METHOD OF SEPARATING SYLVITE FROM SYLVINITE ORE

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SYLVINITE ORE

GRINDING

SIZING

FINES SEPARATION

GROUND ORE STORAGE

WASHING

COOLER

SOLIDS SLURRY TO FILTRATION

DRYING

CONDENSER

CHARGING

WASHING MEDIUM

FINES

GROUND ORE STORAGE

% KCI IN TAILS

% KCI IN CONC.

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2,839,190

METHOD OF SEPARATING SYLVITE FROM SYLVINITE ORE

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9 Claims. (Cl. 209—127)

The instant invention relates to the separation of sylvite from sylvite-bearing minerals. Still more particularly, it relates to the electrostatic separation of halite and sylvite occurring naturally or produced artificially.

Separation of sylvite from potash ore such as sylvite ore may be accomplished by a variety of methods. In one method, the potassium chloride is extracted with a hot solution saturated with respect to sodium chloride. The solution is flash-cooled to deposit potassium chloride crystals which are then filtered, washed, and dried. The mother liquor is then recycled.

In another process, sylvite ore is pulverized and flotation agents are used to convey potassium chloride away from the sodium chloride or halite. Following flotation, the sylvite is water-washed to partially leach away residual halite, and dried.

These processes have one or more disadvantages. The process wherein potassium chloride is extracted from hot saturated brine, for example, is expensive, due to the large quantities of potassium chloride remaining uncrystallized and recycled in the mother liquor, and due to the large heat requirements. In the flotation process, processing costs are relatively high because of the nonrecoverability of expensive reagents utilized to selectively coat one of the ore constituents and thereby selectively float the sylvite or halite away from the other nonfloated material with which it is associated in nature.

It is well known that particles of minerals which can be induced to take electrical charges of opposite sign can be separated from one another by a number of systems whereby electrical equipment sets up repulsive forces for particles having one charge, and attractive forces for particles of opposite charge. In one form of apparatus involving a charged electrical drum, particles having a charge opposite to that of the drum are held by attraction to the surface of the drum and thus moved through an arc of a greater number of degrees than particles having the same charge as the drum. The material dropping from the roll or drum thus may be caught in different hoppers or chutes.

Such systems have, however, failed to effect any appreciable separation of dry potassium chloride particles from other dry ingredients found in association with potassium chloride ores. The systems found effective to induce dry particles of other minerals to accept an appreciable and substantial electrostatic charge have not been effective in the treatment of potassium chloride particles, because the sylvite particles have not become sufficiently strongly charged with reference to the gangue compounds of the ore to effect a selective electrostatic separation.

It is an object of the present invention to overcome the limitations and disadvantages of the above discussed prior art.

It is an object of the present invention to provide a relatively inexpensive method of recovering sylvite from sylvite-containing ores.

It is a further object to provide a beneficiation method wherein the use of costly reagents such as are employed in flotation processes is avoided.

It is still another object to provide a method wherein the dry products are separated and recovered in condition ready for shipment.

It is still another object to provide a method for the recovery of sylvite in which the ore is comminuted and treated so that the sylvite particles will accept a definite charge and then, when selectively charged, the particles are separated by deflection from a straight path when falling free in an electrostatic field.

These and other objects of the present invention will become more apparent from the following description in which one embodiment of the invention will be set forth in detail.

It has now been discovered that sylvite ore such as is found in the Carlsbad section of New Mexico can be separated into its component parts, i.e., sylvite can be separated from sodium chloride or halite particles if such particles are properly washed prior to charging, then selectively charged and subjected to the attractive or repulsive forces of an electrostatic field.

The process of the present invention comprises crushing sylvite ore containing potassium chloride or sylvite to produce a granular liberated product, washing the granular product with a medium in which at least the potassium chloride component, preferably both components, of the ore constituents is substantially insoluble, inducing the washed particles to accept an electric charge, and passing the charged granular material as freely-falling bodies through a zone where the particles deviate from a straight path due to the forces exerted by an electrostatic field, the deflection of the differently charged particles towards poles of opposite polarity resulting in separation of the chemically dissimilar particles.

In the operation of this process the feed material, generally a raw ore, is crushed sufficiently to unlock its constituents and screened by the usual procedures to produce a granular product having a particle size in the range of approximately —24 mesh and +200 mesh determined by standard screens. Particles in this size range are sufficiently comminuted or disintegrated to separate the ore into individual particles of sylvite on the one hand, and minerals associated therewith, such as halite, on the other hand.

Comminuted particles of sylvite-bearing ore, such as sylvite ore from the Carlsbad section of New Mexico, mixed ore (i.e., potassium magnesium sulfate, potassium chloride and sodium chloride), and like ore material, after sizing are subjected to a washing step. The washing step effects removal of slime material from the surface of the particles. The term "slime" as used herein includes fine particles having the same chemical composition as one of the principal components of the ore, as well as material chemically different from the principal components of the ore such as montmorillonite type clays. The washing medium is one in which the potassium chloride, and preferably both sylvite and halite, is substantially insoluble so that there will be substantially no loss of potash. In addition, the washing medium must have sufficient volatility to evaporate leaving a clean surface without requiring the application of high temperatures or extremely low pressures. By "high temperatures" is meant temperatures higher than approximately 300° C. By "low pressures" is meant a vacuum of atmospheric pressure) of, for example, at least 25 inches of mercury. These restrictions do not mean that higher temperatures or other vacuum conditions are not usable, but that such
conditions are not economically feasible in most plant operations. From a purely theoretical standpoint, any limitations, kerosene, benzene, toluene, and the like; liquid oxygen-containing derivatives of hydrocarbons, such as di-chlor ethylene, tri-chlor ethylene, tri-bromo ethylene, and the like; liquid oxygen-containing derivatives of hydrocarbons, e.g., aliphatic alcohols such as methyl alcohol, ethyl alcohol, propyl alcohol, isopropyl alcohol, n-butyl alcohol, sec-butyl alcohol, tertiary butyl alcohol, n-amyl alcohol, methyl n-propyl carboline, and the like; amino, nitro, and halogen substituted aliphatic alcohols, and the like; liquid unsaturated alcohol such as allyl alcohol cyclic alcohols such as furfuryl alcohol, di- allyl carbinitol, and the like. Additional useful washing media from others such as ethyl ether, propyl ether, \( \beta \), dichlor diethyl ether, and the like; mixed ethers such as methylethyl ether, ethylpropyl ether, and the like; esters of organic and inorganic acids such as ethyl acetate, butyl propionate, secondary and tertiary butyl nitrate, ethyl chloride, ethyl bromide, n-hexyl chloride, and equivalent compounds; ketones such as acetone, and the like; and compatible mixtures thereof.

Following the washing, it is necessary that the washing medium be removed and the comminuted sylvaite be rendered substantially dry. In climates where relative humidities of the order of 20% or less prevail, the drying of sylvaite presents no particular problem inasmuch as the hygroscopic particles cannot pick up sufficient moisture to render the particle surfaces moist. In climates where humidity is high, as for example the relative humidity reaches in the range of 75% to 95%, the air contacting the sylvaite must be conditioned to have a low moisture content.

Drying of the particles may be accomplished by heating the washed ore material to a temperature in the range of about 30° C. to about 300° C. For example, propyl alcohol may be evaporated at temperatures in the range of about 30° C. under vacuum and about 100° C. without vacuum, or by blowing hot air through a comminuted material or by subjecting a relatively thin layer of particles to infrared heating rays or other equivalent drying methods.

Drying of the washed ore particles may be carried out in apparatus such as a tunnel dryer, rotary kiln, fluidized bed dryer, or equivalent apparatus. By the positioning of a condensor in the exhaust or vacuum line, the washing medium may be recovered for reuse. Recovery of this washing medium material reduces the processing cost, inasmuch as approximately 97% to 99% recovery can be obtained under good plant operating practice.

After the comminuted material is dried, the particles are induced to accept an electrical charge. As distinguished from other methods in common use wherein, for example, material is not merely polarized as in the case of pyroelectric crystals, the charging of the particles may be and preferably is carried out in the absence of an electric field. Sylvaite particles may be differentially electrified to carry an electrical charge of different character or of different magnitude from the other components of the comminuted mixture. Differential electrification may be created by utilizing the contact potential phenomenon such as by frictional or rubbing contact between particles either when in contact with a grounded donor plate or not. When the quantities of different ore components are not widely disproportionate, contact potential charging may be effectively carried out by agitation or movement of the mixture. Under such conditions a donor plate is not critical to the operation. When charging concentrate, particularly, of relatively high purity, contact potential charging will give only weak charging of one component of the mixture and at this stage use of a donor element is generally advantageous. By "grounded donor plate" is meant an element or pressor function which merely result in the volatilization of the washing medium and which do not fuse or volatilize the salts.

Liquid media useful for the above washing operation are the liquid hydrocarbons such as the hexanes, heptanes, octanes, decanes, kerosene, benzene, toluene, and the like; liquid oxygen-containing derivatives of hydrocarbons, such as di-chlor ethylene, tri-chlor ethylene, tri-bromo ethylene, and the like; liquid oxygen-containing derivatives of hydrocarbons, e.g., aliphatic alcohols such as methyl alcohol, ethyl alcohol, propyl alcohol, isopropyl alcohol, n-butyl alcohol, sec-butyl alcohol, tertiary butyl alcohol, n-amyl alcohol, methyl n-propyl carboline, and the like; amino, nitro, and halogen substituted aliphatic alcohols, and the like; liquid unsaturated alcohol such as allyl alcohol cyclic alcohols such as furfuryl alcohol, diallyl carbinitol, and the like. Additional useful washing media from others such as ethyl ether, propyl ether, \( \beta \), dichlor diethyl ether, and the like; mixed ethers such as methylethyl ether, ethylpropyl ether, and the like; esters of organic and inorganic acids such as ethyl acetate, butyl propionate, secondary and tertiary butyl nitrate, ethyl chloride, ethyl bromide, n-hexyl chloride, and equivalent compounds; ketones such as acetone, and the like; and compatible mixtures thereof.

Carged particles are fed as free-falling bodies between the electrodes of one or more electrostatic separating units, i.e., charged particles are fed to air contacting the sylvaite-bearing ore feed to the separator. The diameter of the electrostatic field which will effectively separate the charged particles will vary with the average particle size of the sylvaite-bearing ore feed to the separator. The voltage may vary from 5,000 volts per inch of distance between electrodes in separating material of relatively fine particle size in the range of approximately 125 to approximately 200 mesh, to 15,000 volts per inch of distance separating electrodes for separating coarser particles. In all such discussion of field strength, it must be borne in mind that corona discharges which ionize air are to be avoided. In general, it is preferred to operate with a total impressed difference in potential of about 70,000 to about 90,000 volts at a voltage of about 20,000 volts and as high as about 200,000 volts are utilized on occasion. This voltage should be maintained at a high direct voltage potential substantially free of alternating current components; i.e., filtered D.C. current should be low in the so-called A.C. ripple. A steady supply of D.C. voltage may also be obtained without expensive filtering apparatus by the use of such equipment as radio frequency power supply.

The process for separating ore into its components will be more fully understood from the following description given in the following example with reference to the separation of sylvaite from sylvinite ore given in conjunction with the drawings in which:

Figure 1 is a diagrammatic flow sheet of thebeneficiation process.

Figure 2 is a chart showing the beneficiation accomplished with and without the washing operation.

For a more detailed illustration of the operation of the instant process, reference is made to Figure 1. In this flow sheet, sylvaite ore is transferred from a storage station 10 by a conveyor belt 11 to a grinding mill 12. At mill 12 the sylvaite ore is broken up into a size substantially completely free from the halite portion of the ore. The crude material is delivered by conveyor 13 to a sizing station 14 and screened to a size generally of −24 mesh, the coarse material being returned by conveyor 15 to grinding mill 12. Fine particles, i.e., generally less than 200 mesh size, are removed at separating station 16 to which the −24 mesh material is delivered by a pneumatic conveyor.
and are discarded. The material of a size in the range of about -24 and about +200 mesh is dropped from separator 16 to a storage or surge bin 18.

Coarse material from surge bin 18 was screened four times to remove fines, heated, charged, and fed to the electrostatic separation apparatus without the wash treatment of the instant invention. The results of this separation are shown in Figure 2. The difference in beneficiation of surge bin ore is clearly shown by comparison with results of the instant process. In carrying out the instant novel process, feed material from surge bin 18 is delivered to washing station 20 by conveyor 19. At washing station 20, the sized ore is preferably fed centrally to a flow of washing medium such as commercial grade ethyl alcohol pumped through pipe 21 from a storage station 22. The washed ore is conveyed through a drying oven 23 by belt 24 where the ore is heated to a temperature of approximately 150°C. Vapors are withdrawn through conduit 25 and are condensed at 26. The recovered liquid is delivered through pipe 27 to storage station 22, from which it is recirculated to the washing station 20. Washing medium plus dissolved and suspended solids from washing station 20 is cooled as at cooler 28 by circulation of cooling medium to crystallize any dissolved solids such as sodium chloride. Solids slurry is removed from cooler 28 and the solids removed from the associated washing medium as by a conventional filter operation. The solids free washing medium from cooler 28 is delivered to storage station 22 through pipe 29. Washed ore issuing from oven 23 in substantially dry condition is passed by conveyor 30 to a grounded donor element 31 which preferably is in the form of a galvanized iron delivery chute where the particles pick up electrons and become charged.

The ore thus charged is subjected, preferably immediately and before appreciable cooling, to an electrostatic field created by a suitable electrostatic separator 32, preferably of the type where the particles, as free-falling bodies, pass between oppositely charged electrode plates spaced six inches apart and having a potential gradient of 5,000 to 12,000 volts per inch of distance separating the electrodes.

Upon passing through electrostatic field, a sylvinite concentrate product 33, a middling fraction 34, and a halite fraction 35 are obtained.

Example I

A sylvinite ore was comminuted and screened to produce a feed having a particle size in the range of -28 and +100 mesh. The ore was washed with an alcoholic medium consisting of methyl alcohol and ethyl alcohol. The washed ore was dried by placing the ore in an electric oven for approximately one hour. When dry, the warm ore was passed over a grounded, galvanized iron chute and then fell between the plate electrodes having a potential gradient of approximately 12,000 volts per inch.

Results are as follows for one pass between the electrodes:

<table>
<thead>
<tr>
<th>Fraction</th>
<th>Percent KCl</th>
<th>Percent NaCl</th>
</tr>
</thead>
<tbody>
<tr>
<td>Feed</td>
<td>100.0</td>
<td>73.0</td>
</tr>
<tr>
<td>KCl</td>
<td>24.0</td>
<td>20.9</td>
</tr>
<tr>
<td>NaCl</td>
<td>20.5</td>
<td>32.4</td>
</tr>
<tr>
<td>Middling</td>
<td>21.7</td>
<td>68.7</td>
</tr>
</tbody>
</table>

Upon passing the middling fractions through additional stages of electrostatic separation, a recovery of sylvinite of approximately 90% was obtained, the final product therefrom having a purity of about 95%.

Example II

A sylvinite ore was comminuted to the same particle size as in Example I. The ore was washed with trichloroethylene using approximately 320 cubic centimeters per 100 grams of ore. The washed ore was dried by placing in an electric oven for approximately one hour. The ore was dried in the same electric oven utilized to dry the washed ore of Example I. The warm ore was charged by passing over a grounded iron chute. The ore and the washed ore fell between electrodes having a potential gradient of approximately 12,000 volts per inch.

Results are as follows for one pass between the electrodes:

<table>
<thead>
<tr>
<th>Fraction</th>
<th>Percent KCl</th>
<th>Percent NaCl</th>
</tr>
</thead>
<tbody>
<tr>
<td>Feed</td>
<td>33.8</td>
<td>91.0</td>
</tr>
<tr>
<td>KCl</td>
<td>4.3</td>
<td>20.5</td>
</tr>
<tr>
<td>NaCl</td>
<td>68.2</td>
<td>20.9</td>
</tr>
<tr>
<td>Middling</td>
<td>32.4</td>
<td>21.7</td>
</tr>
</tbody>
</table>

Figure 2 shows that for one-pass separations, without a washing treatment, the concentration of sylvinite in the tail fraction rises rapidly when increasing the sylvinite content of the concentrate. On the other hand, when utilizing alcohol washed ore, the sylvinite content of the tail fraction increases only slowly until attempts are made to raise the sylvinite content of the concentrate above 77%.

This application is a continuation-in-part of my application Serial No. 199,023, filed December 4, 1950, now forfeited, and entitled, Method of Separating Sylvinite From Sylvinite Ore.

Having thus fully described and illustrated the character of the invention, what is desired to be secured and claimed by Letters Patent is:

1. The method of beneficiating potash ores containing sylvinite comprising comminuting the ore, washing the comminuted ore with a substantially nonaqueous, nonresidue forming organic liquid medium in which at least the sylvinite component of the ore constituents is substantially insoluble, drying the washed ore, selectively inducing transfer of electrons to the washed particles thereby creating a charge thereon, subjecting the charged ore to the attractive and repulsive forces of a high potential electrostatic field, and recovering a product rich in sylvinite content and a product low in sylvinite content.

2. The method of beneficiating potash ores containing sylvinite comprising comminuting the ore, washing the comminuted ore with a substantially nonaqueous, nonresidue forming organic liquid medium in which at least the sylvinite component of the ore constituents is substantially insoluble, heating the washed ore to a temperature in the range between about 60°F. and about 300°F., whereby the washing medium is evaporated and the ore dried, inducing the hot dried particles to accept an electric charge, subjecting the charged ore to the attractive and repulsive forces of a high potential electrostatic field, and recovering a product rich in sylvinite content and a product low in sylvinite content.

3. The method of beneficiating potash ores containing sylvinite comprising comminuting the ore, washing the ore with a substantially nonaqueous, nonresidue forming organic liquid medium in which at least the sylvinite component of the ore constituents is substantially insoluble, drying the washed ore, selectively inducing the transfer of electrons to the washed particles thereby creating a charge thereon, passing charged ore through an electrostatic field having a potential gradient in the range of between about 5,000 volts per inch and 15,000 volts per inch and having a total impressed difference in potential of between about 20,000 volts and about 200,000 volts, and recovering a product rich in sylvinite content and a product low in sylvinite content.

4. The method of beneficiating potash ores containing sylvinite comprising comminuting the ore, washing the
comminuted ore with a normally liquid medium selected from the group consisting of halogen-containing and oxygen-containing derivatives of hydrocarbons, drying the washed ore, selectively inducing transfer of electrons to the washed particles thereby creating a charge thereon, subjecting the charged ore to the attractive and repulsive forces of a high potential electrostatic field, and recovering a product rich in sylvinite content and a product low in sylvinite content.

5. The method of beneficiating potash ores comprising comminuting the ores, washing the comminuted ore with a substantially nonaqueous alcoholic medium, drying the washed ore, selectively inducing transfer of electrons to the washed particles thereby creating a charge thereon, subjecting the charged ore to the attractive and repulsive forces of a high potential electrostatic field, and recovering a product rich in sylvinite content and a product low in sylvinite content.

6. The method of beneficiating potash ores comprising comminuting the ore, washing the comminuted ore with substantially anhydrous ethyl alcohol, drying the washed ore, selectively inducing transfer of electrons to the washed particles thereby creating a charge thereon, subjecting the charged ore to the attractive and repulsive forces of a high potential electrostatic field, and recovering a product rich in sylvinite content and a product low in sylvinite content.

7. The method of beneficiating potash ores comprising comminuting the ore, washing the comminuted ore with trichloroethylene, drying the washed ore, selectively inducing transfer of electrons to the washed particles thereby creating a charge thereon, subjecting the charged ore to the attractive and repulsive forces of a high potential electrostatic field, and recovering a product rich in sylvinite content and a product low in sylvinite content.

8. The method of beneficiating sylvinite ore comprising comminuting the ore, washing the comminuted ore with a substantially nonaqueous alcoholic medium, heating the washed ore to a temperature in the range of between about 60°F. and about 300°F. and simultaneously subjecting the washed ore to pressures less than atmospheric, condensing the released vapors to recover alcohol for recirculation in the washing system, selectively inducing transfer of electrons to the dry washed particles, subjecting the charged ore to the attractive and repulsive forces of a high potential electrostatic field, and recovering a product rich in sylvinite content and a product low in sylvinite content.

9. The method of beneficiating potash ores comprising comminuting the ore, washing the comminuted ore with a substantially nonaqueous alcoholic medium, heating the washed ore to a temperature in the range of between about 60°F. and about 300°F. and simultaneously subjecting the charged ore to pressures less than atmospheric, condensing the released vapors to recover alcohol for recirculation in the washing system, inducing transfer of electrons to the dry washed particles, passing the charged ore as freely-falling bodies between the electrodes of a high potential electrostatic field, collecting a sylvinite-rich portion and a sylvinite-lean portion and at least one portion of sylvinite contained intermediate therebetween, and recycling the intermediate portion whereby a sylvinite-rich portion is recovered for addition to said sylvinite-rich product.

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