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MINERAL GRINDING AIDS

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ABSTRACT OF THE DISCLOSURE

Portland cement and minerals are ground with a grinding aid having the formula R—O—X wherein R is alkyl of 1 to 20 carbon atoms, phenyl or naphthyl and X is a metal above hydrogen in the electromotive series.

This invention relates to grinding minerals and more particularly to the use of an additive for improving the grinding efficiency and pack set characteristics of minerals.

In the processing of minerals, a grinding operation is generally employed either in the unprocessed or semi-processed state to reduce the particular mineral to relatively small particle size. It is desirable in this grinding step to have as efficient an operation as possible, that is, to reduce the particular mineral to the desired particle size at a relatively rapid rate.

A grinding aid is frequently employed in such a grinding operation to assist in the grinding of the minerals either by increasing the rate of production or by increasing the fineness of the particles at the same rate of production without having adverse effects or any of the properties of the ground product.

Cleavage of the particles during grinding of the minerals exposes fresh or nacent surfaces which have high energy due probably to the breaking of ionic bonds. The surface forces of the ground particles persist for some time after grinding and lead to compaction or pack set and/or poor fluidity if they are not reduced. Mineral particles when compacted by vibration, e.g., when transported in a hopper car often become semi-rigid and will not flow until considerable mechanical effort has been applied to break up the compaction. Therefore, it is also desirable that a material be employed to reduce the above-described adhesion of the particles.

The term "pack set" as used herein is intended to refer to the agglomeration or adhesion of particles by, for example, storing or transporting in bulk. Adhesion results from surface forces, the majority of which are believed to be created during the grinding of the minerals. "Pack set index" is a relative term which indicates numerically how prone a particular material is to start flowing after it is stored or transported in bulk. "Pack set index ratio" is the relative pack set index of the untreated sample compared to the treated sample. This ratio is used to permit comparison between different samples of the mineral.

The pack set index is determined in the following manner:

One hundred grams of the mineral are placed in a 250 milliliter Erhlenmeyer flask set on top of a variable vibrator. The flask containing the mineral is vibrated 15 seconds after which it is removed from the vibrator and set into a jig with the axis of the flask lying horizontally. The flask is then rotated around its axis until the mineral which is compacted in the bottom of the flask collapses. The flask is twisted at 180° angles at approximately 100 twists per minute. The number of 180° twists required for the mineral sample to collapse establishes the pack set index. Thus, the greater the energy required to break up the bed, the higher will be the pack set index.

A novel additive has now been found which will function as a grinding aid and a pack set inhibitor for minerals. The novel additive of the present invention is a compound of the formula

R—O—X

wherein R is an alkyl radical or an aryl radical and X is a metal above hydrogen in the electromotive series wherein hydrogen is in its standard state and has an electrode potential of 0.

The term "alkyl" as used herein is intended to refer to branched as well as straight chain alkyl radicals. Preferably the alkyl group contains 1 to 20 carbons, more preferably, 1 to 4 carbons. In a particularly preferred embodiment, it is a methyl group. The term "aryl" as used herein is intended to refer to a phenyl or naphthyl radical. It should be understood that the alkyl and aryl group may be substituted by, e.g., nitro, halogen, preferably chlorine; alkyl, preferably a 1 to 5 carbon group, more preferably methyl; aryl, hydroxyl, amino, and alkoxy, preferably a 1 to 5 carbon alkoxy group.

The metal represented by X is a metal above hydrogen in the electromotive series, preferably an alkali metal or an alkaline earth metal. Particularly preferred are sodium and calcium, as illustrative of additives within the scope of the present invention, mention may be made of the following:

Sodium methoxide
Sodium phenoxide
Calcium phenoxide
Calcium butoxide
Aluminum ethoxide
Magnesium methoxide
Aluminum methoxide
Aluminum isopropoxide
Aluminum-T-butoxide
Potassium O-methylphenoxide

The additives within the scope of the present invention are prepared by conventional means known to the art. For example, the metal hydroxide is reacted with a compound of the formula R—OH wherein R is aryl as defined above or the metal is reacted with a compound of the formula R—OH wherein R is alkyl as defined above.

The additive is interground with the mineral in the grinding mill to provide increased grinding efficiency as well as other advantageous results, e.g., inhibiting pack set of bulk stored materials. It has also been found that the novel additive of the present invention serves to provide fluidity to the ground minerals when they are being transported by conveying systems, particularly by pneumatic air systems.

The term "mineral" as used herein is intended to refer to naturally occurring inorganic minerals such as phosphate rock, partially processed minerals such as concentrated iron ore, and mixtures of minerals such as cement clinker or ceramics. As examples of such minerals that can be processed with the additive of the present invention, mention may be made of beryllium oxide, limestone, gypsum, clays and bauxite.

The grinding aids of the present invention are particularly preferred for use with cement, particularly portland cement.

Portland cement represents a class of hydraulic cements and is comprised essentially of two calcium silicates and a lesser amount of calcium aluminate. These cements are produced by heating an intimate mixture of finely divided calcareous material (limestone) and argillaceous material (clay) to form a clinker. The clinker is ground with the addition of about 5 percent gypsum, or some other form of calcium sulfate, to obtain the desired setting qualities in the finished cement. It is to the clinker

that the novel additive of this invention is preferably added to increase grinding efficiency and to inhibit subsequent pack set in the finished cement.

The additives of the present invention are employed in either dry or liquid form. For convenience, the additive is in water solution to permit accurate metering into the mill stream. In instances where the additive is not very soluble in water, it can be utilized in liquid form by emulsifying with a suitable wetting agent, for example, sodium dodecyl benzene sulfonate. The addition is accomplished either prior to the grinding or the additive is introduced into the grinding mill simultaneously with the mineral. If the additive is employed merely for the reduction of pack set or for fluidizing purposes, it is added at any convenient point in the processing.

The additive is employed effectively over a relatively wide range. The preferred range is about 0.001 to 1 percent based on the weight of the mineral, i.e., the weight of additive solids based on the weight of the mineral solids (herein referred to as solids on solids). In a particularly preferred embodiment, the amount of additive employed is about .005 to .04 percent. Higher levels are employed if grinding to a relatively high surface area and the amount of additive is limited solely by the desired surface area and the degree of fluidity desired.

In Table 1, the effectiveness of the additives of the present invention is recorded as well as data on pack set characteristics. The data was collected on Type 1 portland cement ground in a laboratory steel ball mill for 5,216 revolutions at 210° F.

TABLE 1

Additive	Amount of additive (percent solids on solids)	Blaine surface area, cm.²/g.	Percent increase over blank	Pack set index
Blank		3114		57.7
Sodium phenoxide	0.033	3373	8.30	34.7
Sodium methoxide	0.015	3259	4.66	
Sodium phenoxide	0.060	3473	11.52	14.0

In Table 2, the air-entertainment data and setting time data are reported. The percent air entrained was determined by the ASTM C185 Air Entrainment Test and the setting time by the ASTM C191 Vicat Setting Time Test. The water/cement ratio was 0.67.

TABLE 2

Additive	Amount of additive (percent solids on solids)	Percent air	Setting time, hours:min.
Blank		13.3	2:00
Sodium phenoxide	0.033	11.0	1:50
Sodium methoxide	0.015	10.8	1:40
Sodium phenoxide	0.060	10.8	1:25

Table 2 shows that the additive of the present invention does not result in excessive air-entertainment. The additive also advantageously reduces the setting time.

The compressive strength of cements ground by the novel additive of the present invention is reported in Table 3. The compressive strengths were determined by the ASTM C109 Compressive Strength Test. The water/cement ratio was 0.47.

TABLE 3

Additive	Amout of additive (percent solids on solids)	Compressive strength, p.s.i. 1 day	7 days
Blank		883	347
Sodium phenoxide	0.033	1103	3905
Sodium methoxide	0.015	1043	3530
Sodium phenoxide	0.060	1267	4403

The additive of the present invention is employed preferably as the sole grinding aid but it should be understood that it can also be employed with a mixture of one or more grinding aids or in admixture with other additives.

The additives of the present invention are also employed with satisfactory results in grinding other minerals in addition to cement, such as phosphate rock and iron ore.

Since changes may be made in the above products and processes without departing from the scope of the invention, it is intended that all matter contained in the above description shall be interpreted as illustrative and not in a limiting sense.

What is claimed is:

1. A novel composition comprising a solid material selected from the group consisting of minerals and portland cement and, intimately admixed therewith, from about 0.001 to 1 percent by weight based on the weight of said solid material of a compound of the formula

R—O—X

wherein R is selected from the group consisting of alkyl of 1–20 carbon atoms, phenyl and naphthyl radicals and X is a metal above hydrogen in the electromotive series.

2. The composition as defined in claim 1 wherein said compound is sodium phenoxide.

3. A composition as defined in claim 1 wherein said compound is sodium methoxide.

4. A composition as defined in claim 2 wherein sodium phenoxide is present at a level of 0.060 percent based on the weight of the solid material.

5. The composition as defined in claim 1 wherein said solid material is portland cement.

6. The method which comprises intergrinding a solid material selected from the group consisting of minerals and portland cement with from about 0.001 to 1 percent by weight based on the weight of said solid material of a compound of the formula

R—O—X

wherein R is selected from the group consisting of alkyl of 1–20 carbon atoms, phenyl and naphthyl radicals and X is a metal above hydrogen in the electromotive series.

7. The method as defined in claim 6 wherein said compound is added to the solid material prior to introducing the solid material into the grinding zone.

8. The method as defined in claim 6 wherein said compound is sodium phenoxide.

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