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PRODUCTION OF IRON COMPOUNDS

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This invention relates to the production of iron compounds adapted especially for use as pigments and for other purposes.

In Patents Nos. 1,327,061 and 1,368,748, Penniman and Zoph have described a process of producing iron compounds which has been used commercially for many years. Briefly, the process comprises the addition of a colloidal hydrate of iron, precipitated from a solution of an iron salt, to a water solution of an iron salt, and oxidation in the presence of metallic iron by the introduction of an oxidizing agent such as air with steam in quantity sufficient to maintain a preferred temperature of about 60° C. It has been the practice in the commercial use of the Penniman and Zoph process to avoid any temperature exceeding substantially that specified by the patentees. Higher temperatures were believed to be detrimental to the product and to involve such a waste of steam as to be commercially impracticable because the proportion of steam required increases rapidly with each degree of temperature rise obtained, particularly while a steady and copious current of air is passing through the solution.

It is the object of the present invention to provide an improved process of producing iron compounds and products thereof which have more desirable characteristics.

Other objects and advantages of the invention will be apparent as it is better understood by reference to the following specification, in which I have described the preferred procedure for the accomplishment of the intended purpose.

I have discovered that, contrary to expectation, the oxidation of solutions of iron compounds in the presence of metallic iron can be conducted more effectively at temperatures at or close to the boiling point and preferably at or about 90° to 95° C. Such relatively high temperatures, as compared with the temperatures commonly employed in the Penniman and Zoph process, require a supply of steam greatly exceeding that necessary to maintain a temperature of, for example, 60° C. However, the acceleration of the reaction and the improved characteristics of the product are such as to fully warrant the additional expenditure for the steam supply.

In carrying out the invention, it is necessary to modify the equipment used heretofore in the Penniman and Zoph process to afford a sufficient outlet for the much greater volume of steam and air which escapes from the solution during the operation. This, of course, involves losses of heat supplied in the form of steam. Additional losses occur because of the greater volume of air which

is introduced simultaneously for the purpose of oxidation and the corresponding accelerated discharge of the unused portion of the air. However, as indicated, I have found it more economical and otherwise desirable to conduct the operation at the higher temperatures indicated.

In addition to the improvement effected in the procedure, the products obtained are superior to those produced by the Penniman and Zoph process. The latter affords a product which is ochreous yellow, varying in depth of color from light (lemon) yellow, through a medium (orange) yellow, to a deep (dark orange) yellow. The variation in shade depends upon the length of time—number of days—during which the operation continues. As the result of the operation herein described, the products have lighter shades and are clearer yellows, less brownish in appearance in oil, stronger in undertone (when reduced with white) and the undertone is more greenish-yellow in contrast to the reddish characteristic of the product obtained at lower temperatures. Moreover, the red pigments resulting from the calcination of yellow pigments produced in accordance with the present invention are brighter and stronger. I am, therefore, able to produce a more acceptable product having numerous advantages in its commercial applications. The product is ferric oxide monohydrate having one molecule of chemically combined water ($\text{Fe}_2\text{O}_3 \cdot \text{H}_2\text{O}$) and usually has in addition approximately two percent (2%) of adsorbed water.

In carrying out the invention, I prepare a colloidal precipitate or nucleus of ferric hydrate, preferably by the addition of a suitable alkaline agent, such as caustic soda or potash, to a solution of a soluble ferric salt. Of the available alkali and alkaline earth precipitating agents which might be used, caustic soda produces pigments of the highest quality. A soluble ferrous salt may be precipitated similarly, and the precipitate may be oxidized by the introduction of air to the ferric form. The colloidal ferric hydrate is then mixed in the same or in a separate receptacle with a solution of an iron salt. Metallic iron is immersed in the solution, and steam is introduced at a rate sufficient to raise the temperature to, and to maintain it at or about, 90° to 95° C., while an oxidizing agent such as air is simultaneously introduced in order to effect the oxidation and precipitation of the desired pigment material. Oxidation and reduction by the metallic iron occur continuously in the solution in a cyclic manner, and the continued operation results in the formation of a pigment which in the earlier

stages is a light shade of yellow, changing through the continued operation of the process to darker shades of yellow. The process is continued until the desired shade is attained. Introduction of the colloidal ferric hydrate in the initial stage of the process prevents the formation of crystalline products which reduce the value and effectiveness of the material for pigment purposes.

The proportions employed in the practice of the invention may vary widely. It is desirable to employ the nuclear material in the proportion of about 10 to 50 pounds to each 1000 gallons of the solution. This, however, is merely an example of good commercial practice. The proportions are not critical, and can be increased or reduced very considerably without substantial modification of the results.

The invention and its advantages depend upon operation at a temperature at or about the boiling point of the solution. I have found that temperatures at or about 90° to 95° C. produce the best results, but the invention is not limited to the specific range indicated.

Various changes may be made in the procedure and particularly in details of operation without departing from the invention or sacrificing the advantages thereof.

I claim:

1. The method of preparing an hydrated oxide of iron suitable for pigments which comprises immersing metallic iron in a solution of an iron salt, heating and maintaining the temperature of the solution at approximately 90° to 95° C., and simultaneously supplying a gas containing free oxygen to the solution.

2. The method of preparing an hydrated oxide of iron suitable for pigments which comprises immersing metallic iron in a solution of an iron salt containing a precipitated iron hydrate, heating and maintaining the temperature of the solution at approximately 90° to 95° C., and simultaneously supplying a gas containing free oxygen to the solution.

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