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Examiner

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AMIDE GRINDING AID

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ABSTRACT OF THE DISCLOSURE

Minerals and portland cement are intergrou[illegible] with a compound of the formula R—$CONH_2$ where[illegible] R is an alkyl or a phenyl group (e.g. acetamide) to enhance the efficiency of the grinding operation. The mine[illegible]l can be a naturally occurring inorganic mineral or a partially processed mineral.

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, either in the unprocessed or semiprocessed state, a grinding operation is generally necessary to reduce the particular mineral to a 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.

Cleavage of the particles during grinding of the minerals exposes fresh or nascent surfaces which have high energies due probably to the breaking of ionic bonds. The surface forces of the ground particles persists 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. On the other hand, undue reduction or the complete absence of surface forces is undesirable because the finely ground mineral becomes excessively fluid.

A grinding aid is a material which assists in grinding of 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 on any of the properties of the ground product.

The term "pack set" as used herein is intended to refer to the agglomeration or adhesion of particles by, e.g., storing or transporting in bulk. Adhesion results from surface forces, the majority of which are created during the grinding of the minerals. "Pack set index" is a relative term which numerically indicates 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.

Pack set index is determined in the following manner: 100 grams of the mineral is placed in a 250 milliliter Erlenmeyer flask set on top of a variable vibrator. The flask containing a mineral is vibrated 15 seconds after which time it is removed from the vibrator and fed into a jig with the axis of the flask lying horizontally. The flask is then rotated about its axis until the mineral which is compacted on the bottom of the flask collapses. The flask is twisted by turning 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 requirement

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 an amide, represented by the formula R—$CONH_2$ wherein R is an alkyl or phenyl group. In a preferred embodiment R is an alkyl group of 5 carbons or less. In a particularly preferred embodiment, the additive is acetamide. The term "phenyl" as used herein is intended to include substituted phenyl groups, e.g., alkyl substituted phenyl groups.

As examples of additional amides suitable for use within the scope of the present invention, mention may be made of the following: methylpropanamide, benzamide, butyramide, propanamide.

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 also 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 mixture of minerals such as cement clinker or ceramics. As examples of such minerals which 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 amterial (limestone) and argillaceous material (clay) to fusion to form a clinker. The clinker is ground with the addition of about 2% 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 a water solution to permit accurate metering into the mineral stream. In instances where the additive is not very soluble in water, it can be utilized in liquid form by emulsifying it with a suitable wetting agent, e.g., 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 reduction of pack set and fluidizing purposes, it is added at any convenient point in the processing.

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 cement additives other than grinding aids. A particularly preferred composition includes the additive of the present invention and triethanolamine.

The additive is employed effectively over a relatively wide range. The preferred range is about 0.001 to 1%, and more preferably about 0.005 to about 0.04% based on the weight of the mineral, i.e., weight of additive solids based on weight of mineral solids (herein referred to as solids on solids). In a particularly preferred embodiment about 0.025% of the additive based on the weight of the

mineral is employed. The higher levels are employed if grinding to a relatively high surface area, and the amount of additive used is limited only by the desired surface area and fluidity of the finished product.

In Table 1 the effectiveness of acetamide as a grinding aid is reported along with data on pack set characteristics. The data was collected on a Type I portland cement ground in a laboratory steel ball mill for 4400 revolutions at 220° F. Acetamide was employed as a 35% aqueous solution.

TABLE 1

	Blaine surface area cm.²/g.	Pack set index
Amount of additive (percent solid on solid):		
0	2,800	19.0
0.001	2,805	9.5
0.005	2,827	
0.000	2,878	6
0.015	2,882	4.5
0.020	3,018	4.5
0.030	3,000	3.5
0.040	3,041	1
0.050	3,048	2

In Table 2 ASTM C185 air entrainment data is reported on cements interground with acetamide. In all the compositions tested, the water/cement ratio was 0.68. The data shows that the use of acetamide as a grinding aid does not result in a greater degree of air entrainment than the blank.

Table 2

Amount of additive (percent solid on solid):	Percent air
0	9.9
0.001	9.6
0.005	9.2
0.010	8.7
0.015	9.3
0.020	9.2
0.030	9.6
0.040	10.1
0.050	9.3

In Table 3 the ASTM C109 compressive strength data is shown of mortars prepared from cements interground with acetamide. In all compositions the water-cement ratio was 0.48. The cements were ground for 4400 revolutions at 220° F.

TABLE 3

	Compressive strength p.s.i.		
	1-day	7-day	28-day
Amount of additive (percent solid on solid):			
0	1,042	3,358	4,575
0.005	925	3,400	4,838
0.015	1,065	3,475	4,625
0.020	1,005	3,700	4,775
0.030	1,158	3,725	4,713

Table 3 shows that the compressive strengths of cements containing the novel additive of the present invention are generally substantially higher than the blank.

Table 4 shows a comparison of setting times of cements which contain no grinding aid, a commercial grinding aid, and acetamide.

TABLE 4

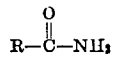
Additive	Amount of additive (percent solid on solid)	Setting Time, hours (ASTM C191)
Blank		3.12
Commercial grinding aid	0.04	3.08
Acetamide	0.04	3.00

Table 4 indicates that the novel additive of the present invention does not adversely affect the setting time of the cement.

While the additive of the present invention is described primarily in terms of grinding cement clinker, satisfactory results are obtained when the additive is utilized in grinding other minerals.

What is claimed is:

1. A composition consisting essentially of a solid material selected from the group consisting of naturally occurring inorganic minerals, partially processed minerals and portland cement, and intimately admixed therewith a compound of the formula

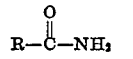


wherein R is phenyl, the amount of said compound present being sufficient to increase grinding efficiency when the composition is ground in a grinding mill.

2. A composition as defined in claim 1 wherein said compound is used at a level of about 0.001 to 1% by weight based on the weight of said solid material.

3. A composition as defined in claim 1 wherein said solid material is portland cement.

4. The method which comprises intergrinding a solid material selected from the group consisting of naturally occurring inorganic minerals, partially processed minerals and portland cement with a compound of the formula



wherein R is selected from the group consisting of alkyl of up to 5 carbon atoms and phenyl, the amount of said compound present being sufficient to increase grinding efficiency.

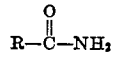
5. The method as defined in claim 4 wherein said compound is added to the solid material prior to introducing the solid material into the grinding zone.

6. The method as defined in claim 4 wherein said compound is present at a level of about 0.001 to 1% by weight based on the weight of said solid material.

7. The method as defined in claim 4 wherein said compound is acetamide.

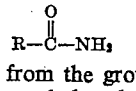
8. The method as defined in claim 4 wherein said solid material is portland cement.

9. A composition consisting essentially of portland cement and, intimately admixed therewith about 0.001 to 1% by weight, based on the weight of said portland cement, of a compound of the formula



wherein R is phenyl.

10. The method which comprises intergrinding portland cement with about 0.001 to 1% by weight, based on the weight of said portland cement, of a compound of the formula



wherein R is selected from the group consisting of alkyl of up to 5 carbon atoms and phenyl.

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