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PROCESSES FOR SEPARATION OF ASBESTOS FIBER FROM ASSOCIATED SOLIDS

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My invention relates more particularly to recovery of asbestos fiber that has been used in the composition of diaphragms serving to separate the anode and cathode compartments of electrolytic cells, such as those employed in the decomposition of alkali metal chlorides for production of caustic alkali and chlorine. Such cells commonly employ graphite anodes and foraminous steel cathodes. The ends of the anodes are commonly sealed in with bitumen or its equivalent. The electrolyte is an aqueous solution of the alkali chloride, commonly sodium chloride. This is fed into the anode compartment of the cell and percolates through the diaphragm, becoming electrolyzed in its passage to a solution containing caustic soda and undecomposed sodium chloride. This electrolyte always contains more or less foreign matter. The diaphragm acts as a filter to remove such matter. The graphite slowly disintegrates, and some of it finds its way in the form of fine particles into the electrolyte and thence to the pores of the diaphragm. The electrolyte always contains traces of calcium salts, such as the sulphate and silicate. Some of these are derived from the mine from which the sodium chloride was obtained and some are picked up from the concrete used to insulate the anode from the cathode, or as a cover for the cell. The calcium becomes transformed in the cell to its hydroxide, which is likewise deposited in the pores of the diaphragm. The surface of the bitumen becomes more or less chlorinated and some of it likewise finds its way into the diaphragm. The result is that the porosity of the diaphragm gradually diminishes. Eventually a time is reached when it is no longer fit to serve its purpose. The diaphragm is then removed and replaced by a fresh diaphragm. The diaphragm may have been formed of a sheet of asbestos paper applied by hand, or it may have been formed from the loose fiber in situ by the process of U. S. Patents, Nos. 1,862,244; 1,862,245 and 1,865,152. In any case, the material removed is a sodden mass, slimy with caustic soda, salt and finely granular deposit and black from graphite. Approximately two thirds of this mass represents the original asbestos fiber and one third material that has been gathered during operation of the cell.

Asbestos fiber has a specific gravity of 2.1 to 2.8. In order to maintain it in suspension for the purpose of depositing it in the form of a diaphragm in the cell, in accordance with the above mentioned patents, it is customary to use, as the suspension medium, the effluent from the cell itself, having a specific gravity of about 1.2. The fiber would not be expected to remain in suspension in this medium, but in practice it is found that the caustic soda in the suspension medium acts to gelatinize and swell the fiber, thus reducing the specific gravity of the fiber to such an extent that it will remain in suspension for a substantially longer time than is needed for the operation of forming the diaphragm.

I have now discovered that in its gelatinized condition minute air bubbles attach themselves very readily to the asbestos fiber, and with aeration these may easily become sufficient actually to float the fiber to the surface of the suspension liquid, even when this is pure water. Thus, if a quantity of the mass be agitated with 20 to 100 times its weight of water in such a way as to detach the fibers from each other, disperse them throughout the suspension and entrain air, when the agitation is discontinued the asbestos will rise to the top and the other solids, which are not gelatinized, will settle to the bottom. The asbestos may then be readily separated from the other solids and recovered in form suitable for re-use.

One way of performing the separation may be illustrated by simply shaking up the suspension in a liter graduate, for example. The air thus entrained will be found sufficient to float the asbestos fiber, leaving the other solids to settle to the bottom. Upon standing, the asbestos will give up its flotation charge of air, or if the graduate be subjected to a vacuum, or the water boiled, the asbestos will lose its air immediately. In either case the asbestos will then settle and rest upon the other solids, proving that the segregation is due to flotation, and surface tension phenomena.

Another way in which the bubbles may be caused to attach themselves to the fiber is to suspend the fiber in cold water and then heat the suspension. The heating drives out sufficient of the air dissolved in the water to cause flotation of the fiber.

It should be noted that the asbestos is already gelatinized as it comes from the cell, but it may be desirable to give it a preliminary wash and gelatinize it again by soaking in caustic soda solution before performing the flotation operation, and this is especially true if the mass has been allowed to dry. Instead of subjecting the mass to a preliminary soaking in caustic soda solution, the entire operation may be conducted in a solution of caustic soda, which may also contain

sodium chloride, such as a solution containing 200 to 300 grams per liter of NaCl and 90 grams per liter of NaOH. However, I find that the segregation is most rapid and positive when the suspension medium is water at 20° to 60° C., and preferably 50° C., and the concentration of solids .02 to .05 per cent by weight, and preferably .04 per cent.

The flotation of the asbestos may be assisted by first passing the mass through a beater of the paper pulp type. The fibers thus detached from each other may then be placed in the suspension medium, which should be initially cold. By heating the medium slightly, enough air will then be expelled to float the fiber.

Another method of breaking up the mass and liberating the foreign matter is to bubble air through the suspension. This not only detaches the fibers from each other, but at the same time aerates the suspension and leaves the material in a condition to segregate naturally as soon as the agitation is discontinued.

When the fibers have been detached from each other and supplied with flotation air by either of the above described methods, the actual separation may be performed by imparting a gentle rotary movement to the suspension in a circular vessel. When the vessel has been filled in this way to its rim, the fiber will float over the rim, leaving the other solids behind. The rotary movement may be obtained by causing the fresh liquid to flow in tangentially with respect to the suspension.

Instead of adding fresh suspension liquid to float the fiber over the rim, fresh suspension of fiber in the liquid may be added continuously. The fiber is then continuously floated over the rim and the other solids are continuously removed from the bottom of the vessel. This may be facilitated by making the vessel of conical form, with the apex at the bottom; or in other words, performing the operation in a cone separator. The fiber then floats over the rim and the solids are withdrawn at the apex.

In practice, with a cone separator having a diameter of about four feet and an equal depth, and a rotary movement of about one rotation in 2 to 3 minutes, the separation is found to be very effective.

If preferred, the suspension may be led into the cone separator cold, and the heating to expel the flotation air from the liquid may be performed in the separator itself, as by direct injection of steam or in a steam jacket.

The recovered asbestos, in the form of a slurry, may be caught on a 100 to 200 mesh screen, washed, dewatered, and stored damp; or it may be dehydrated, degelatinized by heat and stored dry.

The caustic soda employed in the process, if it be necessary to use any beyond that with which the mass of diaphragm material is naturally wet, is of course recycled and the net consumption is very small.

In place of caustic soda, any other caustic alkali, such as caustic potash or lithium may be used to gelatinize and swell the fiber. Alkali metal halides, as well as many other alkali metal salts, such as the citrates and borates, likewise have a gelatinizing effect on the fiber, although it is generally inferior to that of caustic soda. The alkaline earth hydroxides are likewise inferior to the caustic alkali for this purpose. This would seem to indicate that there is some particular virtue for my purpose in the alkali metals as such, regardless of the combination in which they may occur.

When the asbestos to be recovered is derived from caustic soda chlorine cells, which are by far the largest users of asbestos for diaphragm purposes, the cheapest and most available caustic alkali is naturally the caustic soda with which the material is already wet.

The asbestos recovered from cell diaphragm by my process is so nearly in its original condition that when it is redeposited in the form of a diaphragm covering the foraminous metal cathode the rate of percolation of the electrolyte through the diaphragm is found to be substantially the same as in the case of diaphragm formed from fiber previously unused for the purpose. Also, the rate at which the permeability of diaphragms formed from reused fiber diminishes, due to deposits of foreign matter, and consequently the useful life of diaphragms thus constituted, is found to be normal. It is found that 80 per cent of the asbestos applied to the cells may be recovered and reused by my process, thus reducing the net consumption of asbestos fiber to about one fifth of what it would otherwise be.

I claim as my invention:

1. The method of recovering and conditioning for re-use asbestos fiber that has been used in the composition of a diaphragm overlying the foraminous cathode of an electrolytic cell producing chlorine and caustic alkali, where it has been in contact with caustic alkali and brine and become swollen by the gelatinizing action thereof and the diaphragm has accumulated a deposit, including carbonaceous and calcareous matter filtered or precipitated out of the electrolyte, which comprises agitating the mass with 20 to 100 times its weight of water to detach the fibers from each other and from the particles of foreign matter and cause minute air bubbles to attach themselves selectively to the individual swollen fibers; diluting the mass still further until it is in presence of not less than 2,000 times its weight of water; allowing the diluted mass to segregate; and recovering the fibers from the upper part of the resulting suspension.

2. The method of recovering and conditioning for re-use asbestos fiber that has been used in the composition of a diaphragm overlying the foraminous cathode of an electrolytic cell producing chlorine and caustic alkali, where it has been in contact with caustic alkali and brine and become swollen by the gelatinizing action thereof and the diaphragm has accumulated a deposit, including carbonaceous and calcareous matter filtered or precipitated out of the electrolyte, which comprises agitating the mass, while its fibers are still swollen by said gelatinizing action, with 20 to 100 times its weight of water to detach the fibers from each other and from the particles of foreign matter and cause minute air bubbles to attach themselves selectively to the individual swollen fibers; diluting the mass still further until it is in presence of not less than 2,000 times its weight of water; allowing the diluted mass to segregate; and recovering the fibers from the upper part of the resulting suspension.

3. The method of recovering and conditioning for re-use asbestos fiber that has been used in the composition of a diaphragm overlying the foraminous cathode of an electrolytic cell producing chlorine and caustic alkali, where it has been in contact with caustic alkali and brine and become swollen by the gelatinizing action thereof and the diaphragm has accumulated a deposit, including

carbonaceous and calcareous matter filtered or precipitated out of the electrolyte, which comprises washing the mass; agitating the mass with 20 to 100 times its weight of water rendered alkaline by the addition of caustic alkali thereto, to detach the fibers from each other and from the particles of foreign matter and cause minute air bubbles to attach themselves selectively to the individual swollen fibers; diluting the mass still further until it is in presence of not less than 2,000 times its weight of water; allowing the diluted mass to segregate; and recovering the fibers from the upper part of the resulting suspension.

4. The method of recovering and conditioning for re-use asbestos fiber that has been used in the composition of a diaphragm overlying the foraminous cathode of an electrolytic cell producing chlorine and caustic alkali, where it has been in contact with caustic alkali and brine and become swollen by the gelatinizing action thereof and the diaphragm has accumulated a deposit, including carbonaceous and calcareous matter filtered or precipitated out of the electrolyte, which comprises agitating the mass, in contact with air, with 20 to 100 times its weight of water to detach the fibers from each other and from the particles of foreign matter and cause minute air bubbles to attach themselves selectively to the individual swollen fibers; diluting the mass still further until it is in presence of not elss than 2,000 times its weight of water; allowing the diluted mass to segregate; and recovering the fibers from the upper part of the resulting suspension.

5. The method of recovering and conditioning for re-use asbestos fiber that has been used in the composition of a diaphragm overlying the foraminous cathode of an electrolytic cell producing chlorine and caustic alkali, where it has been in contact with caustic alkali and brine and become swollen by the gelatinizing action thereof and the diaphragm has accumulated a deposit, including carbonaceous and calcareous matter filtered or precipitated out of the electrolyte, which comprises agitating the mass with 20 to 100 times its weight of water to detach the fibers from each other and from the particles of foreign matter; bubbling air through the mass to cause minute air bubbles to attach themselves selectively to the individual swollen fibers; diluting the mass still further until it is in presence of not less than 2,000 times its weight of water; allowing the diluted mass to segregate; and recovering the fibers from the upper part of the resulting suspension.

6. The method of recovering and conditioning for re-use asbestos fiber that has been used in the composition of a diaphragm overlying the foraminous cathode of an electrolytic cell producing chlorine and caustic alkali, where it has been in contact with caustic alkali and brine and become swollen by the gelatinizing action thereof and the diaphragm has accumulated a deposit, including carbonaceous and calcareous matter filtered or precipitated out of the electrolyte, which comprises agitating the mass with 20 to 100 times its weight of cold water to detach the fibers from each other and from the particles of foreign matter; warming the water to drive air out of solution in it and cause minute air bubbles to attach themselves selectively to the individual swollen fibers; diluting the mass still further until it is in presence of not less than 2,000 times its weight of water; allowing the diluted mass to segregate; and recovering the fibers from the upper part of the resulting suspension.

7. The method of recovering and conditioning for re-use asbestos fiber that has been used in the composition of a diaphragm overlying the foraminous cathode of an electrolytic cell producing chlorine and caustic alkali, where it has been in contact with caustic alkali and brine and become swollen by the gelatinizing action thereof and the diaphragm has accumulated a deposit, including carbonaceous and calcareous matter filtered or precipitated out of the electrolyte, which comprises agitating the mass with 20 to 100 times its weight of water to detach the fibers from each other and from the particles of foreign matter and cause minute air bubbles to attach themselves selectively to the individual swollen fibers; diluting the mass still further until it is in presence of 2,000 to 5,000 times its weight of water; allowing the diluted mass to segregate; and recovering the fiber from the resulting suspension.

8. The method of recovering and conditioning for re-use asbestos fiber that has been used in the composition of a diaphragm overlying the foraminous cathode of an electrolytic cell producing chlorine and caustic alkali, where it has been in contact with caustic alkali and brine and become swollen by the gelatinizing action thereof and the diaphragm has accumulated a deposit, including carbonaceous and calcareous matter filtered or precipitated out of the electrolyte, which comprises agitating the mass with 20 to 100 times its weight of water to detach the fibers from each other and from the particles of foreign matter and cause minute air bubbles to attach themselves selectively to the individual swollen fibers; diluting the mass still further until it is in presence of not less than 2,000 times its weight of water; allowing the diluted mass to segregate at 20° to 60° C., and recovering the fibers from the upper part of the resulting suspension.

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