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BENEFICIATION OF LOW GRADE MAGNESITE ORES

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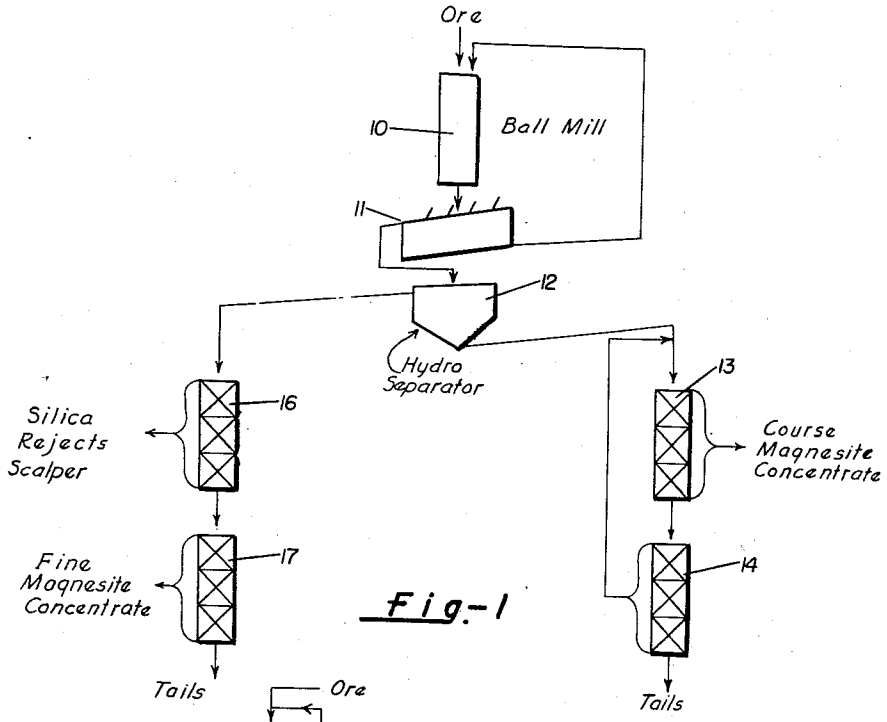


Fig.-1

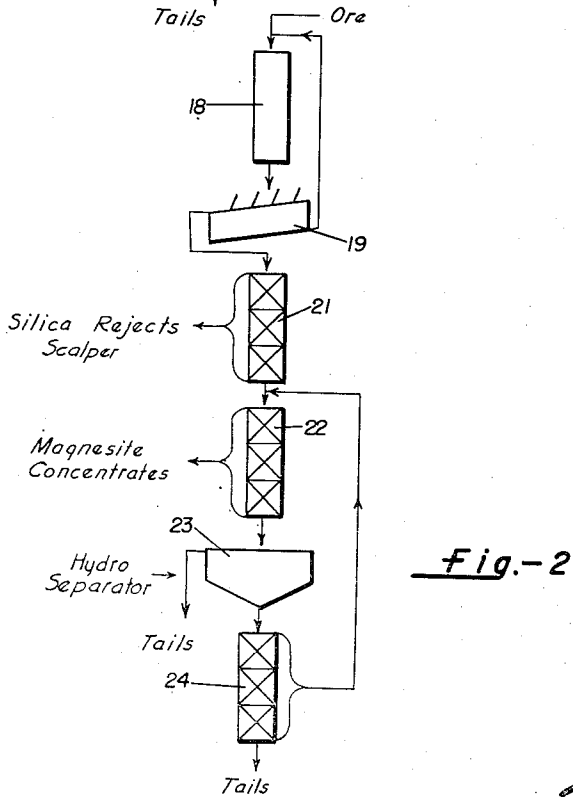


Fig.-2

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BENEFICIATION OF LOW GRADE MAGNESITE ORES

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8 Claims. (Cl. 209—166)

Our invention relates to a process for the treatment of magnesium, refractory-type ores by froth flotation in such manner as to yield a concentrate suitable for use in forming refractory compositions.

Magnesite ores have in the past been subjected to froth flotation to purify the ore sufficiently for the production of metallic magnesium. In such processes considerable care has been required to eliminate, insofar as possible, the naturally occurring impurities, such as silica compounds, limestone, alumina compounds, and the like, with the result that the cost of the process has necessarily been relatively high. The cost of these processes is further accentuated by the grinding characteristics of the material which are such as to yield appreciable quantities of ultra-fine material which must be eliminated, as by scalping, prior to froth flotation if excessive reagent consummation is to be avoided.

According to the present invention we have discovered an economical, efficient process for treating low grade magnesium refractory-type ores. Such ores are of the brucite-magnesite type which are soft, easily ground, and contain such siliceous impurities as serpentine, feldspar, mica, silica, talc, etc. Our process refines the low grade ore to produce a calcined magnesium refractory containing less than about five percent silica. A minor amount, i. e., less than five percent, and preferably 1–2½%, of silica is not detrimental in the calcine, since a certain amount of silica to form a frit is required. Higher siliceous contaminants, however, are detrimental and must be removed to provide a satisfactory refractory from such low grade ore. In calcining, about half the weight is lost as carbon dioxide and water. Thus if head ore is calcined the sinter would run about 6% silicon dioxide, or in instances, even more.

A typical analysis of the low grade ores is:

	Percent
SiO_2	3.0
CaO	3.5
MgO	40.0
R_2O_3	1.0
$HClO_4$ insoluble	3.5
Water and carbon dioxide	Balance

Note that the perchloric acid insoluble amount includes the silicon dioxide amount already listed. This type of ore is low in lime, and may contain substantial amounts of brucite. Generally, magnesite ores contain dolomite as the principal contaminant. The large amounts of brucite substantially change the characteristics of the ore, especially as to flotation beneficiation. The normal procedures for the purification of magnesite are not satisfactory for the brucite-magnesite ores.

One of the principal objects of our invention is the provision of an inexpensive process and collector reagents for treating low grade brucite-magnesite ore in such manner as to yield a concentrate suitable for use in the fabrication of refractories.

Another important object of our invention is to provide

collector reagents for use in the process of the type described which are relatively insensitive to the presence of slimes or ultra-fine particles.

A further object of our invention is the provision of a process of the type described, wherein a substantial portion of the impurities naturally present are eliminated, and some being allowed to remain with the concentrate to assist in the formation of a binder for the refractory composition during subsequent processing.

Additional objects of our invention, which refer to various novel reagents and methods of treatment, will be disclosed in the following detailed description and in the appended drawings, in which:

Fig. 1 is a diagrammatic illustration of a suitable flow sheet incorporating our invention; and

Fig. 2 is a diagrammatic illustration of a modified form of flow sheet incorporating our invention.

In one form of the invention, we grind our ore, which is mined in Nye County, Nevada, to approximately minus 200 mesh. The ore must be finely ground so as to separate the impurities. One impurity is serpentine which is dispersed throughout the ore, and only by grinding to such fine sizes will the process be efficient, economical, and provide a high yield. Preferably, the ore is ground to minus 200 mesh, which provides for substantial separation of the constituents of the ore. The grind to minus 200 mesh gives substantial quantities of ground ore which is about 300 mesh or finer. Since the brucite-magnesite ore is very soft, the grinding is easy. A pulp of the ground ore is then subjected to a mild froth flotation treatment which eliminates from the pulp the easily floated portion of the siliceous contaminants such as the various forms of talc and chloritic minerals. We then add to the pulp, preferably by stages, a magnesium material collector reagent, and subject the pulp to froth flotation, with the result that a refractory grade magnesium material, together with a small proportion of silica is concentrated in the froth overflow. The excess silica contaminant, which is usually in granular form, and the excess calcite, dolomite, etc. and a small amount of magnesite is discharged from the froth flotation stages as tailings. By proper control of reagent addition and processing, the quantities of calcium and silica-bearing contaminants collected in the concentrate may be controlled in such manner that they will permit additions and adjustments to form a suitable binder for the refractory during subsequent processing.

If desired, the ore, after grinding, may be classified prior to froth flotation and only the classifier underflow is subjected to froth flotation, since it has been found that in some ores the classifier overflow is high enough in silicon dioxide for rejection as a tailing. Also, if desired, the magnesium material flotation stage may be divided into primary and scavenger units separated by a classifier. The overflow or fines of this classifier may be discharged from the system without further processing, while the underflow, containing the relatively coarser particles, is subjected to further froth flotation in the scavenger unit, the concentrate from this unit being recycled to the primary flotation cells and the underflow discharged as tailings from the process.

Referring to the drawings, we have illustrated in Fig. 1 a typical flow sheet, wherein brucite-magnesite ore of the type obtained near the town of Gabbs in Nye County, Nevada, is fed in conventional manner to a ball mill **10** with water and discharged into a suitable Dorr or Akins classifier **11**, wherein the ore pulp is classified into a coarse fraction and a fine fraction. If brucite is present in the ore, the pulp will have a pH of about 8.5. The portion of the ore coarser than approximately 200 mesh is recycled through the ball mill **10**, while the minus 200 mesh fraction is discharged from the classifier **11** to a second

classifier 12, which is a hydro separator or a bowl classifier.

The classifier 12 divides the ore into two approximately equal portions, one portion being plus about 300 mesh and the other being minus about 300 mesh. The coarser, or plus 300 mesh, is discharged into a primary coarse brucite-magnesite froth flotation unit 13, it being understood that each froth flotation unit may include several cells. A suitable brucite-magnesite collector reagent is added to the pulp, preferably by stages during passage of the pulp through the primary flotation unit 13, with the result that the relatively coarse brucite-magnesite particles are collected and concentrated in the froth overflow of the flotation unit 13.

The underflow or tailings from the primary flotation cells 13 is discharged through a scavenger flotation unit 14 of similar design, where additional magnesium material collector reagent may be added. Most of the remaining magnesium material is concentrated in the froth overflow of the flotation cells 14 and are recycled for passage through the primary unit 13 in conventional manner. The underflow from the unit 14 is discharged from the system as tailing reject.

The overflow of the classifier 12 is passed through a silica froth flotation unit 16, limited quantities of a suitable silica collector reagent being added, preferably by stages, in this operation. The effect of this flotation stage is to concentrate in the froth overflow of the unit 16 the easily floated portions of the silica contaminants naturally present in the ore and which concentrate largely in the overflow of the classifier 12. These easily floated particles are usually silica minerals having a relatively flaky, rather than granular, form, and include the various talc and some chloritic minerals commonly encountered in the type of ore described. The underflow from the flotation unit 16 contains the magnesite values and is discharged into a fine magnesite froth flotation unit 17, where suitable magnesite collector reagents are added, preferably by stages.

This stage of the operation concentrates in the froth overflow the magnesium minerals in the fine portion of the ore. It will be found, however, that the brucite-magnesite collector reagent employed also collects and floats a small proportion of the calcium-bearing minerals naturally present. Calcium carbonate, or calcite, and dolomite are the predominant minerals in this category. Also since the pulp contains limited quantities of the silica collector reagent added in the unit 16, it will be found that a small quantity of silica is also incorporated in the froth concentrate. When the quantity of collector reagent added in the unit 16 is properly gauged and the units 16 and 17 operated in accordance with the teachings of this invention, it will be found that the calcium-bearing and silica-bearing minerals incorporated in the overflow from the unit 17 will be in such proportion as to permit adjustments and additions to form a suitable binder during future processing of the concentrate. The underflow from the unit 17 contains the unfloated silica-bearing minerals of the fine fraction of the ground ore, these usually being granular in nature, together with a certain amount of dolomite, calcite, and other impurities and are discharged as a reject tailing.

In Fig. 2, we have illustrated a somewhat modified process, wherein ore of the type described is fed in conventional manner to a ball mill 18 and discharged as an aqueous pulp into a classifier 19, similar to classifier 11. In this process, the classifier 19 may be operated in such manner as to recycle plus 325 mesh ore particles to the ball mill 18 for further grinding, while the underflow from the classifier 19 is discharged to a silica froth flotation unit 21, similar to the unit 16, previously described. A suitable silica collector reagent is added at this point, preferably by stages, and the easily floated portion of the silica contaminants present in the ore discharged as a froth reject from the flotation unit 21. The underflow from the unit 21 contains a substantial portion of the silica contaminants present, together with the brucite-magnesite ore and the calcium-bearing minerals. This underflow is discharged to a primary magnesium material froth flotation unit 22, where suitable collector reagents are added, preferably by stages. The magnesium values are thereby concentrated in the froth overflow from the flotation unit 22, together with a portion of the silica and calcium-bearing mineral contaminants present in the ore, this portion of the operation being somewhat similar to the operation of the froth flotation unit 17 previously described.

In this instance, however, the underflow from the flotation cell 22 is discharged into a classifier 23, which is operated in such manner that the coarse particles are discharged into a scavenger magnesium material froth flotation cell 24 for further treatment. The overflow from the classifier 23 consists predominantly of silicate minerals and other impurities suitable for discharge from the system as tailing reject.

Froth concentrate produced by the scavenger battery 24 contains most of the remaining brucite-magnesite ore particles, the underflow consists of undesirable impurities suitable for discharge from the process at tailings. The froth concentrate from the scavenger flotation unit 24 is recycled through the unit 22 in conventional manner.

We have discovered a new collector for silica, which is efficient and yet inexpensive in the process for recovering magnesium values from low grade brucite-magnesite ores. The collector is a combination of cresylic acid (usually derived from petroleum sources) and certain high molecular weight aliphatic ammonium acetates; the first of which is known as Aquaquad 12, which is a lauryl trimethyl ammonium acetate. Another collector reagent suitable for the combination is an amine and has a general chemical formula RNH_3 acetate, in which R represents the rosin acids derived from tall oil. Still another suitable reagent for use in our collector is commercially known as Armac S, which is a variable composition and consists of the various saturated and unsaturated fatty acids derived from soy beans and reacted with ammonia to form amines after which the amines are reacted with acetic acid to form the corresponding acetate.

The collector reagents suitable for use in the magnesium material flotation stages include a reagent known as Armour Neo Fat S142, essentially a refined tall oil product of somewhat variable composition. A typical analysis of this reagent indicates the following approximate composition:

Oleic acid	percent	46.0
Linoleic acid	do	39.0
Linolenic acid	do	3.0
Rosin acid	do	12.0
Mean molecular weight		295.2
Iodine value (Wijs)		130.0
Neutralization value		190.0

Other aliphatic carboxylic acids may be used, the choice being largely economical. The tall oil product is currently less expensive than other suitable fats or oils. If combined with a suitable frother, pure forms of the acids indicated above may be employed as magnesite collector reageants. The cost of such acids, however, is excessive and their use therefore is not feasible industrially.

Another tall oil derivative, commercially known as Triple Distilled Indusoil, may also be employed as a magnesium material flotation reagent. This composition, similar to Neo Fat S142, contains abietic, linoleic, and linolenic acids, usable directly or in saponified form. The characteristics of this reagent are substantially as follows:

Specific gravity at 60° F		0.96–0.98
Saybolt viscosity at 210°F		50–70
Flash point	° F	350–380
Fire point	° F	410–430

Acid number	180
Saponification number	181
Fatty acids approximately—percent	57
Rosin acids approximately—do	33

In addition to the above-described reagents, napthenic acids may also be employed as collector reagents for magnesium materials. In the magnesium material flotation, cresylic acid or a suitable substitute may be employed as a frother. The collector reagent for the silica may be varied to suit the particular batch of ore being processed. In general, the amine acetate may be used in amounts from 0.10 to 0.75 pound per ton of head ore, and the cresylic acid may also be varied from about 0.10 to 0.75 pound per ton of head ore. The magnesium material collector may be used in amounts of 0.30 to 5.0 pound per ton, determined by the requirements of the ore.

To illustrate the specific application of these reagents and the flow sheets illustrated, a series of tests were run utilizing the arrangement disclosed in Fig. 1. In these tests, Aquaquad 12 in the amount of about 0.18 pound per ton of head ore were fed to the flotation unit **16** of Fig. 1 in stages, together with about 0.16 pound per ton of cresylic acid. About 0.15 pound of Neo Fat S142 per ton of ore were fed to the flotation unit **17**. About 0.32 pound of Neo Fat S142 per ton of ore were fed to the flotation unit **13** in two equal stages, and 0.35 pound per ton of ore of the same reagent were added in two stages to the scavenger unit **14**. The results of the beneficiation were as follows:

Table I

	Percent weight [1]	Con.	Percent Acid Insoluble	Tail
Ball Mill Classifier Overflow	100.0		3.31	
Akins Classifier Coarse	53.6		2.89	
Akins Classifier Fines	46.4		3.42	
Coarse Concentrate	46.3	46.3	1.37	
Coarse Tails	7.3		11.52	7.3
Fines Froth Rejects	7.3		7.00	7.3
Fines Concentrate	30.0	30.0	1.73	
Fines Tails	9.1		6.94	9.1
Calculated Total Concentrate	76.3		1.51	
Calculated Total Tails	23.7		8.38	
		76.3		23.7

[1] By timed sample.

Similarly, the following amounts of reagent were added as indicated in the circuit shown in Fig. 2. About 0.40 pound and about 0.11 pound of RNH_3 acetate (where R represents rosin acids) and cresylic acids, respectively, per ton of head ore is added to the pulp in the scalper circuit **21**. The magnesium material in the primary circuit **22** is floated by adding about 0.25 pound of Triple Distilled Indusoil per ton of ore, and, also, by adding about 0.43 pound of the Triple Distilled Indusoil to the scavenger circuit **24**. The collector is added in three equal stages to the primary magnesium material circuit, and in two equal stages in the scavenger circuit. The results of the flotation are similar to the results shown in Table I, and are as follows:

Table II

	Weight Recovery	Percent Insoluble
Mg Concentrate	86.5	1.50
Scalper Froth	2.6	23.90
Hydro Separator Overflow	6.3	9.40
Scavenger Tail	.6	14.10
	100.0	

The Mg concentrate will yield a calcine of 3% SiO_2 or less since the insoluble matter is somewhat higher in value than pure SiO_2.

While specific examples have been utilized to fully describe the invention, there is no intent to limit the scope thereof to the precise details so set forth, except insofar as defined by the following claims.

We claim:

1. In the art of concentrating magnesium material from brucite-magnesite ores, the steps of reducing such an ore to a finely divided condition in which a substantial portion is in the size range of less than about 300 mesh, forming a free flowing pulp of said finely divided ore having a pH of about 8.5, subjecting said pulp to a froth flotation treatment in the presence of a silica collector reagent comprising crecylic acid and a high molecular weight aliphatic amine acetate for the froth flotation removal of a substantial portion of siliceous contaminants from the pulp, and subjecting the silica-depleted pulp to a second froth flotation treatment in the presence of a collector reagent for magnesium materials whereby to obtain a magnesium material concentrate containing about 1–2½% silica by weight.

2. In the art of concentrating magnesium material from brucite-magnesite ores, the steps of reducing the ore to a finely divided condition in which a substantial portion will be in the size range of less than about 300 mesh, forming a free flowing pulp of said finely divided ore having a pH of about 8.5, subjecting said pulp to a froth flotation treatment in the presence of a silica collector reagent comprising cresylic acid and a compound of the formula RNH_3Ac, where R is selected from lauryl trimethyl, and rosin acids for the removal of a substantial portion of siliceous contaminants of the pulp, and subjecting the silica-depleted pulp to a second froth flotation treatment in the presence of an aliphatic carboxylic acid as a collector reagent for magnesium material, whereby to obtain a magnesite concentrate containing 1–2½% silica by weight.

3. In the art of concentrating magnesium material from brucite-magnesite ores, the steps of reducing the ore to a finely divided condition in which a substantial portion will be in the size range of less than about 300 mesh, forming a free flowing pulp of said finely divided ore having a pH of about 8.5, subjecting said pulp to a froth flotation treatment in the presence of a silica collector reagent comprising cresylic acid and a compound of the formula RNH_3Ac, where R is selected from lauryl tri-methyl, and rosin acids for the removal of a substantial portion of siliceous contaminants of the pulp, and subjecting the silica-depleted pulp to a second froth flotation treatment in the presence of a tall oil as a collector reagent for magnesium material, whereby to obtain a magnesite concentrate containing 1–2½% silica by weight.

4. In the art of concentrating magnesium material from brucite-magnesite ores, the steps of reducing the ore to a finely divided condition in which a substantial portion will be in the size range of less than about 300 mesh, forming a free flowing pulp of said finely divided ore having a pH of about 8.5, subjecting said pulp to a froth flotation treatment in the presence of an effective amount of a silica collector reagent comprising cresylic acid and lauryl trimethyl ammonium acetate for the removal of a substantial portion of siliceous contaminants of the pulp, and subjecting the silica-depleted pulp to a second froth flotation treatment in the presence of a tall oil as a collector reagent for magnesium material, whereby to obtain a magnesite concentrate containing 1–2½% silica by weight.

5. In the art of concentrating magnesium materials from brucite-magnesite ores, the steps of grinding such an ore to provide a finely divided material, the major portion of which is less than about 200 mesh, forming a free flowing pulp of such material having a pH of about 8.5, classifying the pulp constituents into minus 300 mesh and plus 300 mesh portions, passing the plus 300 mesh pulp portion directly to a froth flotation treatment in the presence of a high molecular weight aliphatic acid compound

as a collector reagent for magnesium material, passing the minus 300 mesh pulp portion to a scalper flotation treatment in the presence of a collector reagent for siliceous contaminants comprising cresylic acid and $R \cdot NH_3Ac$, wherein R is a high molecular weight aliphatic radical, for removal of a substantial portion of the siliceous contaminants, and subjecting the silica-depleted pulp to a froth flotation treatment in the presence of a high molecular weight aliphatic acid as a collector reagent for magnesium material.

6. In the art of concentrating magnesium values from brucite-magnesite ores, the steps of forming a free flowing pulp of such an ore ground approximately to minus 325 mesh and having a pH of about 8.5, subjecting such pulp to a froth flotation treatment in the presence of a collector reagent for siliceous matter comprising cresylic acid and a high molecular weight aliphatic amine acetate for removal of a substantial portion of the siliceous impurities of the ore, passing the silica-depleted pulp to a froth flotation treatment in the presence of a magnesium material collector reagent for removal of a magnesium material concentrate, subjecting the residual pulp of said flotation stage to a classification treatment for rejection of a fine tailings, subjecting the underflow of said classification stage to a froth flotation in the presence of a magnesium material collector reagent, and returning the concentrate of said latter stage as part of the feed to said initial magnesium material flotation stage.

7. In the art of concentrating magnesium values from brucite-magnesium ores, the steps of forming a free flowing pulp of such an ore ground approximately to minus 325 mesh and having a pH of about 8.5, subjecting such pulp to a froth flotation treatment in the presence of a collector reagent for silica comprising cresylic acid and RNH_3 acetate, wherein R is a high molecular weight aliphatic radical, for removal of a substantial portion of the siliceous content of the ore, passing the silica-depleted pulp to a froth flotation treatment in the presence of a high molecular weight aliphatic acid composition as a magnesium material collector reagent for removal of a magnesium material concentrate, subjecting the residual pulp of said flotation stage to a classification treatment for rejection of a fine tailings, subjecting the underflow of said classification stage to froth flotation in the presence of said magnesium material collector reagent, and returning the concentrate of said latter stage as part of the feed to said first magnesium material flotation stage.

8. The process of treating brucite-magnesium ores for the recovery of magnesium values as a concentrate of the treatment, which comprises forming an aqueous pulp of such an ore having a major portion of its solid content in a size range of about —300 mesh, maintaining the circulating pulp of the treatment at a pH of about 8.5, and subjecting said pulp to a two-stage flotation separation with cresylic acid and a higher molecular weight, aliphatic amine introduced as a collector reagent in one stage of said concentration so as to collect siliceous content of the ore as a froth concentrate of such a separation, and introducing a collector for the magnesium materials as a collector in another stage of said concentration so as to concentrate the magnesium values of the ore as a concentrate of said other stage.

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