chemical forces whereby the molecular attraction between the crystalline fibers is broken and the fiber surfaces substantively modified by an adsorbed layer. This is evident from the gelatinous nature of the resulting product and a group of properties characteristic of stable colloidal dispersions.

As a more detailed modification of this theory, it may be that the surface-active agent first penetrates between the fibers and there causes them to part from each other by the interposition of a surface layer of the less soluble magnesium compound of the wetting agent which acts to overcome the intermolecular forces and separate the fibers individually. Also, many of the effective surface-active agents I have selected herein are precipitated as their magnesium compounds from solutions of the strengths suitable for rapid opening of asbestos by solutions of magnesium chloride provided they do not contain a "sequestering agent," for example sodium hexametaphosphate or some other agent adapted to prevent precipitation of a magnesium or calcium curd in ordinary detergent use. Both of these observations lend evidence to the theory of penetration followed by surface precipitation on the asbestos fiber, which is known to react chemically like magnesium hydroxide.

It may also be that there is a balance between precipitation and solubilization of the magnesium compound of the opening agent or rate of precipitation, or a selective range of solubility of the magnesium compound of the opening agent which is necessary for adequate separation of the fibers from one another, since I have noted that the so-called "sequestering" agents for calcium and magnesium (Calgon—a glassy pyro phosphate) which solubilizes magnesium, and carbon dioxide, which forms the very insoluble magnesium carbonate, both hinder the opening of asbestos strongly, when present in large proportions, in the presence of Aerosol OT which by itself opens the asbestos readily.

According to the best theories of asbestos structure, it carries on its outer surface magnesium atoms attached to oxygen and hydroxyl, and therefore shows the surface properties of magnesium oxide and magnesium hydroxide. It is believed that asbestos reacts chemically with the wetting agent to form an initial surface layer at least of the anion of the wetting agent attached to the magnesium in the asbestos surface. In corroboration of this, I have found that there is a small proportion of organic matter which cannot be extracted from my dispersed asbestos by any solvent which does not destroy the asbestos. This organic matter is of the order of 1% of the total weight of the dispersed asbestos, and varies as the exposed surface. Oxygenated solvents such as alcohols, ketones and ethers are usually suitable for removing adsorbed but unfixed wetting agent and the unfixed Mg salt of the wetting agent, which latter may form by reaction with the Mg ions in solution, previously dissolved out of the asbestos by the aqueous medium.

Thus, according to these theories and observations, it appears that the Mg atoms in the asbestos fiber surface attract and hold the hydrophilic end of the wetting agent molecule, usually the inorganic end, the chains of the molecule orienting themselves towards the surface at active Mg atoms, and additional molecules of wetting agent fill the spaces in between these oriented chains, orienting themselves with the hydrophilic end outward, held by Van der Waals forces. The substitution of this system for the secondary valence forces between unit fibers splits them permanently apart. This produces a structure having an overall negative charge, and dispersoid in character in a suitable liquid medium. It is believed that these fibers have a lubricated and spaced relationship in the dispersed condition because the fiber together with a layer attracted to the fiber and more or less immobilized by attractive forces carries a net electric charge. In the case of my dispersions the sign of this electric charge, as indicated by precipitation reactions and by migration in an electric field, is invariably negative.

According to established principles of electrostatics the attached negative charge will attract the positive ions present in the surrounding electrolyte and also repel the negative charges present therein, so that a compensating layer of positive charge density will surround the particle. According to accepted principles of kinetic theory the compensating layer will retain a mean thickness which depends on the concentrations and charges of ions present; the expected thickness would be 20 angstrom units for 1% aerosol, and 62 angstrom units for 0.1% aerosol. When fibers approach each other within some small multiple of the screening distance, the electrostatic repulsion comes into play.

A carefully checked analysis of a Canadian chrysotile spinning fiber (Bell's Asbestos Mines 3R) shows the following composition:

	Percent
SiO_2	38.8
R_2O_3 (Fe, Al, Cr)	8.5
MgO	39.6
Volatile (H_2O)	13.3

After treatment with Aerosol OT in 1% aqueous solution, the water-washed fiber contained 11.8% methyl alcohol extractable material which was essentially Aerosol OT and the magnesium salt of Aerosol OT, and 88.2% of opened asbestos and non-extractable organic matter, which comprises the anion of Aerosol OT in chemical combination with the surface Mg atoms of the asbestos. The methyl alcohol extract was 11.05% organic and .75% inorganic containing the following: Fe, Si, Mg, SO_4 and Na.

The methyl alcohol extracted fiber had the following composition:

	Percent
SiO_2	38.3
R_2O_3	8.0
MgO	39.6
Volatile	13.9
SO_4	0.1

The organic volatile which is the fixed Aerosol OT anion was 0.6% of the total weight. It is thus seen that there is essentially no proportional change in the inorganic composition of the asbestos after treatment, and that a small proportion of the wetting agent becomes fixed to the asbestos surface. A small proportion of the asbestos dissolves in the aqueous opening medium and some of this is removed by the methyl alcohol extraction together with the Aerosol OT and its Mg salt.

In general, stable dispersions of my asbestos solids in liquids have the following properties:

1. The fibers do not adhere, but repel each other because of electric charges attached to them; in this case the charges are electro-negative.

2. Most of the fibers are of small diameter within the colloidal range 10 Å. to 5000 Å.; in this case, from electron micrographs, there seems to be an average unit fiber transverse diameter size between 200 and 500 Å. Most unit fibers are longer than the field of the micrograph. They are very stiff, as evidenced by their slight curva-

ture. The background contains small unassociated particles of fiber.

3. The fibers remain suspended in the liquid phase long periods and do not coalesce or coagulate. Supernatant liquid is cloudy because of very fine suspended solid matter.

4. The fibers exhibit electrophoretic properties, that is, they will migrate in an electrostatic field, to the anode in this case.

5. The area of the fibers in a treated mass of asbestos of a given weight, as in any dispersion of a solid, is much greater than in the case of a mixture of liquid and purely mechanically opened fiber. I estimate the total area of my dispersed fiber to be at least ten times that of commercial mill fiber when the latter is used in its preparation.

6. A smooth dispersion of my asbestos has a uniform gray appearance and a pearly shimmer when in motion.

7. It has gelatinous flow and high viscosity for solids content, the latter varying with the method of measurement.

As distinguished from my dispersed fiber, asbestos material and particularly asbestos fibers in undispersed or coagulated condition in liquid shows the following properties:

1. Flocking of fiber masses with clear liquid between.

2. The unit fibers in partial bundles adhere in more or less definite orientation parallel to one another and the loose fibers are coagulated or flocculated in jack-straw condition.

3. The fibers have small charge, if any, and attract each other.

4. The color in liquid is whiter—more light scattering. There is no pearly shimmer when in motion.

5. They are much less electrophoretic because of much lower charge and larger masses.

6. There is rapid settling, giving supernatant liquid relatively free of fiber.

7. Electron micrograph of fibers of small diameter shows jack-straw appearance with fibers adhering to form an uneven surface, and tangling together, and background clear of small fiber bits.

The following examples are illustrative of my invention, although they are not to be considered in limitation thereof:

Example 1

Specimens of rock asbestos of the Canadian chrysotile variety of about 2 inches in length and of about ¼ inch by ¼ inch in cross-section were immersed and completely covered by fifteen times their weight of a 1% aqueous solution of dioctyl sodium sulfosuccinate (Aerosol OT) at room temperature. When the pieces were first immersed, they were characteristically dark green or almost black, relatively free of fibers on their surfaces and of a coarse, hard and stonelike nature. Within a short period of time, a striking change occurred, namely, the development on the exposed surfaces of a silvery sheen with a bluish tint. On disturbing these surfaces, it was noticed that the silvery sheen was due to the formation of loose fiber which could easily be wiped from the hard rock beneath. As the dispersion continued, the loose fiber increased in quantity and the hard rock portion decreased in size until after a few days all of the rock particles had become softened and only loose parallel fibers remained in the form of a swollen gelatinous mass. The only material which did not become softened was the associated impurities, such as the naturally associated serpentine or magnetite. The mass of fibers so produced was highly gelatinous, held water strongly, was of an extremely slimy nature and had strong colloidal jellylike characteristics.

When the specimens of rock had been left undisturbed, it was noticed at the end of the period that the rock which previously had dimensions of about ¼ inch by ¼ inch had now increased, due to a swelling effect, to about ½ inch by ½ inch, although the entire mass had become softened and the original angular contours had been rounded by the loosening and swelling. The original dark color of the rock had completely given way to light silvery gray or gray-blue masses of fibers, and when a portion of this was spread out and dried, the resulting layer was a brilliant white. This increase in brightness over fiber mechanically opened, felted, spread out and dried, is a further indication of the extremely fine subdivision of fibers obtained by my process.

The jelly-like mass of loose fibers formed from a piece of rock can be pulled out in a long string which, on drying, leaves an oriented length of fibers. When twisted, this makes a fine yarn which is very strong. When layers of this gelatinous material are dried, the product is extremely soft and smooth, has a soapy or unctuous feel in which hard bits of fiber are absent. It adheres to the fingers but not to itself. This adhesion is not a function of the organic material as it still persists on removal of the latter. Thus, the character of these dried films is evidence that a state of extremely and uniformly fine fiber subdivision has been reached.

The foregoing is an extreme example illustrating the highly effective character of my invention in that it shows the ability to open a very tightly bound, naturally occurring, macroscopic bundle of fiber in a static manner. It illustrates the ability to obtain high subdivision of the natural rock while substantially retaining naturally occurring fiber length.

It will, of course, be understood that the statically opened material requires some relatively mild mechanical action such as agitation before the opened fibers can be availed of for fabrication into ultimate products for a smooth distribution of the fibers in liquid is required. This may be readily effected by agitation of the opened material in the desired amount of diluting liquid by stirring with addition, if required, of sufficient detergent of the same or different character for sustaining the dispersion to fit it for the further use intended. In this example, since there is already present 15% Aerosol OT by weight based on the asbestos, no additional aerisol is necessary and the material may be agitated to produce a 6⅔% slurry which will remain in stable dispersion. If desired, this may be further diluted down with water, without the addition of any more wetting agent, to produce approximately a 1% slurry.

Example 2

A sample of rock, same as used in Example 1, was treated with fifteen times its weight of a solution of 7½% Triton 770 solids, and was completely softened in four hours at room temperature.

While I ordinarily relate the surface-active agent proportion to the asbestos, the concentration of the surface-active agent in water also has a pronounced effect within limits. For the

purpose of static opening of the asbestos in the case of Aerisol OT, below 1% there is progressive slowing of action, and above 5% the rate of opening also is reduced. My preferred range for high activity and rapid opening is between ¼% and 5%. Below ¼% the action slows down greatly and above 5% the solution becomes viscous and apparently loses effectiveness. In the case of Triton 770, the best range for opening is between 1% and 9% based on solids. Seven and one-half per cent is an extremely effective concentration, opening a ¼ inch piece of asbestos rock in a few hours at room temperature. In one example of static opening as above the liquid to asbestos ratio was 9 to 1.

Example 3

A mixture of so-called spinning fiber was fully immersed in a 1% solution of dioctyl sodium sulfosuccinate in water in the proportion of 100 grams of asbestos to 300 grams of solution, and showed a certain fluidity. In order to maintain this fluidity, it was found necessary, upon standing in the atmosphere for successive days, to add more solution until at the end of, say, a week, the required additions to maintain this fluidity had increased the liquid to 700 grams. This is an indication that more fibrous surface, requiring proportionately more wetting, had been developed over this period, and that in order to produce more fibrous surfaces, more subdivision of fibers had occurred.

I have calculated that in the above case over ten times as many fibers must have formed by subdivision of the original fibers to require this additional wetting. During this treatment of these mechanically opened fibers, small splinters of originally aggregated fiber masses were completely softened and brought down to a general state of fine subdivision.

Example 4

Mill fiber (the bagged asbestos fiber of commerce) is placed in a muller of the type used for making ceramic mixes and putties and is wet down with a 1% solution of Aerosol OT in the proportion of 6 parts by weight of liquid to 1 of fiber. The mixer is run for about one hour and additional 0.1% aerosol solution is added to allow easier emptying at, say 11% solids. The mix is by then thoroughly softened and plastic. It is further diluted with 0.1% aerosol OT solution to about ½% asbestos concentration and passed several times through a pump having a rubber impeller and becomes a smooth gray gelatinous fluid slurry ready for use in extrusion or paper making.

Example 5

Mill fiber of spinning grade, for example, Bell's Asbestos Mines 3R, is placed in a mixer with vertical cylindrical bars rotating therein and is wet down with 12 parts by weight of 1% Aerosol OT solution. It is then mixed slowly over a period sufficient to develop in the mix a maximum viscosity. As the opening occurs, the viscosity increases due to the liberation of new fibers and relatively large splints and spicules become softened and separate into component fibers. When the maximum viscosity has been attained and the mix is of satisfactory smoothness, a further quantity of diluting liquid which is a 0.1% solution of Aerosol OT in water is added to reduce the concentration to ½% fiber. At this concentration, the mass is smooth, gray, relatively free from unopened bundles and is still gelatinous. It may then be used for extrusion, casting or paper making.

Example 6

Instead of adding the Aerosol OT in different concentrations, the 100 parts of fiber are wet at once with 27 parts of Aerosol OT in 1% water solution (2700 parts liquid), and mixed in this concentration, until as before, it has become adequately smooth. Analysis of the liquid phase at this point shows that the concentration of Aerosol OT has dropped to .23% indicating that .77% has become adsorbed on the fiber. Water is then added slowly to dilute the entire mix to ½% fiber concentration. In this case, the proportions are selected so that at no time during the dilution will the effective aerosol concentration drop below 0.1% in the liquid phase.

Example 7

A slurry was prepared for the manufacture of thin asbestos tissue. To 10,000 lbs. of water at 160° F. was added 125 lbs. of sodium oleate as red oil soap. This was dissolved and 250 lbs. of #2 Crude asbestos (a long spinning grade), which had been previously pan crushed for 8 minutes, was mixed therewith in a mixer with vertical cylindrical bars rotating therein. This was continued for two hours in order to develop a smooth viscous dispersion, and produced a slurry with a 2½% asbestos concentration. It was then further diluted by gradual addition of warm water to a concentration of ⅜%, while continuing the mixing over a period of two hours. This developed slurry was smooth, contained no hard bits of fiber, and was gray and homogeneous in appearance. It had a freeness on a Williams freeness tester (3 grams asbestos in 1,000 c. c.) of about seventy thousand seconds. After screening to remove bits of rock, picrolite (a pseudo asbestos fiber occurring in chrysotile rock formations) and any clots of fiber remaining in dead spots in the mixer, the fiber was ready for running on a Fourdrinier tissue machine and was made into a smooth tissue with a thickness of .0005 inch and weight of 1 gram per sq. ft. (6.5 lbs. per ream). The tissue was, in spite of its thinness, tough, strong, of high density, and good dielectric strength, with fiber highly oriented in the machine direction.

Example 8

A slurry was prepared as in Example 7 for making asbestos tissue. To 1000 gallons of water was added 312 lbs. of 25% Aerosol OT in water and dissolved, then 212 lbs. of 3R spinning grade asbestos was added which on stirring for 4 hours produced a smooth slurry at 2½% asbestos concentration. This was further diluted by adding water slowly to the 2½% slurry in the volume proportion of 9 parts of 2½% slurry to 47 parts water. This produced a smooth ⅜% dispersion on mixing for 4 hours. It had a freeness of 70,000 seconds on a Williams freeness tester. The slurry thus made was run on a Fourdrinier tissue paper machine at 85 ft. per minute, and a tissue was made weighing 0.7 gram per sq. ft., about .00035 inch thick. This tissue was well formed, smooth, uniform in fiber distribution, tough, strong, stretchy, and highly oriented in the machine direction. It contained about 5% organic matter from the Aerosol OT which was divided into 4.5% adsorbed and 0.5% free residue resulting from the evaporation of the aerosol solution

accompanying the fiber in the wet web as it met the dryer. The wet web was about 22% solids and 78% water. This tissue is particularly adaptable for spinning into fine, high asbestos content yarn, because of its slight hygroscopic nature and stretchiness.

For the production of products which are thicker or of different shape than those which can be produced on paper-making equipment or by lamination of paper machine products, as for casting where flowable masses are required, it is desirable to use dispersions of higher concentration than shown in the above Examples 7 and 8. In these cases a slurry is first produced as previously so as to get maximum fiber separation without entrapment of air bubbles, which have a very damaging effect on these dispersions. A viscous asbestos dispersion, otherwise good, may become very stringy by beating air therein and this must, therefore, be avoided in handling viscous dispersions. I, therefore, remove excess liquid from my well-made, relatively-dilute dispersions by evaporation or slow filtration or centrifuging or other means, in all cases avoiding any manipulation which develops frothing. The smooth thickened flowable masses thus produced are now adaptable for casting into shapes which may then be dried in a suitable fashion.

I may, of course, also directly prepare and use asbestos dispersions of high concentrations while retaining a sufficient amount of free water containing a dispersing agent to assist in the dispersion and individualization of the fibers. The maximum is roughly of the order of 10% depending on the specific agent used, which may vary the consistency of the slurry considerably.

The gelatinous material formed in accordance with the foregoing examples can be strained through slotted plates, passed through small orifices, and in general acts more like a viscous jelly than a mass of ordinary asbestos fibers in water. Fine transparent films of considerable area can be produced therefrom which, when laid on a glass slide, dried and examined under a microscope, show that the film was composed of cobwebby asbestos fibers which were invisible to the naked eye in the wet film and are also invisible as individual fibers under the optical microscope.

As hereinbefore indicated, and as further illustrated by examples, fibers which have been previously opened by mechanical or disruptive methods have also been found to be potentially further subdivisible and can be greatly refined by treatment in accordance with my invention.

I have found the most satisfactory type of asbestos for dispersion by this process is Canadian chrysotile, which disperses most rapidly and most thoroughly of the various types of chrysotile available. South African, Cyprus and Russian chrysotile disperse at slower rates and much less completely but can be used. Italian amphibole disperses fully. Arizona chrysotile disperses almost as well as Canadian chrysotile. Temperature has a pronounced effect on dispersion of chrysotile asbestos by this means. At boiling temperature, the rate is increased about four times so that the effect produced at room temperature in four days is produced at 212° F. in one day or less, for static opening of rock. Heat generally accelerates the production of dispersion made with agitation also.

For each surface-active agent, there appears to be a minimum proportion which is adsorbed on the asbestos and therefore required for opening up the asbestos and this must be supplied by using an adequate volume of adequately concentrated agent. The proportions herein stated of concentrations and volumes relative to the asbestos are characteristic examples. However, in addition to the minimum amount required and adsorbed by the asbestos, a further concentration should remain in the liquid for the purpose of dispersing the previously opened fibers. In this case, I find that beyond the amount necessary to satisfy the fibers a certain concentration of the agent in the liquid phase is necessary to maintain a stable dispersion. Referring to Figure 3, this is .035% for Aerosol OT, and in Figure 4 .047% for sodium oleate.

The minimum proportion of Aerosol OT which will produce a stable dispersion with Canadian chrysotile asbestos, as shown by Fig. 5, is about 4% based on the fiber at high concentrations, such as an asbestos content of 10%. For practical purposes the amount of this agent would be increased to a minimum of 6%. All other effective surface-active agents show higher breakpoints than this. For example, as shown in Fig. 6, in the case of sodium oleate, the minimum proportion at the breakpoint is about 10% based on the fiber at like high asbestos concentration of 10%, and for practical purposes, this would be increased to at least 15% based on the fiber. It may be noted that the minimum proportion of wetting agent based on the asbestos is lowest at the higher concentration and increases gradually to the 1% asbestos range and very sharply below the 1% range. These data are collected in Figures 5 and 6 for Aerosol OT and sodium oleate respectively. Smaller proportions may be used where stability is not required or where the maximum dispersing effect is not required since there is a gradation from complete dispersion down to complete clotting as the concentration of surface-active agent in the water is reduced and which is readily observable by making up slurries with graduated reduction of agent concentration down to 0%.

As shown in the analyses of treated fiber previously given, a proportion of free wetting agent remains on the fiber (the methyl alcohol extract) after a filtration and drying process adsorbed on the fiber. This proportion varies with the concentration of the agent or agents in the aqueous liquid, the dryness of the filtered or otherwise separated fiber and the particular agent used. This residue may be of value for its modifying properties on the fiber. For example, if it is waxy, or oily, or somewhat hygroscopic, it may function as a lubricant for the fibers in operations such as spinning, twisting, weaving, drafting, etc. It can later be removed by extraction with a suitable solvent, heating to decompose it, or reaction with some other chemical agent to modify its properties. The proportion left on the fiber may vary from about 5% of the fiber weight in a paper tissue-making process (a filtering operation) to 30% or more where the entire liquid is dried along with the fiber. Of this residue, 10% to 30% may be free and from 90% to 70% is adsorbed. If the material is left as a lubricant during processing, its later removal by any of the means described will increase the strength of the product by removal of the lubricant between the fibers. Heat decomposition residues may also act as a bond, according to my observations.

For example, as the opening of the asbestos progresses, the proportion of aerosol remaining in the liquid phase drops and this must be taken

into account in obtaining full opening and full dispersion, either by having a large enough amount available from the beginning or additional aerosol being added, if necessary, or the concentrations increased in any suitable manner to maintain the necessary excess of agent.

It should be emphasized that these dispersions act in many respects like emulsions or dispersions of other substances, such as oils or resins, in that it is necessary on dilution to maintain an adequate concentration of protective colloidal material and to dilute slowly so as not to shock the dispersion. If plain water is added at too fast a rate, even though the overall proportion of contained protective is high enough above the safe limit, the dispersion may flocculate. Also, if water is added to reduce the concentration of protective material below the safe limit, flocculation will occur. Furthermore, if acids, salts of weak bases, polyvalent cations or detergent cations are added to this dispersion which is protected by the protective, clotting will result.

It is usually desirable to separate associated impurities such as serpentine fragments, magnetite inclusions, etc., which do not open, and portions of original rock containing altered asbestos which is not amenable to fiberization, and other impurities, and this may be accomplished by taking advantage of the jellylike water-holding characteristics of the opened mass by straining it through fine slotted screen plates such as are used in paper and pulp screening. The mass will even pass through wire screen, say, 10 to 20 mesh, but tends to tangle badly on such screen because two ends of the same fiber may pass through two different openings in the screen and thereby hang on the screen. Dilution in the manner described is desirable before straining to reduce tangling of the fibers. I find concentrations of ¼% to 1% asbestos a good range.

The proportion of liquid is usually more than ten times the weight of the asbestos to be opened and dispersed, since relative freedom on the part of the asbestos from a confined condition aids in the opening and dispersion, and this is attained by keeping the mass quite wet. It has been found possible in spite of the gelatinous condition of the fibers to filter through fine cloths or screens a considerable portion of the liquid phase which apparently is not strongly attached to the fibers and may be considered as free liquid. However, beyond the state at which free liquid can be expressed, the fiber and the liquid together can pass through screens and orifices and the liquid content in this range should be considered as "bound" water, not necessarily chemically bound water. This "bound" water is, however, evaporable.

When fibrous masses are opened without agitation, I find that the mass so obtained is relatively non-homogeneous, that is, fiber clumps, even though fine, tend to remain together and the mass of fibers shows considerable variation in concentration for different spots. This, of course, is highly undesirable when the dispersed mass is to be poured or extruded or cast, and a smoothing or homogenizing operation is necessary. Some of the agents mentioned in my list do not effectively open fibers in the static condition, as, for example, sodium oleate, and in these cases agitation or mixing is required from the start of the process. Also, as a matter of efficiency, this mixing is ordinarily desirable whenever mill fibers are used with any of the agents.

It is to be understood that where the intent is to preserve the great proportional length of these extremely fine fibers, mixing and smoothing operations should be adapted to avoid tangling, roping and tearing of the fibers and should, therefore, be as gentle as possible, commensurate with obtaining the desired result. For example, mixing in low concentrations or in slurries of low viscosity, and slow speeds, tends to maintain fiber length whereas the opposite conditions, while attaining the smoothness more rapidly, result in fiber length reduction. Paper beaters, Jordan engines, and the like attrition machines are highly undesirable where fiber length maintenance is important.

Passage through slots, orifices, pipe constructions or Venturi devices, against baffle devices, or slow speed mixers without sharp moving edges or rotation centers in the slurry, slow speed pumps with large clearances and flexible impellers are all suitable to provide homogenizing with minimum fiber length reduction.

The desirable action is one which gently releases the single fibers, previously chemically separated, from proximity to their original neighbors and frees them for independent existence.

As indicated, the products of my process have novel and varied utility and my invention allows the production of asbestos materials of a character and quality hitherto unattainable and unknown in conventional fiberizing methods. The gelatinous dispersion may be employed in various degrees of concentration or dilution, from the viscous or gelatinous material as produced in concentrates to the great state of dilution common in paper-making processes.

The gelatinous dispersion in various concentration may be sprayed, extruded, cast, molded, or felted into films, sheets or shapes of desired character, shape, width, or thickness, and dried by evaporation or filtration.

A mass of gelatinous fibers, formed in accordance with my invention, may be cast or extruded in directionalized formation and the fibers may be separated and moved along their longitudinal axes by reason of their smooth individualized unit nature.

For example, a ½ to 2% dispersion of spinning fibers may be extruded through a $\frac{1}{32}$ inside diameter hypodermic syringe needle without clogging provided the dispersion has been freed from lumps.

It may be employed alone or as a fibrous reinforcement in admixture with binders or fillers for the production of bonded structures, such as insulation material, friction materials, packings, and gaskets to provide toughness and strength.

The dried fibrous product may be disintegrated for use of the fibers or it may be repulped by wetting it with a dilute solution of an agent of the class employed for originally opening and dispersing the fibers, to again produce the gelatinous fibrous dispersion.

Paper made of fibrous asbestos in opened and dispersed form and where the dispersion and individualization of the fibers is maintained in dilution in accordance with my process, as previously described, can be sheeted out in desired thicknesses down to very thin tissuelike webs by paper-making apparatus or other means, without the aid of a binder, to produce smooth webs of high strength and relatively free of tiny splinters of asbestos characteristic of paper made from asbestos fibers prepared by prior opening processes. Also, very long fibers, such as spinning and crude length fibers, which in the ordinary

clotted form cannot be run on a paper machine into smooth paper because of the clotted condition, are easily handled in my dispersed individualized fiber condition and can be formed and sheeted into very smooth and very thin webs, such as .0015 inch thick, and if desired, as low as .0002 inch thick. The paper so made is extremely dense to the passage of gases or liquids therethrough due to the felting of very fine individualized fibers, and has therefore a very finely porous structure, advantageous for electrical insulation and fine filter uses. Its dielectric strength is several times as great as a comparable thickness of ordinary asbestos sheet, and its gas permeability many times less. The paper is characterized by extreme softness, limpness and "hand." With long fibers, the fibers are highly oriented in the machine direction and a narrow strip can be torn without material change in width for long distances while tissue cannot be torn uniformly across its length. This orientation is of great value in spinning operations. It has high draping properties, clings to the fingers, but not to itself, has a silvery sheen in reflected light and thin areas appear cloudy gray to transmitted light. It is very tough and can be stretched and distorted considerably before it tears. The latter properties of course depend to some extent on the length of the fibers used.

The fibrous web may be wound on itself in the standard method for making thick laminated wet machine-type boards or may be otherwise laminated, and these again exhibit the toughness, strength and distortability of the thin webs.

My novel sheet material, after sizing, may be employed for use as writing and printing paper for permanence or for ordinary asbestos paper uses.

Sheets or tapes may be cut or formed to ribbon width, by either extruding, casting or the paper-making process, for twisting into coarse or fine yarn capable of considerable drafting or stretching, and by employing the indicated very thin webs, fine yarns can be formed and fine fabrics woven therefrom.

The fibers may be colored in the dispersion or later to provide papers and woven fabrics of more permanent attractive appearance.

The thin webs may be used in the form of tapes for winding wire for electrical insulation, as wrappings for heat insulation or for the production of wound packing and gasket rings. The thin web material may also be employed for such purposes as wrapping or packaging for protecting delicate polished surfaces or structures from mechanical abrasion or from the atmosphere and for filtration or filter structures. The dried material in sheet form can be used for diaphragms for electrolytic cells. It may also be employed as a carrying film for plastics or rubbers, or as a base for various coatings or coated fabrics, for the production of leather-like sheet products in combination with a rubber binder, and for a multitude of other uses, either alone or in various combinations.

Paper made from my opened fiber dispersion where the diluent is water and which, as a result of dilution, does not contain enough residual or added dispersing or deflocculating agent of the classes mentioned to keep it dispersed, is similar to ordinary asbestos paper in strength. However, because of the very fine fibers comprising its structure, it is likewise extremely smooth and soft and free from the characteristic small splinters and spicules found in ordinary asbestos paper. Also, due to the fine fiber content, it has very high wet strength and the wet webs are well adapted for handling on paper machines in lower thicknesses than can be ordinarily handled. Thus, thinner paper can be made by standard asbestos paper-making equipment than with ordinary fiber. In this case, the fibers are clotted by the action of water containing insufficient dispering agent, and the dried paper made therefrom shows the general properties of paper made from ordinary asbestos fiber in water suspension which is also in the clotted condition when filtered on the paper machine. Otherwise, however, this paper material is characterized by the highly opened, finely-divided fibers and is of novel character and adapted for many novel uses.

I claim as my invention:

1. As a new composition of matter, a colloidal dispersion of chrysotile asbestos fibers in an aqueous vehicle, containing an organic crysotile asbestos colloidizing agent.

2. The dried residue of the colloidal dispersion of claim 1.

3. The dried residue of the colloidal dispersion of claim 1 in felted sheet form.

4. A new composiiton of matter composed essentially of chrysotile asbestos fibers in colloidal dispersion in an aqueous vehicle, containing an organic chrysotile asbestos colloidizing agent.

5. A new composition of matter characterized essentially by colloidal properties, composed essentially of chrysotile asbestos fibers in colloidal dispersion in an aqueous vehicle, containing an organic chrysotile asbestos colloidizing agent.

6. As a new composition of matter, a colloidal dispersion of chrysotile asbestos fibers in an aqueous vehicle containing organic chrysotile asbestos colloidizing agent, the fiber surfaces being saturated with said agent, the vehicle containing a free amount of said agent maintaining said saturated fibers in colloidal dispersion.

7. As a new composition of matter, a colloidal dispersion of chrysotile asbestos fibers in an aqueous vehicle containing chrysotile asbestos colloidizing agent, the fiber surfaces being saturated with said agent, the vehicle containing a free amount of said agent maintaining said saturated fibers in colloidal dispersion, said dispersing agent being a water soluble fatty acid soap.

8. The composition of claim 7 wherein the colloidizing agent is sodium oleate.

9. As a new composiiton of matter, a colloidal dispersion of chrysotile asbestos fibers in an aqueous vehicle containing chrysotile asbestos colloidizing agent, the fiber surfaces being saturated with said agent, the vehicle containing a free amount of said agent maintaining said saturated fibers in colloidal dispersion, said dispersing agent being a sulfonated ester.

10. The composition of claim 9 wherein the colloidizing agent is dioctyl sodium sulfosuccinate.

11. An alkaline, gelatinous, flowable, stable, colloidal dispersion of chrysotile asbestos fibers in an aqueous liquid containing organic chrysotile asbestos colloidizing agent, the fiber surfaces being saturated with an adsorbed film of said dispersing agent, the liquid vehicle containing a free amount of said agent maintaining said saturated fibers in colloidal dispersion, said fibers being relatively free to move with respect to each other as compared to the untreated asbestos from which they are derived, the majority of the fibers having a diameter between 200 and 500 Angstrom units.

12. The method of treating chrysotile fibrous asbestos agglomerates which comprises, mixing them with an aqueous solution of organic chrysotile asbestos colloidizing agent effective to (*a*) subdivide them into fibrils of colloidal size and to saturate the fibril surfaces with and to deposit thereon an adsorbed layer of said agent (*b*) and to colloidally disperse said saturated fibrils.

13. The method of opening chrysotile asbestos agglomerates and colloidally dispersing the resultant fibers, which comprises combining said agglomerates with an aqueous liquid containing organic chrysotile asbestos colloidizing agent adsorbable on the asbestos and capable of forming an alkaline dispersion therewith, the amount of agent being in excess of that adsorbable on the asbestos and providing an added colloidal dispersion forming and maintaining increment, and mixing said components to bring them into intimate contact until the fiber surfaces become saturated with an adsorbed film of said agent whereby the agglomerates are subdivided into colloidally fine fibers and a colloidal dispersion of said fine fibers is formed.

14. The method of claim 12 further characterized by filtering the dispersion and drying the fibrous asbestos residue.

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