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PRODUCTION OF IMPROVED METAL OXIDE PIGMENT

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ABSTRACT OF THE DISCLOSURE

The enhancement of the pigmentary properties of inorganic pigments and particularly of pigmentary metal oxides, such as titanium dioxide, is discussed. The incorporation characteristics of such pigments is improved by fluid energy milling with steam, followed by a further fluid energy milling with a substantially liquid-free gas chemically inert to the pigment. The preparation of titanium dioxide is described.

BACKGROUND OF THE INVENTION

In the preparation of inorganic pigments and especially in the preparation of pigmentary metal oxides, such as titanium dioxide, various methods have been developed for enhancing such oxides' pigmentary properties for commercial applications, such as in paints. Included among such methods are calcination, hydroclassification, coating the surface of the pigment with hydrous metal oxides and/or organic coatings, other pigment surface treatments, and milling. A particularly important property for pigments utilized in surface coating compositions is their incorporation, i.e., wetting and dispersion, characteristics. Thus, the ability of a pigment to properly incorporate into a vehicle determines, in part, that pigment's commercial acceptability. For example, the degree to which a pigment is dispersed has a decided influence on many key paint properties. Typical of paint properties affected by the degree of pigment dispersion are surface appearance, texture, color development, floating, flooding, settling, and sagging. Of the aforementioned properties, the surface appearance provides a simple means for evaluating the degree of dispersion because the presence of significant pigment aggregates or flocculates within a surface coating give a rough apeparance to the coating. This is especially harmful in the preparation of surface compositions used to produce glossy, smooth coatings, e.g., enamel paints.

The intimate incorporation of a pigment into a paint vehicle by, for example, grinding, can be visualized as occurring in three steps, i.e., wetting, grinding, and dispersion, even though such steps overlap in any actual grind. Wetting refers to the displacement of gases, such as air, or other contaminants, such as water, that are adsorbed on the surface of the pigment particle with subsequent attachment of the wetting medium to the pigment surface. Grinding refers to the mechanical breakup and separation (deagglomeration) of the particle clusters to isolated effective primary particles. Such particles can comprise ultimate or primary particles, i.e., those which cannot be broken down further except by fracture, or a group of ultimate or primary particles that are closely held together and act as a unit working particle, i.e., an effective particle. Dispersion refers to the movement of the wetted ment dispersion and in the case of the Hegman gage, is limited to a minimum particle size of about 2 microns. The Hegman gage has a scale ranging from 0 to 8 and many paint specifications, especially high gloss paints, have a minimum fineness of grind specification of 7 (8 being the optimum). Thus, a relatively small increase in the Hegman gage reading at levels near the optimum, e.g., increases of from ¼ to ½, is representative of a significant improvement in the dispersion rating of a pigment.

SUMMARY OF THE INVENTION

This invention relates to the production of improved inorganic pigments, particularly pigmentary metal oxides, and more particularly pigmentary white metal oxides, such as titanium dioxide. In particular, this invention relates to the production of pigments of improved grit, fineness, texture characteristics, and tint efficiency which comprises subjecting the pigment to at least one fluid energy milling with a steam comprising gas or vapor and thereafter to at least one fluid energy milling with a substantially liquid-free gas chemically inert to the pigment.

In a further embodiment of the invention, pigmentary metal oxides, such as titanium dioxide, that have received a coating of at least one hydrous metal oxide are submitted to the two-stage sequential fluid energy milling heretofore described.

In still a further embodiment of the invention, blends of two or more inorganic pigments are subjected to the two-stage sequential fluid energy milling of the presently described process.

DESCRIPTION OF THE DRAWINGS

The attached figure is a plot of Hegman fineness gage readings as a function of mixing time, in minutes, in a paint dispersing apparatus for titanium dioxide pigment fluid energy milled in two stages wth various combinations of steam and nitrogen.

DETAILED DESCRIPTION

The ability of a pigment to incorporate (disperse) in a surface coating composition is an important property of the pigment and has a bearing on the pigment's commercial acceptability. It has now been discovered that this property can be improved by a particular fluid energy milling sequence. This discovery is exemplified hereinafter with pigmentary titanium dioxide but is not limited thereto.

Titanium dioxide pigment is produced commercially by at least two different manufacturing processes. One such process is known as the sulfate or acid process. In the sulfate process, a titaniferous ore, such as ilmenite, is digested in sulfuric acid to form a digest cake. The cake is dissolved in an equeous medium to form a sulfate solution which, after clarification and concentration, is hydrolyzed to precipitate an insoluble titanium dioxide hydrolysate. The hydrolysate is filtered, washed, and eventually calcined at temperatures ranging between 750° C. and 1000° C. Such calcination develops the rutile crystal structure of the titanium dioxide and dehydrates chemically combined water from the pigment.

Another and more recent process for preparing titanium dioxide pigment involves the vapor phase reaction, e.g., oxidation or hydrolysis, of a titanium compound, particularly a titanium tetrahalide at elevated