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TEMPERATURE CONTROL IN A FLOTATION PROCESS

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23 Claims. (Cl. 209—167)

This invention relates to improvements in the so-called flotation process for the separation of minerals in liquid suspension, and its primary object is to promote and expedite the separative action, by temperature regulation at one or more stages of the process.

While the invention may be applied to the treatment of different ores, it is particularly adapted for use in the concentration of ores containing minerals of widely differing solubility characteristics, and it is another object of the invention to increase the differentiating effect of flotation reagents upon minerals in suspension in a flotation process, by maintaining a temperature condition under which the differential action of the reagents upon the minerals is accentuated to a degree conducive toward effecting a substantially clean separation of one mineral from another mineral or from foreign matter with which it is associated in the natural formation of the ore.

It is customary in the flotation process to produce the required aeration of the material by mechanical agitation, and a further object of the invention may be found in the provision of a cooling influence which counteracts the increase in temperature incident to the agitational action upon the material.

The invention is of especial value in a process of separating sodium chloride or halite and potassium chloride or sylvite by flotation and the following description will be made with reference to the application of the invention to ores of this particular character.

The solubility of both halite and sylvite is comparatively high and the degree of solubility of the sylvite is considerably higher than that of the halite. It follows that under temperature variations in a pulp in which these minerals are suspended, the solubility of the halite changes very slowly in comparison with that of the sylvite.

Since the flotation process is dependent upon the differential action of reagents upon a mineral to be separated, by coating the particles thereof with a substance which causes the same to adhere to rising air-bubbles, the process is essentially a surface phenomenon. Minerals or other matter in the ore which are to be separated from the rising mineral either are not coated or are differentially coated, so that only those particles coated with a substance which causes them to adhere to the rising air-bubbles will rise to the surface of the liquid mass, to be recovered by overflow or the like.

It follows that an ideal situation in the flotation process will be that in which all the halite in the material is constantly present in solid form, and the proper differential action on the surface of its particles will be effectively produced and maintained throughout the process.

The sylvite owing to its high rate of solubility with temperature increase, is more affected by temperature variations than the halite. Normally neither mineral will, by itself, collect in the froth of the flotation cell and to produce the most stable flotation, the halite surfaces must be subjected to the reaction of the reagent while the sylvite surfaces may be differentially reacted upon or remain comparatively in their natural condition. Temperature conditions in the material are thus an influence which to a large extent controls the separative action.

If by the mechanical agitation of the material or by other causes such as climatic changes, the temperature of the material increases, part of the metallic particles are brought into solution or the delicate flotation surfaces are dissolved off. Also varying temperatures at different stages of the process will cause erratic flotation.

With the above facts in mind it will be seen that by maintaining an even temperature throughout the material under treatment, at which substantially all the material is maintained in suspension, or prevented from going into solution, a clean separation of the sylvite from the halite may be effected.

At a lowered temperature, sylvite will deposit out of solution onto the existing sylvite surfaces, and be recovered in the potassium chloride concentration, thereby promoting its separation from the sodium chloride rising to the surface.

If at the higher temperature coated halite surfaces are covered by other halite deposited out of solution, additional flotation agent can be used to maintain the flotation surface, particularly at the tail end of the flotation period.

Ordinarily a reduction of temperature is required to produce the desired result, and this may be accomplished through the medium of a
jacket around the flotation cell, or a coil within the same, through which a cooling fluid is circulated. It is also possible however, that due to climatic conditions or other factors, an increase in temperature is necessary to bring the material to or near the ideal condition above stated, and it is to be understood that by referring to temperature-regulation, both conditions are included.

In the accompanying drawing, Figure 1 is a diagram showing the comparative solubility characteristics of the potassium chloride and sodium chloride in an aqueous pulp of the ore.

Figure 2 is an outline of a flotation cell to which the invention is applied.

Figure 3 is a similar view showing a different method of temperature-regulation.

Figure 4 is a diagrammatic view of a system for recovering the sylvite in a flotation process, showing the application of the invention at different stages thereof, and Figure 5 is a modification of the system depicted in Figure 4.

In the treatment of halite-sylvite ores by flotation, the ore in a finely-divided condition, is mixed in a saturated solution of the minerals, in a flotation vessel of conventional form. In this cell the material is mechanically agitated and aerated by one or more rotating propellers, and it is at the same time subjected to the influence of suitable flotation agents.

In accordance with the present invention, the material in the cell is at the same time cooled to an even temperature of predetermined degree by the use of a jacket around the vessel or a coil within the same through which a cooling fluid, either liquid or gaseous, is circulated. If as stated hereinafter, it is required to increase the temperature of the material, a heating fluid is, of course, substituted for the cooling fluid.

Repeating further to the drawing, the material is fed into the flotation vessel at 5, the froth with the mineral entrapped therein, is discharged by overflow as at 7, and matter other than that rising to the surface is discharged at the bottom of the vessel at 8. The pulp in the vessel is aerated and agitated by rotating propellers as shown at 8. In Figure 2, a cooling fluid entering at 10 passes through a jacket 12 around the vessel, and leaves the jacket at 13 after having lowered the temperature of the material inside the vessel to a predetermined degree.

In Figure 3, a coil 14 inside the vessel is substituted for the jacket.

In Figures 4 and 5 is shown a system by which the froth delivered from the flotation vessel is treated under temperature regulation, to recover sylvite which may have escaped with the halite, after which the halite is recovered from the brine solution and the latter returned to the flotation vessel for reuse.

Repeating first to Figure 4, the overflow of the flotation vessel passes in the direction of the line 15 to a vessel 16, containing an agitator 17. In this vessel the sodium chloride or halite froth is heated through the medium of a coil 18, causing any sylvite which escaped with the halite due to attachments or other causes, to dissolve. The warmed pulp then overflows as at 19 into a thickener 20 where the sylvite-depleted halite settles out and the solution returns in the direction 21 to the head or feed end of the flotation vessel.

Excess sylvite in solution, will be deposited out of the solution by the cooling influence in the flotation vessel, to be recovered with the sylvite concentrate at 8.

In Figure 5, the heating fluid enters the coil 18 of the agitator 16 from a compressor 22, and after having heated the material in the agitator passes in a cooled condition, past an expansion valve 23 to the coil 14 or jacket 12 of the flotation vessel. Heating coils 24 in the agitator provide additional heat to balance radiation losses and heat loss with halite waste.

While in the treatment of ores in accordance with the invention, the critical temperature at which the material is maintained must necessarily vary in accordance with varying conditions, it has been found that in the flotation of potassium chloride ores, a temperature of approximately sixteen degrees centigrade in the flotation vessel has produced satisfactory results, in the presence of a reagent composed of a brine-soluble fatty acid and a brine-soluble metallic cation, which has been the subject of an application for patent, separate from the present one, namely, Serial No. 755,577, filed December 1934, for Flotation process. My co-pending applications, Serial No. 7,219, filed February 19, 1935, for Concentration process, Serial No. 11,669, filed March 14, 1935, for Flotation process and reagent, and 28,985, filed June 28, 1935, also disclose other reagents and treatments well suited for use in the froth flotation of these ores. Subject matter disclosed but not claimed herein have been claimed in the aforementioned applications.

It is to be understood that the liquid phase of the treatment is a saturated solution of sodium chloride and potassium chloride, and the solubility of halite and sylvite in this solution have been illustrated in Figure 1. The upright column of figures represents grams of KCl or NaCl per 100 grams of water and the column of figures extending along the bottom of the chart represents temperature in degrees centigrade.

The solubility characteristics of these minerals in a solution of the same is tabulated on page 77 of "Fortschritte in Kali-Industrie", by Dr. C. Hermann, Berlin, published by Theodore Steinkopf, Dresden and Leipzig, 1927, as follows:

<table>
<thead>
<tr>
<th>Temperature °C.</th>
<th>KCl</th>
<th>NaCl</th>
</tr>
</thead>
<tbody>
<tr>
<td>10</td>
<td>12.5</td>
<td>20.7</td>
</tr>
<tr>
<td>20</td>
<td>14.7</td>
<td>20.2</td>
</tr>
<tr>
<td>30</td>
<td>17.2</td>
<td>20.1</td>
</tr>
<tr>
<td>40</td>
<td>19.5</td>
<td>20.1</td>
</tr>
<tr>
<td>50</td>
<td>21.7</td>
<td>20.3</td>
</tr>
<tr>
<td>60</td>
<td>24.6</td>
<td>27.2</td>
</tr>
<tr>
<td>70</td>
<td>27.3</td>
<td>28.8</td>
</tr>
<tr>
<td>80</td>
<td>30.0</td>
<td>28.4</td>
</tr>
<tr>
<td>90</td>
<td>32.5</td>
<td>28.1</td>
</tr>
<tr>
<td>100</td>
<td>35.9</td>
<td>28.3</td>
</tr>
</tbody>
</table>

The foregoing figures are true, only when there are ample quantities of NaCl and KCl in the solid phase in contact with the liquid phase to permit the latter to become fully saturated with respect to both salts. When the liquid phase is not in contact with a sufficient quantity of the solid phase of one of the salts, the equilibria above noted does not exist.

To permit a complete comprehension of solubility characteristics of the pulp, treated by the present process, the following chart illustrates the equilibria of the system when there is a de-
Equilibria for the system KCl—NaCl—H₂O

<table>
<thead>
<tr>
<th>Temperature degrees Fahrenheit</th>
<th>Per cent. sodium chloride in saturated brine</th>
</tr>
</thead>
<tbody>
<tr>
<td>70</td>
<td>21.2</td>
</tr>
<tr>
<td>80</td>
<td>21.4</td>
</tr>
<tr>
<td>90</td>
<td>21.5</td>
</tr>
<tr>
<td>100</td>
<td>21.7</td>
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<tr>
<td>110</td>
<td>21.9</td>
</tr>
<tr>
<td>120</td>
<td>22.1</td>
</tr>
<tr>
<td>130</td>
<td>22.2</td>
</tr>
<tr>
<td>140</td>
<td>22.4</td>
</tr>
<tr>
<td>150</td>
<td>22.6</td>
</tr>
<tr>
<td>160</td>
<td>22.8</td>
</tr>
<tr>
<td>170</td>
<td>23.0</td>
</tr>
</tbody>
</table>

The boxed figures are from equation (B) see below.

\[ \% \text{NaCl} = 0.3 + 0.43 \times 10^{-5} \times 70 - 0.43 \times 10^{-5} \times 100 \]

(A) = Equation when deficiency exists in the NaCl solid phase.
(B) = Equation when no deficiency exists in the solid phases.
(C) = Equation when deficiency exists in the KCl solid phase.

2. A selective flotation process for separating soluble constituents of an ore having different solubility characteristics that vary under temperature variations, which comprises introducing a halite-sylvite ore into a saturated solution of the same to form a pulp at a selected temperature, subjecting the pulp to a froth flotation process, including the use of a reagent having a selective affinity for one of said constituents, and separating the said constituents by inducing a crystallization of one of the soluble constituents and dissolution of the other soluble constituent of the saturated solution by raising its temperature above that of the selected temperature.

3. A selective flotation process for separating soluble constituents of an ore having different solubility characteristics that vary under temperature variations, which comprises introducing a halite-sylvite ore into a saturated solution of the same to form a pulp at a selected temperature, subjecting the pulp to a froth flotation process, including the use of a reagent having a selective affinity for one of said constituents, and maintaining the pulp during said treatment at a substantially uniform temperature at which the soluble halite particles will not go into solution in the pulp.

4. A selective flotation process for separating soluble constituents of an ore having different solubility characteristics that vary under temperature variations, which comprises introducing a halite-sylvite ore into a saturated solution of the same to form a pulp, subjecting the pulp to a froth flotation process, including the use of a reagent having a selective affinity for one of said constituents, and maintaining the pulp during the said treatment at a temperature at which the soluble sylvite particles will not go into solution in the pulp.

5. A selective flotation process for separating soluble constituents of an ore having different solubility characteristics that vary under temperature variations, which comprises introducing a halite-sylvite ore into a saturated solution of the same to form a pulp, subjecting the pulp to froth flotation, including the use of a reagent...
having a selective affinity for the halite and gangue constituents, maintaining an operating temperature during flotation at which soluble halite particles in the pulp will not go into solution, then raising the temperature of froth product of flotation to dissolve any syline in the sample, then separating the solids from the solution.

6. A selective flotation process for separating soluble constituents of an ore having different solubility characteristics that vary under temperature variations, which comprises introducing a halite-sylvite ore into a saturated solution of the same to form a pulp, subjecting the pulp to froth flotation, including the use of a reagent having a selective affinity for the halite and gangue constituents, subjecting the pulp during the flotation treatment to a cooling influence sufficient to counteract the rise in temperature due to the froth flotation treatment, recovering syline from the froth product of flotation by heating said product sufficiently to bring syline particles into solution, then removing the solid constituents from the solution, and finally returning the solution containing the dissolved syline for re-use in the flotation treatment.

7. A selective flotation process for separating soluble constituents of an ore having different solubility characteristics that vary under temperature variations, which comprises introducing a halite-sylvite ore into a saturated solution of the same to form a pulp, subjecting the pulp to froth flotation, including the use of a reagent having a selective affinity for the halite and gangue constituents, subjecting the pulp during the flotation treatment to a cooling influence sufficient to counteract any rise in temperature incident to the froth flotation treatment, recovering syline from the froth product of flotation by passing the same into solution in a heated fluid, then removing the solid constituents from the solution, then returning the solution containing the dissolved syline for re-use in the flotation action, cooling said heated fluid, and utilizing it to cool the material during the flotation action.

8. A selective flotation process for separating soluble constituents of an ore having different solubility characteristics that vary under temperature variations, which comprises introducing a halite-sylvite ore into a saturated solution of the same to form a pulp, subjecting the pulp to froth flotation, including the use of a reagent having a selective affinity for the halite and gangue constituents, whereby a froth containing halite and some syline is produced, then agitating the froth product of flotation while raising its temperature sufficient to bring the syline into solution but not high enough to bring the halite into solution, removing the solution from the undissolved constituents of the froth product, and returning said solution for reuse in the flotation treatment.

9. In a method of froth flotation separation of two soluble minerals, one of which is soluble at a lower temperature than the other, the steps of entering a mixture of syline ore into a saturated solution of the ore to form a pulp, subjecting the pulp to a froth flotation treatment in the presence of a flotation reagent having a selective affinity for halite constituents whereby a froth overflow product is formed, maintaining a temperature during the flotation treatment below that at which syline particles will be dissolved, then subjecting the froth overflow product to a temperature high enough to cause particles of syline to pass into solution and below that at which the halite particles will pass into solution, and subjecting the said overflow product to a separation treatment for the removal of solids from the solution.

10. In a flotation process for the treatment of ores containing potassium chloride and sodium chloride, the steps of suspending said particles in a saturated solution of the ore, treating said particles to froth flotation, and maintaining a substantially uniform temperature during the froth flotation treatment, said temperature being one at which the liquid phase will neither deposit out of solution nor dissolve either of said soluble ore constituents.

11. In a flotation process for the treatment of ores containing potassium chloride and sodium chloride suspended in a saturated solution of the ore inclusive of a froth flotation treatment, the steps of maintaining a substantially uniform temperature during the froth flotation treatment, said temperature being one at which the liquid phase will neither deposit out of solution nor dissolve either of said soluble ore constituents.

12. In a flotation process for the treatment of sylvinites ore, the steps of raising the temperature of a product of flotation containing solid phases of potassium chloride and other ore constituents thereby causing the said potassium chloride in the solid phase to enter into solution in its liquid phase, then separating the liquid from the solid phase, and then cooling the separated liquid phase so as to cause potassium chloride solids to separate therefrom.

13. In a flotation process for the treatment of sylvinites ore, the steps of raising the temperature of a product of flotation containing solid phases of potassium chloride and other ore constituents thereby causing the said potassium chloride in the solid phase to enter into solution in its liquid phase, then separating the liquid from the solid phase, and then cooling the separated liquid phase so as to cause potassium chloride solids to separate therefrom.

14. In a flotation process for the treatment of sylvinites ore by suspension in a pulp at a predetermined temperature and treatment to froth flotation, the steps of raising the temperature of the froth product of flotation to cause potassium chloride in the solid phase in said product to enter into solution in the liquid phase.

15. The process of treating sylvinites ore, which comprises introducing sylvinites ore in finely-divided condition into a saturated solution of the ore to form a pulp, subjecting the undissolved sodium chloride particles of the pulp to the action of a froth flotation reagent having a selective affinity therefor, removing the resulting froth carrying sodium chloride, subjecting said froth to heat and agitation, whereby any potassium chloride particles associated with said sodium chloride particles will be dissolved, and then separating the undissolved sodium chloride particles from the solution.

16. In a process of treating sylvinites ore, the steps of subjecting such sylvinites ore in a saturated solution thereof to a flotation treatment, subjecting the froth concentrate of flotation to agitation at a controlled temperature to effect separation between the undissolved mineral par-
articles in the froth by dissolving one of the con-
stituents into the solution, removing the sepa-
rated mineral particles, and returning the solu-
tion to the process.

17. In a process of treating sylvinite ores, the
steps of subjecting such ores in a saturated solu-
tion of the ore to a flotation treatment in the
presence of a reagent having a selective affinity
for sodium chloride, maintaining the tempera-
ture of the flotation pulp substantially uniform
during the flotation operation, and then raising
the temperature of the froth concentrate of the
flotation operation.

18. In a process of treating sylvinite ores, the
steps of subjecting such ores in a saturated solu-
tion of the ore to a flotation treatment in the
presence of a reagent having a selective affinity
for sodium chloride, maintaining the tempera-
ture of the flotation pulp substantially uniform
during the flotation operation, and then raising
the temperature of the froth concentrate of the
flotation operation, whereby any potassium chlo-
ride associated with sodium chloride in the froth
is dissolved and sodium chloride passes out of
solution, separating sodium chloride solids from
the solution, and then lowering the temperature of
the solution to cause potassium chloride to
pass out of solution.

19. In a flotation process for the treatment of
sylvinite ores, by suspension in a pulp at a pre-
determined temperature and treatment to froth
flotation, the step of raising the temperature of
the froth product of flotation to cause sodium
chloride in the liquid phase in said product to
deposit out of solution into solid form.

20. A cyclic froth flotation process for the
treatment of sylvinite ore, which comprises sub-
jecting the ore to froth flotation in a pulp com-
posed of solid phases of the ore suspended in a
liquid phase which is a saturated solution of the
ore, returning the liquid phase to the process for
reuse in flotation at controlled temperature, and
regulating said flotation temperature by subject-
ing said liquid phase to an artificial withdrawal
of heat compensating for the temperature in-
crease generated in the prior cyclic movement.

21. In a cyclic froth flotation process for the
treatment of sylvinite ore, subjecting the ore to
froth flotation at a substantially uniform tem-
perature in a pulp composed of solid phases of
the ore suspended in a liquid phase which is a
saturated solution of the ore, returning the liq-
uid phase to the process for reuse in flotation at
said controlled temperature, and successively
raising and lowering the temperature of the solu-
tion between repetitions of flotation to crystal-
лизate constituents of the saturated solution sepa-
rately at different stages of the cycle.

22. In a cyclic froth flotation process for the
treatment of sylvinite ore, subjecting the ore to
froth flotation at a substantially uniform tem-
perature in a pulp composed of solid phases of
the ore suspended in a liquid phase which is a
saturated solution of the ore, returning the liq-
uid phase to the process for reuse in flotation at
said controlled temperature, and reducing the
temperature of said circulating saturated solu-
tion containing solid ore constituents thereby
enriching the potassium chloride solids content
thereof by deposition of potassium chloride out
of said liquid phase in solid form.

23. In a cyclic froth flotation process for the
treatment of sylvinite ore, subjecting the ore to
froth flotation at a substantially uniform tem-
perature in a pulp composed of solid phases of
the ore suspended in a liquid phase which is a
saturated solution of the ore, returning the liq-
uid phase to the process for reuse in flotation at
said controlled temperature, and subjecting said
circulating pulp as a preliminary to its use in
flotation, to an artificial transfer of heat to com-
pensate for its change in temperature occasioned
in the cyclic movement.

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