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3,079,331 PROCESS OF RECOVERING COAL FINES Elmer W. Gieseke, Fountain City, Tenn., assignor to American Cyanamid Company, New York, N.Y., a corporation of Maine No Drawing. Filed Aug. 27, 1959, Ser. No. 836,338 2 Claims. (Cl. 210—53)

This invention relates to a process of recovering naturally flotatable coal fines. More specifically, it relates $_{10}$ to a process of recovering finely divided coal which is naturally flotatable, which process comprises the successive steps of adding to a slurry of such coal fines a reagent such as soluble lignin sulfonates, dextrin, sulfonated nigrosines or mixtures thereof, then decanting the slurry 15 from such coal as settles, adding to the decanted slurry a flocculating agent consisting of a mixture of a water soluble polyacrylic polymer and pregelatinized starch and decanting the liquid from the coal fines.

some very finely divided coal. Such finely divided coal passes on through the wet sieving process as a slurry. Since there is a considerable quantity of such coal, efforts are made to recover as much as possible. This recovery is complicated by the fact that powdered coal 25 to 100% polymer). is often naturally flotatable and furthermore is often contaminated with oils during the mining operation, which increases the tendency to float. Consequently, this finely divided coal does not settle readily in the tanks to which such slurries are run. Often some of the coal forms in 30 either at the desliming stage as described above or in the a froth on top of the tank and will not settle. In the past the froth of unsettled finely divided coal has been run into a second tank in which starch and lime are added in an effort to induce settling. However, because of the tendency of the slurry to froth, while some was 35 final settling of all waste solids. Even here normally the recovered by this method, the recovery has not been satisfactory and some coal fines always remain slurried in the effluent liquor. Such losses not only are in economic waste but cause considerable pollutions of streams.

A further disadvantage of the prior operations was in 40 fact that the froth, which was formed from the finely divided coal not only cannot be settled but similarly cannot be filtered when formed in a flotation process. Thus this frothing interfered with both settling by flocculation and with the recovery of such fines by a flotation process. 45

I have found first that the frothing of finely divided coal can be reduced and even eliminated, by the addition to the aqueous slurry of soluble lignin sulfonates, dextrin, sulfonated nigrosines and mixtures thereof, and that, with the froth thus eliminated, a certain amount of 50 further settling occurs from such fines and the remaining slurry can be either filtered or flocculated for filtration. I have further found that the flocculation of such a defoamed slurry can be effectively and economically carried out by adding thereto a mixture of a water soluble 55 polyacrylic polymer and pregelatinized starch.

The coal fines which are recoverable by the process of my invention are those which naturally float in water and which do not settle readily. In the processing of coal the minus 1/4" screen sized coal is normally deslimed 60 in large bowl desilters, drag tanks or similar devices. Much of the coal floats as a froth product over these desliming devices and normally goes to the thickeners where flocculation agents are usually added to settle the solids. A great deal of material still normally remains 65 floating and is pumped to waste. In the process of my invention the defoaming reagent is placed in the desilming device and the flocculation agent is usually added to the solution in the thickener tanks, although I can perform both operations in the same tank if desired.

The defoaming reagents which may be used in the first step includes water soluble lignin sulfonates such as the

sodium, potassium or calcium salts, dextrin, sulfonated nigrosines and water soluble salts thereof and various mixtures thereof. In order to destroy the froth it is necessary to add at least 0.0005 to 0.001 part of these reagents per 100 parts of the water in the slurry.

The flocculating agents which can be used in conjunction with pregelatinized starch to obtain effective flocculation of the slurry of coal fines include polyacrylic polymers of greater than one hundred thousand molecular weight. The polyacrylic polymer can be polyacrylamides, hydrolyzed polyacrylonitriles, copolymers of acrylamide and acrylic acid and the like. These polymers having acrylic acid are used in the form of the sodium salt. Preferably molecular weights should be greater than 300,000 and the best results are obtained with even higher molecular weights such as 2,000,000 or even higher. In addition one can use copolymers of vinylacetate and maleic anhydride in the form of the sodium salt, when molecular weights of the above ranges In the processing of coal, there is inevitably formed 20 are achieved. These polymers are used in conjunction with pregelatinized starch. At least 0.25 to 10 parts per million parts of water in the slurry of the combined polymer and starch must be used and the polymer to starch ratio must be 1:20 to 1:0 (i.e. the mixture is 5

The coal fines thus flocculated are recovered by filtration and dried.

The process of my invention is usable in the recovery of coal fines from the tailings from the processing of coal, waste effluent from such coal processing plants after more conventional treatment has been used. Normally the waste products from such coal processing plant are passed into what is known as a drag tank in order to permit the coal fines pass on through into the effluent from the plant. The use of the foam regulating reagent plus the flocculation mixture in such drag tanks effectively prevents the passage of coal fines into the river or other depository of waste liquor.

One advantage of my invention is the economic one that a large amount of coal which otherwise passes out unrecovered and is lost can now be recovered. Another advantage of my invention is that the foaming which, in processing plants handling large quantities of such slurries, can build up very badly in the thickening tanks has been eliminated. At times the foam builds up in the thickening tanks to the point where it is necessary to shut the plant down, a process which can be quite uneconomic. This is eliminated by the use of the foam regulating compounds as described above.

My invention can be illustrated by the following examples.

Example 1

At one large coal processing plant the minus one quarter inch screen sized coal is normally deslimed in eight forty four feet diameter bowl desilters. Much of the coal overflows as a froth product and as fines over these desilters and normally goes to two 120 foot diameter thickener tanks where an effort is usually made to settle the solids. The solids recovered in these thickeners is fine coal and normally amounted to about 120 tons per

To the desilter tank discharge is added 5 lbs. per hour per each 1,000 gallons per minute of slurry of a mixture containing 70% crude calcium lignin sulfonate, 29.4% corn dextrin and 0.6% sulfonated nigrosine. The addition is made to the overflow as it flows out of the desilter into one of the thickening tanks. 60 tons per hour of fine coal is recovered in this thickener as underflow and the overflow was run into a second thickener where the

flocculating agents, namely a mixture of 0.25 to 0.5 part of polyacrylamide of approximately 2,000,000 molecular weight and 2.5 to 5.0 parts of pregelatinized starch per million parts of slurry is added. The overflow from the second thickener is sufficiently clear that it can be reused in the plant. The total recovery of coal amounts to about 60 tons per hour greater than obtained in that plant when not using the process of my invention.

Example 2

The procedure of Example 1 is followed using as the defoaming reagent the various following reagents:

(1) 100% crude calcium lignin sulfonate.

(2) A mixture of 100 parts of dextrin and 200 parts of nigrosine.

(3) A mixture of 50% crude calcium lignin sulfonate with 50% sulfonated nigrosine.

The results in each case are similar to that obtained in Example 1.

Example 3

The procedure of Example 1 is followed using in place of the polyacrylamide the following deflocculating agents:

(1) Sodium salt of hydrolyzed polyacrylonitrile of molecular weight 500,000.

(2) The sodium salt of the copolymer of acrylamide and acrylic acid of molecular weight about 1,000,000.

(3) The sodium salt of polyacrylic acid of molecular

weight of approximately 1,000,000.

(4) The sodium salt of the copolymer of vinyl acetate and maleic anhydride of molecular weight about 750,000.

(5) Polyacrylamide of molecular weight 5,000,000. In each case the results are similar to those obtained in Example 1.

Example 4

A coal processing plant which runs its effluent into a drag tank to settle out all solids found a considerable quantity of fine coal dust passing out into the nearby stream. To the drag tank overflowing at the rate of 1000 gallons per minute is added 10 lbs. per hour of a 40 slurry of the foam regulating composition described in Example 1 followed by 1 to 2 lbs. per hour of a mixture consisting of 1 part of polyacrylamide of molecular weight about 2,000,000 and 5 parts of pregelatinized starch. As

Example 5

The procedure of Example 4 is followed except that

4 the flocculation reagent is 1 to 2 pounds per hour, of polyacrylamide of molecular weight about 2,000,000, no starch being used. The overflow comes clear.

I claim:

1. A process of recovering floating coal fines from aqueous slurries topped with coal fine-containing-froths which comprises the successive steps of (1) adding to said slurries about 2.5 to 10 parts per million of a defoaming reagent selected from the group consisting of 10 (a) soluble lignin sulfonate salts and (b) dextrin and sulfonated nigrosines (c) and mixtures thereof, and (2)treating the resulting defoamed, coal fine-containingslurry with a reagent essentially comprising (a) 5 to 100% of a water soluble polymer of molecular weight greater than 100,000, selected from the group consisting of polyacrylamides, hydrolyzed polyacrylonitriles, copolymers of acrylamide and acrylic acid, and the salts of copolymers of vinyl acetate and maleic anhydride, and (b) 0-95% of pregelatinized starch.

2. A process of recovering floating coal fines from aqueous slurries topped with coal fine-containing-froths which comprises the successive steps of (1) adding to said slurries about 2.5 to 10 parts per 1,000,000 of a defoaming reagent essentially comprising a mixture of 70% crude calcium lignin sulfonate, 29.4% corn dextrin and 0.6% sulfonated nigrosine; and (2) treating the resulting defoamed coal fine-containing-slurry with a flocculating agent essentially comprising one part of polyacrylamide of about 2,000,000 molecular weight and 10 parts of

pregelatinized starch.

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