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PROCESS FOR SEPARATING COMMINUTED MATERIALS

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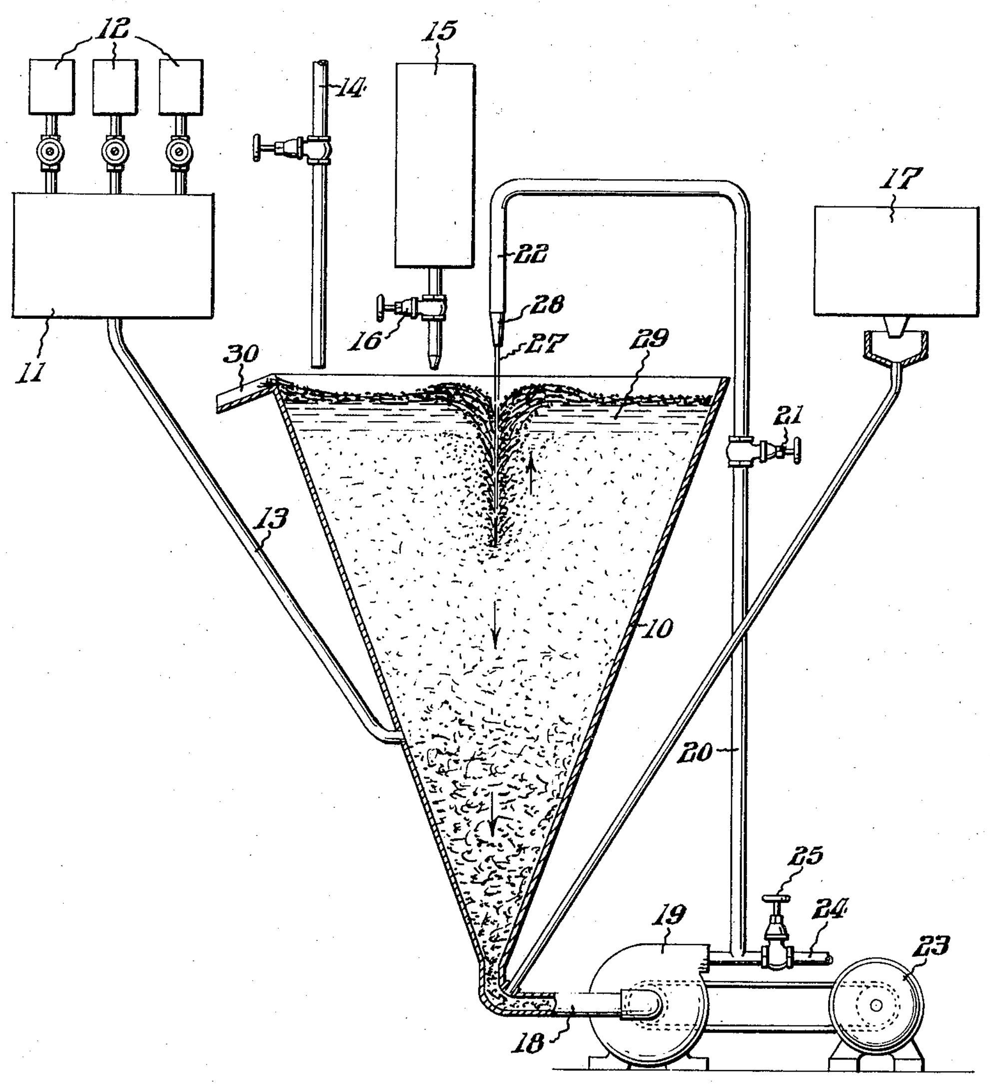

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PROCESS FOR SEPARATING COMMINUTED MATERIALS

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This invention relates to a new and improved process for separating comminuted materials, and more particularly relates to improvements in the froth flotation process for separating desired concentrates in pulps containing comminuted carbonaceous matter, non-metallic minerals, or metallic ores.

The froth flotation process was introduced about the beginning of the present century. The process originally consisted of mixing pulverized ore or the like with water in the presence of a small amount of inorganic acid, such as sulfuric acid, to form a pulp. A small amount of oil was added to the pulp and then air bubbled through it to the surface by means of a submerged impeller. Because the oil was present in such small quantity, it would adhere to the concentrate particles rather than act as a levitating agent. The oil, however, had no affinity for the impurities in the pulp and would not adhere to them. As the air bubbles passed upwardly through the pulp, they would attach themselves to the oil coatings on the concentrate particles with the result that such particles were carried to the pulp surface with the air bubbles. The concentrate, upon reaching the surface of the pulp, was collected for further treatment.

This old process, while reasonably effective for coarse materials, has several disadvantages, one of which is that it can not successfully treat particles of minus 200 mesh. Further, even with coarser particles it was found that a considerable quantity of impurities were carried to the surface of the pulp intermixed with the concentrate. This is because the separation of the concentrates takes place in the body of the pulp. As the air bubbles and their attached oil coated particles ascend to the surface of the pulp, they pass through a medium heavily charged with comminuted impurities, and although the impurities are rejected by the air bubbles, some of them nevertheless become entrapped in the rising mass and are rafted with the concentrate to the surface, and thence removed with the concentrate. As a result, it has been necessary in using the froth flotation process to adopt a flow sheet consisting of plural cells to progressively reduce the amount of impurities in the end product.

Another disadvantage of the froth flotation process has been the high cost of dewatering the concentrate. Because of this high water content, it has been necessary to dry the concentrate by means of revolving drum filters, centrifugal filters, heat drying and other artificial methods.

Numerous attempts have been made to improve upon the froth flotation process. For example, it has long been known that if the particles of the comminuted feed were reduced to minus 200 mesh, the concentrate would be partially freed from the impurities physically combined therewith. However, endeavors to treat such finely divided particles did not prove to be successful since the impurities, due to their minute size, continued to be rafted to the surface of the pulp with the concentrate in substantial quantity.

Another method by which it was hoped to improve the froth flotation process was to subject the pulp to violent agitation by projecting it under pressure through nozzles into comparatively small receptacles from which the agitated pulp would overflow into a larger receptacle where, it was expected, a complete separation would take place with the concentrate rising to the surface of the pulp in the large receptacle. An illustration of this method may be found in Appelqvist et al. Patent No. 1,367,223. However, this method did not succeed in solving the problem since separation still took place in the body of the pulp, and the ascending air bubbles continued to raft impurities to the surface with the concentrate.

Another method, particularly adapted to separating metallic ores, was devised in which amalgams were created in the pulp by the use of very large amounts of oil and pulverized coal. These amalgams had an affinity for the metallic ore and would tend to reject the impurities combined therewith. Examples of this process are disclosed in Trent Patents No. 1,421,862 and No. 1,420,164. While this process produced a somewhat better separation than had theretofore been possible, it was not successful in producing effective separation of particles ranging from 3/16" mesh to 0. Further, the high cost of the agents used in this process to form the amalgam rendered it of no commercial value.

It was later thought that the use of baffles in the flotation cells would serve to reduce the amount of impurities carried off with the concentrate. See, for example, Lasseter Patent No. 2,226,170. In the commercial application of this process, however, it was found that a complete separation still was not achieved since it took place in the body of the pulp.

Yet another method devised to overcome the disadvantages of the froth flotation process was the use of a conical tank having a cylindrical baffle at its center, extending above and below the surface of the pulp, and means for withdrawing pulp from the cell and projecting it, under pressure, through the air against the surface of the pulp within the baffle. See, for example, Phelps Patent No. 2,416,066. It was believed that by projecting the pulp into the baffle under sufficient pressure to carry it and the air bubbles thus formed below the baffle, the bubbles and the concentrate attached thereto would escape around the bottom of the baffle and rise to the surface of the pulp on the outside, whence the concentrate was removed. However, this process did not provide a complete separation for the reason that the separation still took place in the body of the pulp, and impurities were still rafted to the surface with the concentrate.

All of these modifications produced a better separation than had been possible in the original froth flotation process, but none of them succeeded in effecting a complete separation and none of them were effective with particles ranging from minus 200 mesh to 0.

The invention which is about to be described herein is based on the discovery that the impurities which are invariably present in the concentrate after separation in the initial froth flotation cell can be excluded in that cell by changing the locus of separation from down in the body of the pulp to the surface thereof, and by utilizing the principle of capillary attraction and repulsion at the locus of separation. In this invention, the pulp is introduced into a cell and a thin film of oil spread and maintained over the pulp surface. Thereupon, pulp is circulated from the cell to a point above the oil film whence it is forcibly projected through the air and through the oil film into the body of the pulp remaining in the cell under sufficient pressure to cause it to resurge back through and above the oil film and be dispersed from the point of resurgence laterally in the direction of the cell wall. As the pulp is thus dispersed, it impinges against the inner edge of the oil film surrounding the resurging pulp, whence the desired concentrate particles are attracted to the oil film, and remain at the surface of the pulp in the cell, while the impure particles are rejected by the oil and pass into the body of the pulp. This phenomenon is explained by the law of capillary attraction and repulsion, the concentrate being naturally attracted to the oil film while the impurities are repelled by it. The concentrate thus collected at the surface of the pulp is removed by suitable means for further treatment. It is essential in the operation of this invention that the pulp be projected into the pulp remaining in the cell under sufficient pressure to ensure its resurgence above the oil film.

The chief object of this invention is to provide a process of separating desired concentrates in comminuted materials whereby it is possible to effect a complete and exact separation in the initial pulp cell regardless of the particle sizes.

It is a further object of this invention to provide a process for separating desired concentrates in comminuted materials whereby it is possible to effect a complete and exact separation of particles ranging in size from 3⁄16" mesh to 0.

It is a further object of this invention to provide a process for separating desired concentrates in comminuted materials whereby it is possible to effect a complete and exact separation of particles ranging in size from 3⁄16" mesh to 0 regardless of the relative percentages of the particle sizes in the feed.

It is a further object of this invention to provide a method of preventing the entrainment of excess water in the concentrate and at the same time rendering the concentrate self-dewatering to thereby eliminate the necessity for using artificial drying means.

It is a further object of this invention to provide a process of separating concentrates in comminuted carbonaceous materials whereby all impurities are separated from the concentrate to produce pure coal.

It is a further object of this invention to provide a process of separating concentrates in comminuted carbonaceous materials whereby it is possible to produce pure carbonaceous material which can readily be converted into elemental carbon.

It is a further object of this invention to provide a method of separating desired concentrates from comminuted metallic ores whereby the concentrate is completely free of gangue.

It is a further object of this invention to provide a process of separating coal concentrates which are substantially free of fly ash.

Other objects and advantages of this invention will be readily apparent from the following description, reference being had to the accompanying drawing illustrating a preferred embodiment thereof.

In the drawing, which illustrates schematically means for carrying out a preferred embodiment of this invention, the numeral **10** represents a froth flotation cell adapted to receive the pulp which is to be treated. The comminuted raw material which is to be separated is introduced into a feed conditioner **11** where it is commingled with desired agents from sources **12**. Any of the well known agents may be used, such as kerosene, oil, sulfuric acid, and the like, including water, and combined in such quantities and manner as have long been known in the art. After the raw material and the agents have been thoroughly commingled to form a pulp feed, the feed is introduced into the cell **10** by means of conduit **13**. In the cell **10**, the feed is mixed with water supplied from a water source **14** to form a pulp. The ratio of water to feed would depend on the nature of the feed, and would be in accordance with practices of long standing in the art. After mixing the feed and water to form the pulp, a thin film of oil **29** is spread over the surface

of the pulp from an oil supply **15**. Preferably, a hydrocarbon oil is used for this purpose. It is necessary that the oil film **29** remain intact at all times. Accordingly, a valve **16** is provided in the oil supply **15** to permit intermittent additions of oil as required to maintain the oil film **29**.

A small pH control **17** may be provided to introduce agents into the pulp to control its acidity or alkalinity, the nature of such agents, of course, being dependent on the type of material being treated. Any agents well known in the art may be used for this purpose.

In order to circulate the pulp in accordance with this invention, suction conduit **18**, pump **19**, discharge conduit **20**, valve **21** and header **22** are provided. The pump may be driven by any suitable means, such as a motor **23**. Conduit means **24**, having a valve **25**, is provided to permit removal of the impurities and the remaining pulp from cell **10** after the concentrate has been removed.

The operation of this process is as follows. Valve **25** is closed, pulp introduced into cell **10** and oil film **29** spread over its surface. Thereupon, with valve **21** opened, pump **19** is started. Pulp is withdrawn by the pump **19** from the bottom of the cell **10** through conduit **18** and discharged, through conduit **20**, to header **22** whence it is projected, under pressure, through the air and oil film **29**, into the body of the pulp in the cell **10**. The pulp stream **27** is projected into the body of the pulp under pressure sufficient to cause it to resurge back through and above the oil film **29**. In order to ensure that the pulp is projected under sufficient pressure, a nozzle **28** may be provided at the discharge end of header **22**.

Upon its resurgence, the pulp, now thoroughly mixed with air, is dispersed laterally against the inner edge of the oil film which surrounds the resurging pulp. As the resurging pulp is thus dispersed, the desired concentrate particles will be held in the oil, in accordance with the law of capillary attraction, and will be retained at the surface of the pulp by the air bubbles intermixed with the resurging pulp. The impure particles will be repelled by the oil film at its inner edge, in accordance with the law of capillary repulsion, and will pass under the oil film into the body of the pulp. As the concentrate collects at the surface of the pulp, it is removed from the cell through a conduit **30** by any suitable means (not shown) well known in the art, such as a skimmer or compressed air.

The oil film **29** not only attracts and holds the concentrate particles, but it also treats the uncoated particles, if any, with a monoionic layer of oil, thereby ensuring that all of the concentrate particles will be (1) retained at the pulp surface and (2) rendered self-dewatering.

The rate at which the pulp is circulated, and the number of times which it is circulated, is dependent upon the type of material being separated and the agents used and, of course, must be left to the judgment of the operator. As a general rule, however, the entire contents of the pulp may be circulated within a period of between one and two minutes, and the pulp should be re-circulated at least 15 times to effect a complete separation.

After the pulp has been fully treated and the concentrate removed from the cell, valve **21** is closed and valve **25** opened and the residue pulp discharged from the cell **10** by means of pump **19** through conduit **24**. Thereupon, the cell **10** may be recharged and the operation repeated.

Because of the fact that the locus of separation takes place above the surface of the pulp, it is possible by this process to effect a complete separation of particles of 3⁄16" mesh to 0, thereby overcoming one of the main disadvantages heretofore encountered in the froth flotation process. Further, because the separation takes place above the surface of the pulp, a minimum of water is entrained in the concentrate, and this fact, combined with the fact that all the particles of the concentrate will bear a coating of oil, renders the concentrate self-dewatering. By means of this improved process, substantial reduction in the cost of separating comminuted materials is realized since the process may now be carried out in a single cell without the use of artificial dewatering apparatus.

The following tests, run on coal samples, are illustrative of procedures embodying this invention:

Test No. 1

In this test a sample of anthracite silt, furnished by the Hudson Coal Company of Scranton, Pennsylvania, was used. The sample contained particle sizes ranging from 3/16" mesh to 0, and contained 33.50% ash.

The sample was first mixed with 40% water, to which had been added kerosene, at the ratio of 2 pounds per ton of raw feed, and sulfuric acid, at the ratio of 1 pound per ton of raw feed, to form a heavy sludge. The sludge was then introduced into a flotation cell and mixed with water, at the ratio of four parts water to one part sludge, to form a pulp.

The pulp was then subjected to continuous re-circulation in cycles of one minute, for a period of fifteen minutes, in accordance with this invention. It was found by laboratory analysis that the separated concentrate represented 99.5% of the desired values in the feed and that it contained 7.66% ash. Natural drying of the concentrate for a period of thirty-six hours reduced its moisture content from 50% to 7.10%.

Test No. 2

In this test a sample of fly-ash issuing from the combustion of pulverized anthracite coal in the plant of the Pennsylvania Water & Power Co. at Holtwood, Pennsylvania, was used. The sample in this instance contained 50% carbonaceous material and 50% non-combustible matter, and more than 86% of it was composed of particle sizes below 44 microns.

The sample was mixed with kerosene and cresylic acid, each at a ratio of 2 pounds per ton of raw feed, and with sulfuric acid, at a ratio of 1 pound per ton of raw feed, to form a sludge. The sludge was then introduced into a flotation cell and sufficient water added to form a pulp of five parts water to one part sludge.

The pulp was re-circulated in cycles of one minute, continuously for twenty-two minutes, in accordance with this invention. Laboratory analysis of the concentrate disclosed that it comprised slightly more than 99% of the desired values in the original feed, and that its ash content was 15%. The moisture content of the concentrate when removed from the cell was 34.60% and its ignition point was 930° F. After thirty-six hours of natural drainage, the concentrate was found to contain only 11.10% moisture and its ignition point had been reduced to 820° F. After sixty hours of natural drainage, the moisture content was only 4.60%.

Test No. 3

In this test two samples of underflow from thickeners at the Pittsburgh Consolidated Coal Co. cleaning plant at Imperial, Pennsylvania were used. Both samples consisted of bituminous coal having particle sizes ranging from 14 mesh to 0. One of the samples was taken from the 35–60 foot diameter thickeners at the plant and contained 33.6% particles of minus 200 mesh to 0. The other sample was taken from the 85 foot thickener at the plant and contained 73% particles of minus 200 mesh to 0.

These two samples were treated in the exact manner as the sample in Test No. 2, except that the re-circulation continued for twenty-three minutes. It was found upon laboratory analysis that the concentrates from both samples weighed more than 99% of the desired values in the feeds.

Test No. 4

The sample used in this test was furnished by the Lehigh Navigation Coal Co. and consisted of No. 5 buckwheat coal, containing 12.58% ash, reduced to particle sizes ranging from 2.78 microns to 0. This sample was mixed with 40% water and with sulfuric acid, at the ratio of one ounce per ton of raw feed, and cresylic acid, at the ratio of 100 cc. per ton of raw feed. The sludge thus formed was introduced into the flotation cell with sufficient water to form a pulp having eight parts water to one part sludge.

The entire pulp was re-circulated in cycles of one minute, for a period of twenty-four minutes, in accordance with this invention. Laboratory analysis disclosed that the concentrate removed from the cell constituted more than 99% of the desired values in the original feed, and had an ash analysis of 4.14%. The moisture content of the concentrate was reduced from 55% to 6.90% after fifty hours of natural drainage.

Test No. 5

In this test a sample of the reclaimed carbonaceous matter from Test No. 2, containing 15% ash, reduced in size to particles ranging from 1.75 microns to 0, was used. This sample was processed in the exact manner provided in Test No. 4 above. Upon laboratory analysis, it was found that the concentrate constituted more than 99% of the desired values and contained only 4.01% ash. It was further found that the combustible in the ash residue remaining in the pulp was less than 1%. After sixty hours of natural drainage, the moisture content of this sample was reduced from 50% to less than 8%.

Prior to running the above tests, an oil film was formed over the surface of the pulp being processed, and maintained intact throughout the conduct of each test.

The foregoing description of this invention, and the five tests described above illustrating it, are based on a batch type operation. It is possible, however, to carry out this invention by means of a continuous operation. For example, there could be provided an elongated tank into which water mixed with the desired agents would be introduced. Thereupon, a film of oil would be spread and maintained over the surface of the water by means of an appropriate oil supply. The comminuted material to be treated would be continuously introduced into, and carried longitudinally through, the bottom of the tank by means of a submerged screw conveyer. Water in the tank, mixed with the agents, and with comminuted material in suspension, would be circulated, as a pulp, by means of a pump, from the tank to plural headers above the oil film whence it would be projected, under pressure, through the air and through the oil film into the body of the water remaining in the tank. The design of the apparatus must be such that the projected water would strike the comminuted material being carried by the conveyer so as to roil it and intermix it with the water. In addition, the projected water must be discharged under sufficient pressure to cause the turbid mixture to surge above the oil film and be dispersed laterally against the inner edge thereof to effect separation. In this operation, the concentrate collected at the surface of the water in the tank would be removed by suitable means, while the waste material would be carried out of the tank by the screw conveyer.

By using the continuous operation described above, it would be possible to treat comminuted feeds without the necessity of pre-screening them to remove particles larger than 3/16" mesh. The larger particles, of course, would not float, but would be carried off with the refuse.

It is to be understood that the terms and expressions employed herein are used as terms of description, and not of limitation, and that there is no intention, in employing such terms and expressions, of excluding any equivalents of the features, steps and procedures shown and described, for it is recognized that various modifications are possible without departing from the scope of this invention as set forth in the claims.

Having thus described my invention, I claim:

1. The process of separating desired concentrates in comminuted materials comprising commingling comminuted material with selected agents to form a feed, mixing the feed thus formed with water in a cell to form a pulp, providing and maintaining an oil film over the surface of the pulp, conducting pulp from the cell to a point above the oil film and projecting it through the surrounding atmosphere into the pulp remaining in the cell under pressure sufficient to cause it to resurge above the oil film to effect a separation of the concentrate and then removing the concentrate from the cell.

2. The process of claim 1 wherein the material to be treated is first pulverized into particles ranging from 3/16" mesh to 0 in size.

3. The process of separating desired concentrates from pulp comprising introducing pulp into a cell, providing and maintaining an oil film over the surface of the pulp, withdrawing pulp from the cell and projecting it into the pulp remaining in the cell under pressure sufficient to cause the projected pulp to resurge above the oil film to effect a separation of the concentrate.

4. The process of claim 3 wherein the pulp withdrawn from the cell is mixed with air during its projection into the pulp in the cell.

5. The process of claim 4 wherein the pulp withdrawn from the cell is projected into the pulp in the cell from above the oil film.

6. The process of claim 5 wherein the concentrate is removed from the cell after separation.

7. The process of claim 6 wherein a hydrocarbon oil is used.

8. A froth flotation process for separating concentrates from pulp comprising maintaining an oil film over the surface of the pulp, projecting pulp through the oil film into the pulp below under pressure sufficient to cause the projected pulp to resurge above the oil film and removing the concentrate separated from the resurged pulp.

9. The process of claim 8 wherein the projected pulp is mixed with air prior to passing through the oil film.

10. The process of separating desired concentrates in comminuted materials comprising continuously passing comminuted material through a fluid body, providing and maintaining an oil film over the surface of the fluid body, withdrawing quantities of the fluid from the fluid body and projecting it from above the oil film into the fluid body under pressure sufficient to agitate the comminuted materials therein and to cause the turbid mixture thus formed to surge above the oil film to effect a separation of the concentrate.

11. The process of claim 10 wherein the concentrate is continuously removed from the fluid body after separation.

12. The process of claim 11 wherein the projected fluid is mixed with air during its projection into the fluid body.

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