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PRODUCTION OF GAMMA FERRIC OXIDE

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1

This invention relates to the production of ferric oxide, and more particularly to a process for the production of γ -ferric oxide in the form of a finely-divided powder in which the particles are of substantially uniform size.

It is known that ferric oxide, Fe_2O_3 , exists in two forms differing in their crystal structure and in magnetic properties, namely the α form, which is paramagnetic, and the γ form, which is ferro-magnetic and possesses properties that make it suitable, in powdered form, for certain applications such as the formation of the magnetisable coating on recording tape. For this purpose in particular it is desirable that the magnetic powder used should consist of particles which, whether formed of single crystals or aggregates of crystals, are of substantially uniform size and of small diameter, preferably of the order of one micron or less.

It is the object of this invention to provide a method of producing γ -ferric oxide powder conforming to the above conditions.

We have found that γ -ferric oxide can be obtained in the form of a finely-divided powder of substantially uniform particle size by heating finely-divided iron oxalate or iron formate at such a temperature that substantially only ferrosferic oxide, Fe_2O_3 , is produced, and then subjecting the latter compound to controlled oxidation.

According to the present invention, therefore, a process for the production of γ -ferric oxide in the form of a finely-divided powder in which the particles are of substantially uniform size comprises heating finely-divided iron oxalate or iron formate consisting of particles of substantially uniform size, at a temperature below 600°C . in a substantially inert atmosphere until substantially complete conversion to ferrosferic oxide has taken place, and then subjecting the product to suitably controlled oxidation in air at a temperature below 350°C .

The process according to the invention is advantageous in that it is a dry process, solution or fusion of the particles at any stage in the process being avoided, so that the ferrosferic oxide formed in the first stage of the process and the γ -ferric oxide obtained as the final product are both pseudomorphs of the original crystals of iron oxalate or iron formate. Thus the size of

2

the crystals of the γ -ferric oxide produced is determined by the size of the crystals forming the starting material. The latter therefore should be of substantially uniform size and preferably should be less than one micron in diameter. Preferably also the iron oxalate or iron formate employed consists of particles, whether single crystals or crystal aggregates, of diameter of the order of one micron or less.

In carrying out the first stage of the process the iron oxalate or iron formate is heated at a temperature below 600°C ., a suitable temperature being approximately 450°C ., in a container in which an atmosphere substantially inert to ferrosferic oxide is maintained. Thus the atmosphere in the container consists of a gas or mixture of gases which will neither oxidise nor appreciably reduce the ferrosferic oxide formed. Suitable atmospheres are steam, carbon dioxide, or mixtures of one or both of these with nitrogen; a small proportion of hydrogen or carbon monoxide may also be present, provided that the concentration of these gases is not sufficiently great to cause any appreciable reduction of the ferrosferic oxide. A suitable gas mixture may be obtained by the combustion of town gas. The initial heating is continued until the iron oxalate or iron formate is substantially completely converted into pseudomorphous ferrosferic oxide, as indicated by the change of colour of the material from light green or yellow to black. The time required for the conversion is a number of hours, depending on the amount of material being treated and the manner in which it is disposed in the container; for example, a layer of material about an inch thick will require to be heated for about 6 to 7 hours at 450°C . When the heating is carried out under the conditions described, the product does not contain any appreciable amount of ferrous oxide, α -ferric oxide or free iron, although traces of one or more of these substances may be present.

In the second stage of the process, the crystals of ferrosferic oxide are converted, substantially completely, into pseudomorphous γ -ferric oxide by oxidation in air, the conditions being so controlled that the temperature of the oxide is maintained below 350°C ., preferably between 100°C . and 200°C ., throughout the reaction. The maintenance of a relatively low temperature, in

this stage as well as in the first stage of the process of the invention, is necessary to ensure that the ferric oxide produced is predominantly or wholly in the γ , and not in the α form.

One very convenient method of carrying out the oxidation of the ferrosferric oxide to γ -ferric oxide, which is the preferred method particularly when a dense product is required, consists in mixing with the cooled ferrosferric oxide sufficient water to damp it thoroughly, and then drying the damped oxide in air in an oven maintained at a temperature between 100° C. and 200° C., suitably at about 150° C. to 160° C. During the drying process the ferrosferric oxide is converted substantially completely to γ -ferric oxide. The water employed in this method has the effect of preventing an undesired rise in the temperature of the material during the oxidation process, and also appears to have a catalytic effect so that the oxidation takes place readily at a lower temperature (100° C. to 150° C.) than is the case when the ferrosferric oxide is kept dry.

Another method of carrying out the oxidation step consists in heating the dry ferrosferric oxide at a temperature below 350° C., preferably between 150° C. and 200° C., in a restricted supply of air, which may be provided by allowing a slow stream of air to flow over the oxide contained in a closed vessel in an oven or furnace maintained at the required temperature. In this case very careful control of the air supply is desirable in order to prevent a rapid rise in the temperature of the oxide, such as would result in the formation of α -ferric oxide.

One method of producing γ -ferric oxide according to the invention will now be described by way of example.

In this method, a quantity of iron oxalate, in the form of crystalline particles consisting of crystals of substantially uniform size and less than one micron in diameter, is heated in a furnace at 450° C., in an atmosphere produced by the combustion of town gas, until the colour of the material changes completely from yellow to black, indicating that conversion to ferrosferric oxide is substantially complete. The ferrosferric oxide is then cooled to room temperature, damped thoroughly with water and placed in an oven maintained at 160° C. When dry, the product is crushed and consists of substantially pure γ -ferric oxide in the form of crystalline particles consisting of crystals of diameter less than one micron and of a high degree of uniformity in size.

The γ -ferric oxide powder obtained by the process according to the invention may contain some unchanged ferrosferric oxide, but, provided that the conditions described above have been observed, the amount of ferrosferric oxide present is negligible, and other impurities, such as α -ferric oxide, are substantially absent.

The product of the oxidation step may be crushed to break down any cakes or loose aggregates of γ -ferric oxide crystals, so that the final product consists of very small crystalline particles of substantially uniform size, preferably less than one micron in diameter, and mainly formed of single crystals, the size of the crystals depending, as explained above, on the size of the crystals of which the iron oxalate or iron formate initially used is composed.

We claim:

1. A process for the production of substantially pure gamma ferric oxide in the form of a finely-

divided powder consisting of crystalline particles of substantially uniform size and diameter less than one micron, which comprises the steps of heating a finely-divided compound consisting of crystalline particles of substantially uniform size and of diameter less than one micron, which compound is a member of the group consisting of iron oxalate and iron formate, at a temperature below 600° C. in an atmosphere which is substantially non-reactive with ferrosferric oxide at temperatures below 600° C., said heating being continued until the said compound is substantially completely converted to ferrosferric oxide, cooling to about room temperature the ferrosferric oxide thus formed, thoroughly damping the cooled ferrosferric oxide throughout its mass with water, drying the damped ferrosferric oxide in air at a temperature between 100° C. and 200° C. to convert the ferrosferric oxide to substantially pure gamma ferric oxide, crushing the dried product, and recovering the gamma ferric oxide.

2. A process according to claim 1 wherein the first said step of heating said finely-divided iron compound is carried out at a temperature of approximately 450° C.

3. A process for the production of substantially pure gamma ferric oxide in the form of a powder consisting of crystalline particles of substantially uniform size and of diameter less than one micron, which comprises the steps of heating iron oxalate in the form of a yellow crystalline powder consisting of crystals of substantially uniform size and of diameter less than one micron, at a temperature of 450° C. in an inert atmosphere, said heating being continued until the said yellow crystalline powder has been converted completely into a black material constituting ferrosferric oxide, cooling the black material to about room temperature, thoroughly damping the black material throughout its mass with water, drying the damped material in air in an oven maintained at a temperature of 160° C. to convert the ferrosferric oxide to substantially pure gamma ferric oxide, crushing the dried material, and recovering the gamma ferric oxide.

4. A process for the production of gamma ferric oxide in the form of a finely-divided crystalline powder consisting of particles of substantially uniform size, which comprises the steps of heating a finely-divided iron compound selected from the group consisting of iron oxalate and iron formate consisting of particles of substantially uniform size and in the form of small crystals, of diameter not appreciably greater than one micron, which compound is capable of being converted to ferrosferric oxide on heating, the heating being carried out at a temperature below 600° C. in a non-oxidizing atmosphere until the said compound is substantially completely converted to ferrosferric oxide, cooling to about room temperature the ferrosferric oxide thus formed, thoroughly damping the cooled ferrosferric oxide throughout its mass with water, subjecting the damped ferrosferric oxide to oxidation in air at a temperature below 350° C. to convert the ferrosferric oxide to substantially pure gamma ferric oxide, the rate of oxidation being controlled so as to prevent an exothermic rise in the temperature of the oxide above 350° C., and recovering the gamma ferric oxide.

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