METHOD OF PURIFYING HEAVY FERROSILICON SUSPENSIONS EMPLOYED FOR SINK-FLOAT SEPARATION PROCESSES

Rudolf Lutz, Koln-Ehrenfeld, and Gunther Salzmann, Koln-Dentz, Germany, assignors to Kloekner-Humboldt-Dentz Aktiengesellschaft, Koln-Dentz, Germany, a corporation of Germany

No Drawing. Filed Feb. 3, 1966, Ser. No. 6,345
Clain priority, application Germany July 1, 1939
11 Claims. (Cl. 289—166)

Our invention relates to a method of purifying heavy metal media employed in sink-float separation processes, which media contain ferrosilicon as the heavy medium.

A discussion of methods and apparatus for sink-float separation of ores is found on pages 11-104 to 11-124 of the textbook entitled Handbook of Mineral Dressing-Ores and Industrial Minerals, by A. T. Taggart, John Wiley & Sons, New York, fourth printing, 1950. This discussion is incorporated herein by reference. Use of ferrosilicon in this process is discussed on page 11-111. Wet magnetic separation of the ferrosilicon is discussed on page 10-113.

As indicated above, it is known to use ferrosilicon in a fine grain size, as obtained for example by reducing coarser ferrosilicon grain by means of nozzles, as a heavy-medium solid for use in heavy-media baths employed in sink-float processing of ores. In sink-float operations the heavy-medium bath inevitably becomes contaminated by the fines resulting from wearing of the ore by flotation. This changes the apparent specific gravity of the bath, thus making it impossible to use the bath over prolonged periods unless it is purified.

If the contaminating mineral fines are not magnetizable, the bath can readily be freed from contaminants by magnetic separation. As indicated above the ferrosilicon itself is separated from the bath by magnetic methods, and can be returned to the bath circulation, to again act as weighting medium. However, if the fines are magnetizable, for example when magnetite is being processed, the magnetic separation method cannot be employed because the magnet will attract both the magnetizable ferrosilicon and the magnetizable components of the fines produced by friction-weathering. Purification of the bath, in this case, encounters great difficulties. Attempts to recondition the bath by wet-mechanical means, for example on shaker or impact hearths, were not successful. This is due to the fact that the ferrosilicon grains, because of their spherical shape, are not discharged from the impact hearth in accordance with their specific gravity, the grains being discharged together with the lighter constituents of the bath.

An object of this invention is to devise a method for purifying ferrosilicon heavy floatation baths.

Another object of this invention is to provide a method of this kind which can be used successfully when magnetizable fines, resulting from friction-weather of the ore, are contained in the bath.

To this end, and in accordance with our invention, we perform the purification by subjecting the ferrosilicon in the bath to flotation while using, as collector, non-active substances with twelve or more carbon atoms in the alkyl group.

According to a more specific feature of our invention, the purification by flotation of the magnetizable ferrosilicon is effected by using as collectors cation-active substances, for example salts of alkyl amines, having twelve or more carbon atoms in the alkyl group.

Also suitable as collectors in the method of the present invention are anion-active substances, for example alkyl sulfonates R—OSO₃Na having twelve or more carbon atoms in the alkyl group, preferably in conjunction with froth-reducing substances, for example oleic acid, which substances not only diminish the amount of froth being formed but also increase the strength of the froth. In the latter case it has been found particularly advantageous to use the sulfonates with oleic acid in a ratio by weight of 1:3 in mixture, when the flotation is performed in an acidic bath.

The new method permits recovering ferrosilicon from a contaminated heavy-medium bath and its re-employment for preparing a fresh heavy-medium bath.

The following are two examples of the new method. Both examples are directed to flotation of a ferrosilicon containing heavy-medium bath which was previously used as bath in a sink-float operation for a prolonged period of time, and which has become contaminated with approximately 30% by weight of foreign substances mainly consisting of a magnetite-containing iron silicate.

**Example I**

To the contaminated heavy-medium bath were added 1 kg. waterglass per 1 t. heavy bath for dispersing the fine slime, and immediately thereafter 0.65 kg. oleylamyl acetate (salt of acetic acid and oleylamine) per 1 t. heavy-medium bath to act as collector. Thereafter the mixture was stirred for five minutes in order to permit the reaction agents to become active. Subsequently the stirred mixture was supplied to a flotation cell. Flotation was carried out for a duration of eight minutes. The results are indicated in the following table.

<table>
<thead>
<tr>
<th>Product</th>
<th>Percent by Weight</th>
<th>Specific Gravity</th>
<th>Recovery (percent)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Concentrate</td>
<td>77.80</td>
<td>6.15</td>
<td>99.30</td>
</tr>
<tr>
<td>Tailings</td>
<td>22.20</td>
<td>6.46</td>
<td>0.70</td>
</tr>
</tbody>
</table>

| Feed       | 100.00            | 5.77             | 100.00            |

**Example II**

To the contaminated heavy bath were added 1 kg. waterglass per 1 t. heavy bath for dispersing the slime, and 3.0 kg. sulfuric acid per 1 t. heavy bath for adjusting to a pH value of 6.0. Thereafter, 0.1 kg. oleyl-alcohol-sulfate in mixture with 0.3 kg. oleic acid per 1 t. heavy-medium bath were added to reduce the quantity of froth formed and to increase the strength of the froth. The bath was thereafter stirred for five minutes to allow the flotation agents to take effect. Subsequently, the heavy bath was subjected to flotation in a flotation cell for fifteen minutes. The results are indicated in the following table:

<table>
<thead>
<tr>
<th>Product</th>
<th>Percent by Weight</th>
<th>Specific Gravity</th>
<th>Recovery (percent)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Concentrate</td>
<td>78.63</td>
<td>6.12</td>
<td>95.45</td>
</tr>
<tr>
<td>Tailings</td>
<td>21.37</td>
<td>4.53</td>
<td>4.53</td>
</tr>
<tr>
<td>Feed</td>
<td>100.00</td>
<td>5.77</td>
<td>100.00</td>
</tr>
</tbody>
</table>

The oleyl-alcohol-sulfate used in Example II is a sulfuric-acid ester of oleyl-alcohol, having a structural formula of C₂₅H₄₇SO₃Na.

As is apparent from the two above examples, the method of the invention makes it possible to recover practically the entire ferrosilicon content of a heavy-medium bath in form of flotation concentrate. The flotation concentrate is then sprayed with water to destroy the froth and to remove the flotation reaction agents. Thereafter, the...
recovered product is provided with the quantity of water needed for the particular concentration to be used in the subsequent float-sink separation. The suspension is then suitable for reuse as a float-sink heavy bath.

It has also been found particularly advantageous to add to the flotation concentrate, wetting agents, such as for example alkali hydroxide, alkali silicate, or similar substances prior to spraying the flotation concentrate with water for froth removal. This reduces the flocculation of the ferrosilicon resulting from the action of the collector, and also reduces the viscosity of the bath.

In the methods according to the invention as described so far, the ferrosilicon is floated together with the froth as mentioned, but, as mentioned, some flocculation of the ferrosilicon may occur.

It is another object of our invention further improve the above-described methods to the extent that no flocculation of the ferrosilicon will occur. We have found, according to further features of our invention, that this result is achieved by floating the magnetizable impurity minerals in the above-described manner while using as collectors cation-active means, for example resinous amines with 18 or more carbon atoms in the alkyl group and effecting the flotation in an alkaline heavy-medium bath.

Suitable as collectors and simultaneously as froth-forming agents are also anion-active means, for example lauroyl-sarcoside:

\[
\text{C}_18\text{H}_{37}\text{C}_8\text{H}_4=\text{CO} \\
\text{H}_3\text{C}=-\text{N}-\text{CH}_3\text{COONa}
\]

In which case, however, the flotation is carried out in an acidic bath.

According to another feature of the invention, synthetic cellulose derivatives, for example Tylose VHR (cellulose ether wherein two of the four oxy groups are etherified) can be used for depressing the ferrosilicon.

In the improved method according to the invention, the impurities contained in the heavy-medium bath are subjected to flotation together with the froth, whereas the magnetizable ferrosilicon, i.e. the heavy medium, remains in the remainder.

In each of the two examples (III and IV) of the latter method further described below, a ferrosilicon heavy bath was purified which had been previously used for a prolonged period of time as a heavy-medium bath in a float-sink process. This contaminated heavy-medium bath was first subjected to magnetic separation for eliminating the non-magnetic impurities. The magnetic product of this separation (ferrosilicon and magnetic impurities) was thereafter passed through a de-magnetizing coil and was then placed upon a screen for eliminating those impurities whose grain size was greater than that of the ferrosilicon particles. The material passing through the screen contained practically all of the ferrosilicon with a grain size below 200 microns (the major portion being between 60 and 150 microns) as well as the magnetizable ore fines of approximately the same upper grain-size limit. This material which had passed through the screen was then floated. The ore fines contained in the heavy bath subjected to flotation consisted predominantly of magnetite-containing iron-ore particles and was floated with the froth concentrate, whereas the ferrosilicon was contained in the remainder which was again employed for the preparation of heavy baths in the above-mentioned float-sink process.

Details concerning the flotation and its results are stated below.

Example III

To the contaminated heavy-medium bath were added 600 g. soda (sodium carbonate) per 1 t. solids for alkalizing the bath, 200 g. Tylose VHR (cellulose ether wherein two of the four oxy groups are etherified) per 1 t. solids as depressing medium for the ferrosilicon, 250 g. Rosinamine D-acetate per 1 t. solids as collector, and 100 g. Dowfroth (polypropylene glycol ether) per 1 t. solids as froth-forming agent. The duration of the flotation was 15 minutes. The results are stated in the following table.

<table>
<thead>
<tr>
<th>Product</th>
<th>Percent by Weight</th>
<th>Specific Gravity</th>
<th>Ferrosilicon</th>
<th>Impurities</th>
</tr>
</thead>
<tbody>
<tr>
<td>Concentrate</td>
<td>90.9</td>
<td>5.08</td>
<td>4.9</td>
<td>25.4</td>
</tr>
<tr>
<td>Tailings</td>
<td>65.1</td>
<td>3.68</td>
<td>50.1</td>
<td>24.4</td>
</tr>
<tr>
<td>Feed</td>
<td>100.0</td>
<td>5.89</td>
<td>100.0</td>
<td>100.0</td>
</tr>
</tbody>
</table>

Example IV

Added to the heavy-medium bath were 1500 g. sulfuric acid per 1 t. solids for acidulating the bath, 150 g. Tylose VHR per 1 t. solids as compressing medium for the ferrosilicon, and 800 g. lauroyl-sarcoside per 1 t. solids as collector-frother. The duration of the flotation was 20 minutes. The results are stated in the following table:

<table>
<thead>
<tr>
<th>Product</th>
<th>Percent by Weight</th>
<th>Specific Gravity</th>
<th>Ferrosilicon</th>
<th>Impurities</th>
</tr>
</thead>
<tbody>
<tr>
<td>Concentrate</td>
<td>37.6</td>
<td>4.59</td>
<td>2.3</td>
<td>81.1</td>
</tr>
<tr>
<td>Tailings</td>
<td>62.4</td>
<td>4.05</td>
<td>58.1</td>
<td>7.7</td>
</tr>
<tr>
<td>Feed</td>
<td>100.0</td>
<td>5.89</td>
<td>100.0</td>
<td>100.0</td>
</tr>
</tbody>
</table>

Examples III and IV show that the method according to the invention affords a good separation of the ferrosilicon from the ore fines and increases the specific gravity of the heavy medium (tailings) and consequently the resulting heavy-medium bath.

The content of the recovered ferrosilicon in the tailings can be further increased by extending the flotation over a longer period of time. This, however, also has the effect that a higher percentage of ferrosilicon is floated together with the froth. For receiving this latter share of ferrosilicon, a further flotation of the concentrates obtained together with the froth is necessary.

The methods according to the invention need not necessarily be carried out continuously during the float-sink process for purifying the heavy-medium bath. It rather suffices if the bath is purified periodically from time to time as soon as its specific gravity has changed or its consistency has increased, by the higher content of solids caused by impurities, to such an extent that no proper float-sink separation can be continued.

Obviously, many modifications and variations of the present invention are possible in the light of the above teachings. It is therefore to be understood, that within the scope of the appended claims, the invention may be practiced otherwise than as specifically described.

We claim:

1. In a process for sink-float separation of a mixture of solid particles, said mixture including a ferromagnetic material, said process comprising subjecting said mixture to the buoyant action of a body of fluid having such density that it will float the lighter solid particles of said mixture while the heavier particles sink, said body of fluid being a bath comprising magnetizable ferrosilicon as the heavy-medium solid in a suspending liquid, and returning said ferrosilicon, recovered from the body of fluid, to the process the improvement wherein comprising recovering ferrosilicon by subjecting the said heavy-medium bath, that has become contaminated by ferromagnetic fines during said process, to flotation of the ferrosilicon into a flotation concentrate and depressing of said ferromagnetic fines into said bath with the aid and in the presence of an ion-active substance, the ion-active substance being an alkylamine having an open straight chain linkage of at least twelve carbon atoms in the alkyl group, the flotation being carried out in a bath having a pH not greater than 7.0, and...
removing the flotation concentrate containing the ferrosilicon.

2. In a process for sink-float separation of a mixture of solid particles, said mixture including a ferromagnetic material, the said process comprising subjecting said mixture to the buoyant action of a body of fluid having such density that it will float the lighter solid particles of said mixture while the heavier particles sink, said body of fluid being a bath comprising magnetizable ferrosilicon as the heavy-medium solid in a suspending liquid, and returning ferrosilicon, recovered from the body of fluid, to the process; the improvement therein comprising recovering ferrosilicon by subjecting the said heavy-medium bath, that has become contaminated by ferromagnetic fines during said process, to flotation of the ferrosilicon into a flotation concentrate and sinking of said ferromagnetic fines into said bath with the aid and in the presence of oleylamine acetate, the flotation being carried out in a bath having a pH not greater than 7.0 and removing the flotation concentrate containing the ferrosilicon.

3. In a process for sink-float separation of a mixture of solid particles, said mixture including a ferromagnetic material, the said process comprising subjecting said mixture to the buoyant action of a body of fluid having such density that it will float the lighter solid particles of said mixture while the heavier particles sink, said body of fluid being a bath comprising magnetizable ferrosilicon as the heavy-medium solid in a suspending liquid; the improvement therein comprising recovering ferrosilicon by subjecting the said heavy-medium bath, that has become contaminated by ferromagnetic fines during said process, to flotation of the ferrosilicon into a flotation concentrate and sinking of said ferromagnetic fines into said bath with the aid and in the presence of oleylamine acetate, the flotation being carried out in a bath having a pH not greater than 7.0, removing a flotation concentrate containing the ferrosilicon, spraying the concentrate with water to destroy froth and suspending the flotation agents, adding water and recycling the aqueous ferrosilicon mixture to the process, an anti-flocculant agent being added at a stage prior to spraying to reduce floccling of the ferrosilicon by the ion-active substance.

6. In a process for sink-float separation of a mixture of solid particles wherein said mixture includes ferromagnetic ore material, said process comprising subjecting said mixture to the buoyant action of a body of fluid having such density that it will float the lighter solid particles of said mixture while the heavier particles sink, said body of fluid being a bath comprising magnetizable ferrosilicon as the heavy-medium solid in a suspending liquid; the improvement therein comprising recovering ferrosilicon by subjecting the said heavy-medium bath, that has become contaminated by ferromagnetic ore fines during said process, to flotation of the ferrosilicon into a flotation concentrate and sinking of said ferromagnetic ore fines into said bath with the aid and in the presence of a non-reactive substance, said non-reactive substance being a reagent selected from the group consisting of an alkyl amine having at least 12 carbon atoms in its alkyl group, and a salt of an alkyl amine having at least 12 carbon atoms in its alkyl group, the flotation being carried out in a bath having a pH not greater than 7.0, removing the flotation concentrate containing the ferrosilicon, spraying the concentrate with water to destroy froth and to remove the flotation agents, adding water and recycling the aqueous ferrosilicon mixture to the process, alkali silicate being added at a stage prior to the spraying to reduce floccling of the ferrosilicon by the ion-active substance.

4. In a process for sink-float separation of a mixture of solid particles, said mixture including a ferromagnetic material, the said process comprising subjecting said mixture to the buoyant action of a body of fluid having such density that it will float the lighter solid particles of said mixture while the heavier particles sink, said body of fluid being a bath comprising magnetizable ferrosilicon as the heavy-medium solid in a suspending liquid, and returning ferrosilicon, recovered from the body of fluid, to the process; the improvement comprising recovering ferrosilicon by subjecting the said heavy-medium bath, that has become contaminated by ferromagnetic fines during said process, to flotation of the ferrosilicon into a flotation concentrate and sinking of said ferromagnetic fines into said bath with the aid and in the presence of an ion-active substance, said ion-active substance being a reagent selected from the group consisting of an alkyl amine having at least 12 carbon atoms in its alkyl group, and a salt of an alkyl amine having at least 12 carbon atoms in its alkyl group, the flotation being carried out in a bath having a pH not greater than 7.0, removing the flotation concentrate containing the ferrosilicon, spraying the concentrate with water to destroy froth and to remove the flotation agents, adding water and recycling the aqueous ferrosilicon mixture to the process, alkali silicate being added at a stage prior to the spraying to reduce floccling of the ferrosilicon by the ion-active substance.

5. In a process for sink-float separation of a mixture of solid particles, said mixture including a ferromagnetic material, the said process comprising subjecting said mixture to the buoyant action of a body of fluid having such density that it will float the lighter solid particles of said mixture while the heavier particles sink, said body of fluid being a bath comprising magnetizable ferrosilicon as the heavy-medium solid in a suspending liquid; the improvement therein comprising recovering ferrosilicon by subjecting the said heavy-medium bath, that has become contaminated by ferromagnetic fines during said process, to flotation of the ferrosilicon into a flotation concentrate and sinking of said ferromagnetic fines into said bath with the aid and in the presence of an ion-active substance, said ion-active substance being a reagent selected from the group consisting of an alkyl amine having at least 12 carbon atoms in its alkyl group, and a salt of an alkyl amine having at least 12 carbon atoms in its alkyl group, the flotation being carried out in a bath having a pH not greater than 7.0, removing a flotation concentrate containing the ferrosilicon, spraying the concentrate with water to destroy froth and suspending the flotation agents, adding water and recycling the aqueous ferrosilicon mixture to the process, an anti-flocculant agent being added at a stage prior to spraying to reduce floccling of the ferrosilicon by the ion-active substance.

7. In a process for sink-float separation of a mixture of solid particles wherein said mixture includes ferromagnetic ore material, said process comprising subjecting said mixture to the buoyant action of a body of fluid having such density that it will float the lighter solid particles of said mixture while the heavier particles sink, said body of fluid being a bath comprising magnetizable ferrosilicon as the heavy-medium solid in a suspending liquid; the improvement therein comprising recovering ferrosilicon by subjecting the said heavy-medium bath, that has become contaminated by ferromagnetic ore fines during said process, to flotation of the ferrosilicon into a flotation concentrate and sinking of said ferromagnetic ore fines into said bath with the aid and in the presence of an ion-active substance, said ion-active substance being a reagent selected from the group consisting of an alkyl amine having at least 12 carbon atoms in its alkyl group, and a salt of an alkyl amine having at least 12 carbon atoms in its alkyl group, the flotation being carried out in a bath having a pH not greater than 7.0, removing the flotation concentrate containing the ferrosilicon, spraying the concentrate with water to destroy froth and to remove the flotation agents, adding water and recycling the aqueous ferrosilicon mixture to the process, alkali silicate being added at a stage prior to the spraying to reduce floccling of the ferrosilicon by the ion-active substance.
said ferrosilicon, recovered from the body of fluid, to the process; the improvement therein comprising recovering ferrosilicon by subjecting the said heavy-medium bath, that has become contaminated by ferromagnetic fines during said process, to flotation to depress the ferrosilicon into said bath and to float-off said ferromagnetic fines into a flotation concentrate with the aid and in the presence of an ion-active substance, the ion-active substance being an alkyl resin amine with at least eighteen carbon atoms in the alkyl group, the flotation being carried out in an alkaline bath, whereby the ferrosilicon is retained in the bath substantially free of said ferromagnetic contaminants and said bath is again suitable for use in said sink-float separation process for treating ferromagnetic material.

9. In a process for sink-float separation of a mixture of solid particles, said mixture including a ferromagnetic material, said process comprising subjecting said mixture to the buoyant action of a body of fluid having such density that it will float the lighter solid particles of said mixture while the heavier particles sink, said body of fluid being a bath comprising magnetizable ferrosilicon as the heavy-medium solid in a suspending liquid, and returning said ferrosilicon, recovered from the body of fluid, to the process; the improvement therein comprising recovering ferrosilicon by subjecting the said heavy-medium bath, that has become contaminated by ferromagnetic fines during said process, to flotation to depress the ferrosilicon into said bath and to float-off said ferromagnetic fines into a flotation concentrate with the aid and in the presence of lauroyl-sarcoside, the flotation being carried out in an acidic bath, whereby the ferrosilicon is retained in the bath substantially free of said ferromagnetic contaminants and said bath is again suitable for use in said sink-float separation process for treating ferromagnetic material.

10. Method according to claim 7, including the step of adding a selective depressing agent comprising a synthetic cellulose derivative for depressing the ferrosilicon without depressing the ferromagnetic ore fines.

11. Method according to claim 7, including the step of adding a selective depressing agent consisting of a cellulose ether wherein two of the four oxy groups are etherified, for depressing the ferrosilicon without depressing the ferromagnetic ore fines.

References Cited in the file of this patent

UNITED STATES PATENTS

2,450,720 Vaney Oct. 5, 1948
2,607,485 Swainson et al. Aug. 19, 1952
2,728,724 Gloor Dec. 27, 1955

OTHER REFERENCES

Fatty and Resin Acids as Collectors for Iron Oxides, Bureau of Mines, RI 5498, United States Department of Interior, 1959.