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METHOD OF WET FELTING AND LEACHING MINERAL FIBERS AND RESULTANT FELTED PRODUCT

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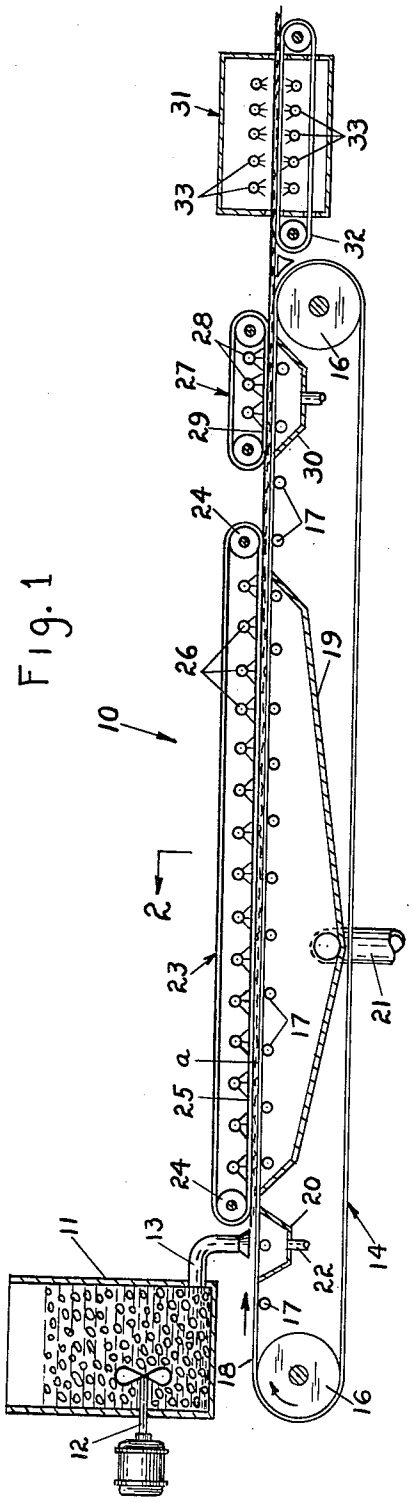


Fig. 1

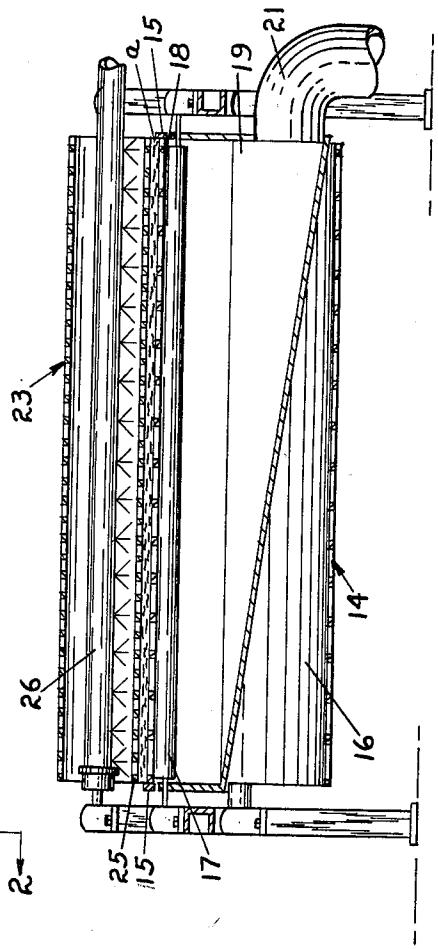


Fig. 2

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METHOD OF WET FELTING AND LEACHING MINERAL FIBERS AND RESULTANT FELTED PRODUCT

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The present invention relates generally to temperature-resistant mats, and more particularly to an improved temperature-resistant mat of fibers and to a method of forming such mats.

It is known that glass fibers, when leached free of their alkali and alkaline glass forming metallic oxides, comprise substantially pure silica and have softening points in the neighborhood of 3000° F. These fibers, when formed into mats, have proven to be particularly useful for high temperature insulation; for example, around the tail-pipes of jet engines.

In the past, two different methods for forming mats of leached glass fibers have been used. One of these is disclosed in Parker et al. Patent No. 2,491,761, wherein a resin-bonded mat of glass fibers is leached with suitable mineral acids and then fired to burn off the resin binder. However, the use of this method had several disadvantages including the burning off of the edges of the mat when the same is subsequently fired. This difficulty was overcome, as stated in Parker et al. Patent No. 2,500,092, by enclosing the resin-bonded mats in an acid-resistant envelope during the leaching process, which envelope must be subsequently removed.

The disadvantages of the methods of these prior patents are more fully pointed out in Parker Patent No. 2,635,390 which discloses a method of forming fiber batts involving first forming a suspension of glass fibers, leaching the fibers while in suspension, and then felting the fibers into a mat. However, this method also has certain drawbacks among which are the fact that a number of process steps are necessary and a certain time period must elapse while the fibers are in the leaching tank and thus a continuous mat cannot be advantageously produced.

Thus in accordance with the above-mentioned prior art, it was necessary in order to form satisfactory high temperature mats to subject a resin-bonded mat to a number of handling operations if it was desired to leach fibers in mat form, or first leach the fibers, wash them, pulp the fibers, and then felt them into a mat.

In accordance wtih the present invention, however, it has been found practical to first form the fibers into a mat or felt, and then leach the fibers in felted form, with the resultant mat having greatly improved strength as compared to mats formed in accordance with the prior art.

Therefore, an important object of the present invention is to provide an improved high temperature fibrous mat and a method of producing such a mat.

Another object of the invention is to provide a novel method of forming an improved high temperature fibrous mat in which glass fibers are first wet-felted into a mat, and a leaching solution is then flowed through the felted mat to remove the non-siliceous glass forming oxides.

A further object of the invention is to provide a high temperature mat of leached glass fibers having improved strength as a result of first wet-felting glass fibers into a mat, and then leaching the non-siliceous glass forming oxides from the matted fibers.

Other objects and advantages of the invention will become more apparent during the course of the following description when taken in connection with the accompanying drawings.

In the drawings, wherein like numerals are employed to designate like parts throughout the same:

FIG. 1 is a schematic view of a leaching and mat forming line adapted to perform the method of the invention; and

FIG. 2 is a transverse sectional view of a portion of the line taken along the line 2—2 of FIG. 1.

Briefly stated, the improved mat of leached fibers provided by the present invention is produced by first forming a suspension of glass fibers, felting the suspended fibers into a mat, and then flowing or percolating a leaching solution through the felted mat of fibers to remove the non-siliceous glass forming oxides, after which the leached mat is dried.

With reference now to the drawings, there is shown in FIG. 1 a mat-forming apparatus 10 for performing the method of the invention in a continuous manner. The apparatus 10 includes a pulping or mixing tank 11 provided with suitable beater means 12 and having an outlet in the form of a downwardly turned pipe or spout 13 communicating with the interior of the tank. Positioned beneath the spout is a continuous, movable foraminous conveyor belt 14 having side bars 15 thereon and supported by means of a pair of support rolls 16 one of which may be driven by suitable motive means to move the belt 14 away from the tank 11.

Suitable idler rolls 17 are spaced between the support rolls 16 to maintain the upper flight 18 of the belt 14 in a substantially horizontal plane. Also positioned between the support rolls 16 is a pair of troughs 19 and 20 having their widths substantially equal to that of the belt 14 and provided with outlets 21 and 22 respectively. As will be later described, these troughs are provided to receive the various liquids used in the method of the invention.

Positioned in spaced relationship above the belt 14 is a second foraminous belt 23 of somewhat shorter length, also provided with a pair of support rolls 24 one of which is preferably driven by suitable means to move the belt 23 at a speed equal to that of the belt 14. Suitable means (not shown) may be provided for regulating the distance between the pair of opposed foraminous belts 14 and 23 which preferably are composed of an acid-resistant material such as "Saran" (registered trademark) having very closely spaced openings.

Arranged above the lower flight 25 of the upper foraminous belt 23 are a plurality of transverse spray pipes 26 closely spaced to one another and covering the distance between the rolls 24. These pipes 26 are provided with a plurality of orifices through which, as will be later described, a leaching solution may be sprayed downwardly onto and through a wet-felted glass fiber mat as it passes therebeneath.

Positioned beyond the belts 14 and 23 is a fiber washing station which also includes a foraminous belt 27 supported and driven similarly to the belt 23 and positioned above the belt 14. Suitable transverse spray pipes 28 are positioned above the lower flight 29 of the belt 27 and are adapted to direct a washing fluid, such as water, downwardly and through the mat as it passes therebeneath, which water is drained by means of a trough 30. As will be later described, when an acid leaching solution is used, it is desirable to wash the mat free of the salts which form when the non-siliceous glass forming oxides are leached from the matted fibers.

To dry the mat after leaving the washing station, an oven 31 having a belt conveyor 32 located therein is positioned immediately beyond the washing station to receive the mat of fibers after being washed. The oven may be equipped with suitable heating means such as burners 33

which will afford a controlled temperature range for the oven of between about 100° C. to 1200° C.

Various types of glass compositions may be used in forming the improved product of the present invention and by the method thereof herein disclosed. For example, a composition known commercially as "E" glass and having approximately the following composition may be used:

	Percent
Silica (SiO_2)	54
Aluminum oxide (Al_2O_3)	16
Titanium oxide (TiO_2)	0.05
Iron oxide (Fe_2O_3)	0.2
Calcium oxide (CaO)	17
Magnesium oxide (MgO)	5
Sodium oxide	0.5
Boric anhydride (B_2O_3)	7.25

In carrying out the method of the invention with fibers of such a composition or with any glass fibers which are acid leachable, the fibers are dropped in bulk form into the tank 11 which is filled with water. While in the tank, the bulk fibers are agitated and separated from one another by the beater means 12 until the fibers are segregated from one another and are in suspension. The suspended fibers are then flowed out of the tank through the spout 13 and filtered or wet-felted into continuous mat form upon the upper flight 18 of the lower foraminous belt 14 which, of course, is driven in a forward direction away from the tank 11. During the felting operation, the water in which the fibers are suspended flows through the belt 14 and is carried away by means of the trough 20.

Prior to actually forming the felted mat, the distance between the upper and lower foraminous belts 14 and 23 is adjusted so that the upper belt 23 will exert a slight amount of pressure upon the wet-felted mat *a*. While retained between the belts and passing beneath the spray heads 26, the wet-felted mat is subjected to a continuous spray of a dilute acid leaching solution such as an aqueous solution of hydrochloric or sulfuric acid. Suitable concentrations of acid are about 5 to 20% by weight. If desired, the acid solution may be heated to a temperature of about 180° F. with the resultant leaching of the glass fibers being accomplished more rapidly as a result of the use of a heated acid solution.

After being sprayed upon the mat, the acid leaching solution flows or percolates downwardly through the mat and in contact with the individual fibers thereof and is collected by the trough 19. If desired, the leaching solution may be recirculated through the spray pipes. In such a case, a certain amount of the solution is wasted and acid is added to maintain the desired solution strength. While the leaching solution is flowing through the glass fiber mat, the non-siliceous glass forming oxides are leached from the fibers until the fibers consist of substantially pure silica and chemically combined water.

The speed of travel of the wet-felted mat beneath the spray pipes will, of course, be dependent upon the temperature of the leaching solution, the length of the leaching zone, i.e., the distance between the endmost spray pipes, and the volume of flow of the leaching solution. These variables may be adjusted depending upon the desired production requirements. Preferable results are obtained when the mat is submerged in leaching solution which is accomplished by spraying a sufficient volume of solution to cause a depth over the mat of about ⅛ inch.

After being leached, the wet-felted mat passes through the washing zone wherein water from the pipes 28 is sprayed over the leached fibers to remove the sulfates or chlorides, depending upon the leaching acid, which collect on the fibers. After being washed, the wet-felted mat is then continued into and through the oven 31 wherein it is dried at a temperature of approximately 100° C. if it is desired that the fibers still contain their water of hydration, or at a temperature of between about 1000° C. and 1200° C. for several minutes if it is desired to form a heat-shrunk mat wherein the fibers contain no water of hydration.

Although the invention, as previously described, may be used with fibers formed of "E" glass, the preferred embodiment of the invention utilizes glass fibers formed of an alkali silicate glass. Such a glass may have a composition of between about 72% to 80% silica and about 28% to 20% alkali metal oxide, preferably sodium oxide. For example, a glass composition composed of about 78.2% silica and 21.8% sodium oxide, as described in my co-pending application Serial No. 562,722, filed February 1, 1956, to which reference is herein made, has proved particularly well suited in the preferred embodiment of the invention. It has been found that fibers of this type leach very rapidly with the afore-mentioned acid solutions and, in fact, may be leached free of alkali oxide through the use of water alone. Thus, when submerging fibers of alkali silicate glass in the tank 11 wherein the fibers are dispersed in water and formed in suspension, a leaching action starts immediately so that by the time the fibers are dispersed and flow through the spout 13 to be wet-felted into a mat they are partially leached and thereby subsequently facilitating a more rapid and efficient leaching of the felted mat. In leaching such a mat of alkali silicate fibers, water may be sprayed through the pipes 26 instead of the afore-mentioned acid leaching solution although water will not leach the fibers free of alkali nearly as rapidly as the acid solutions.

Although, as previously mentioned, leaching by water is somewhat slower than leaching with an acid solution, the cost of the process is considerably cheaper and in most localities ordinary tap water may be used. Another advantage of water leaching is that the process equipment such as the belts 14 and 23 need not be of acid resistant material.

In lieu of a water or acid leaching solution, when sodium silicate fibers are being leached, a 5% solution of calcium chloride, such as that disclosed in my co-pending application Serial No. 393,967, filed November 23, 1953, now U.S. Patent No. 2,823,117, may be used if it is desired to form a mat of leached calcium silicate fibers. In such a case, the calcium chloride solution is sprayed onto the felted mat *a* by means of the pipes 26 and thus flowed through the mat. The leaching reaction results in the extraction of the sodium from the fibers and the replacement therewith by calcium, as is more fully described in my aforementioned co-pending application Serial No. 393,967. The fibers of resultant felted mat have a composition of about 93% silica and 7% calcium oxide when the mat is heated to a temperature of about 100° F. to 1200° F. to remove the chemically combined water.

It is not fully understood how first wet-felting the fibers into mat form, and then leaching the same, while being retained in mat form, should produce a stronger mat than was produced by the prior art methods of forming mats of leached fibers but the fact remains that it does. However, it is known that wet-felting fibers produces a mat of very closely compacted fibers which are interlaced with one another. In advancing the mat beneath the spray heads which distribute the leaching solution, the passage or flow of leaching solution through the mat undoubtedly produces a more dense felt which materially increases the strength of the resultant mat.

Another factor influencing the strength of the mat is the tendency of the leached fibers to shrink and thus interlock when drying. By first forming the fibers into a felted mat and then leaching the mat, all shrinkage of the fibers occurs when they are in mat form. On the other hand, when fibers are first leached and then put in storage prior to being felted into mat form, the effect of the initial fiber shrinkage is lost as far as the resultant mat is concerned.

It has also been found that the fiber diameter plays an

important part in both the leaching operation and the strength of the resultant mat. Fibers having diameters which average less than about 1 micron and preferably between about 0.04 to about 1 micron have proven to be superior over fibers having diameters of greater than about 1 or 1½ microns. Thus, fibers of a diameter of less than about 1 or 1½ microns felt into an extremely dense mat and because of their small diameter a great many more of these fibers become interlocked with one another than is the case with fibers of larger diameter. Also, the smaller fibers leach much more readily and speedily than is the case with larger diameter fibers.

The improved mat may also partially result from the fact that very fine fibers of sub-micron diameter are used. Preferably, the preponderance of the fibers, i.e., over 50%, should be less than ⅜ inch and range between about ⅛ to ⅜ inch. A felted mat of sub-micron diameter fibers, the preponderance of which are less than about ⅜ inch, will result in a stronger more compact mat which will retain its shape due to the self-adherence of the fibers. By flowing a leaching solution through such a mat, the compactness thereof is further increased.

Although the apparatus shown employs upper belts **23** and **27** in the leaching and washing zones respectively, these belts may be eliminated if only a very fine spray of leaching and washing solutions is used. In the preferred manner of carrying out the method of the invention, the mat is literally flooded with leaching solution and, when an acid solution is used, the mat is also flooded with wash water. In such a case, the upper belts **23** and **27** apply sufficient restraint to the upper surface of the mat to prevent any disruption of the fiber formation due to the impingement and flow of leaching liquid therethrough.

It is to be understood that the form of the invention herewith shown and described is to be taken as a preferred embodiment of the same, and various changes may be resorted to without departing from the spirit of the invention or the scope of the subjoined claims.

I claim:

1. A method of forming a temperature-resistant mat, comprising mixing glass fibers having an average diameter of less than about 1 micron in a liquid to form a suspension of said fibers, wet-felting said suspended fibers into a compacted mat on a foraminous surface, and while so felting passing a leaching solution through the felted mat to remove leachable glass forming oxides from the fibers, and thereafter drying the mat.

2. A method of forming a temperature-resistant mat as defined in claim 1, wherein a preponderance of the fibers have lengths less than about ⅜ inch.

3. A method of forming a temperature-resistant fiber mat, comprising a continuously wet felting glass fibers of short lengths and of an average diameter less than about one micron onto a moving foraminous surface to form a compacted mat, and while the mat is supported on said moving foraminous surface passing a leaching solution through said mat to extract leachable oxides from the fibers and to further compact said mat.

4. A method of forming a temperature-resistant mat, comprising wet felting glass fibers of short lengths and of an average diameter less than about one micron into a compacted mat, sandwiching the mat between a pair of moving foraminous surfaces, and then flowing a leaching solution in one direction through said moving foramnous surfaces and mat to extract leachable oxides from the fibers.

5. A method of forming a temperature-resistant mat, comprising felting glass fibers of an average diameter less than about one micron from a liquid medium into compacted mat form on a moving liquid permeable support surface, engaging the upper surface of the felted mat with a second moving liquid permeable surface, and while the mat is positioned between said moving surfaces flowing a leaching solution through said surfaces and mat to extract leachable oxides from the fibers.

6. A wet-felted mat of temperature-resistant fibers resulting from the passing of a leaching solution through a wet felted mat of glass fibers consisting essentially of about 22% to 27% alkali metal oxide and from about 78% to 73% silica to remove the leachable glass forming oxides and in which the fibers of the mat have an average diameter of less than about 1 micron and a preponderance of said fibers have lengths less than about ⅜ inch.

7. A method of continuously forming a temperature-resistant mat comprising felting glass fibers having an average diameter of less than about one micron from a liquid medium into a mat on a moving liquid-permeable support surface, compacting said mat while in motion and, while said mat is being compacted, flowing a leaching solution through said surface and mat to remove the leachable oxides from the fibers.

8. A method of forming a temperature-resistant fibrous mat, comprising mixing short fibers consisting essentially of alkali metal oxide and silica in water, wet felting said fibers into a compacted mat onto a moving liquid-permeable support surface and, while said mat is being compacted on said surfaces passing a leaching solution therethrough to further compact said mat and to remove the alkali metal oxide from the fibers.

9. A method of forming a temperature-resistant mat as defined in claim 8, wherein the fiber composition prior to leaching is about 22% to 27% alkali metal oxide and from about 78% to 73% silica.

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CERTIFICATION OF CORRECTION

Patent No. 3,012,930 December 12, 1961

Dominick Labino

It is hereby certified that error appears in the above numbered patent requiring correction and that the said Letters Patent should read as corrected below.

Column 4, line 52, for "100° F." read -- 1000° F. --; column 5, line 53, strike out "a"; column 6, line 38, for "surfaces" read -- surface --.

Signed and sealed this 24th day of April 1962.

(SEAL)
Attest:

ESTON G. JOHNSON
Attesting Officer

DAVID L. LADD
Commissioner of Patents