PROCESS OF MAKING ACETATE DYESTUFF POWDERS

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This invention relates to a dyestuff composition in the form of a finely divided powder of improved dispersibility and to the method of producing same.

It is well known in the prior art to mill dyestuff compositions with shearing forces to reduce the particle size of the dyestuff. Such dyestuff compositions frequently contain surface active agents and other additives. Following the milling operation the composition, which is in the form of a thick magma, is then removed from the mill, usually after having been fluidized by the addition of water thereto. This composition is then dried in pans at elevated temperatures, after which the composition is subjected to an additional grinding operation to break up the agglomeration which occurs during drying. Such prior art methods are well exemplified by U. S. Patents Nos. 2,079,548 and 2,181,800 and Reissue Patent No. 21,402.

Such prior art methods involve several manipulative steps and the use of several different types of equipment which is disadvantageous for several reasons, among which are loss of time, increased expenses for labor and equipment, loss of materials and final product in the equipment due to incomplete transfer, increased hazards in handling, etc. The addition of fluid to the milled composition to facilitate its removal to the drying pans is a wasteful step since the fluid added must subsequently be removed in the drying operation. Further the agglomeration occurring during the drying in the vacuum pans is almost always not completely compensated for in the subsequent grinding operation.

It is an object of this invention to provide a dyestuff composition of improved dispersibility which is capable of dyeing fibrous materials such as acetate rayon and synthetic polymeric material such as nylon, Orlon, Acrylic, Dacron, Dynel, and the like, in level shades substantially free of spotting and the like.

Another object of this invention is to provide dye compositions in the form of highly dispersible soft fine powders which do not agglomerate to harder and larger particles difficult to disperse.

A further object of this invention is to provide an improved method for producing such dyestuff compositions. Other objects and advantages will appear as the description proceeds.

The aforesaid objects are obtained by the instant invention which comprises milling with shearing forces, a thick magma comprising the dyestuff, a solid dispersing agent and preferably a small amount of a neutral or alkaline acting salt to the desired particle size is obtained, and then drying the composition in the mill to a powder by application of a vacuum thereto without the addition of heat while continuing the milling action. The dyestuff powder composition is then ready for standardization by mixing in usual manner with diluents, bulking agents and the like.

It will be seen that the process of this invention is highly economical and efficient in eliminating various steps heretofore practiced. The dyestuff powder thus directly produced is in most cases in better condition than the powders heretofore produced since the agglomeration usually occurring during drying is avoided. The vacuum treatment in the mill in conjunction with the continued milling action and its concurrent finite generation of heat in situ due to friction is sufficient to dry the composition to a powder without the addition of heat in view of the fact that new surfaces are thereby constantly being exposed to optimum drying conditions.

In carrying out the milling operation, the dyestuff composition must be in the form of a stifle paste or thick magma, whereby the mixer exerts a shearing action on the dye particles to produce a uniform dispersion of particles of the desired size. Generally, the mixer is a Werniger-Pfeiffer or Day kneading machine or a Banbury mixer.

In order for the dyestuff composition to have the required consistency for milling with shearing forces, it should have a total solids content (before application of the vacuum treatment) of about 60 to 85%. Usually, it is sufficient to employ the dyestuff paste as it comes directly from the filter press, with a solids content of about 15 to 35%, and mix it with dry dispersing agent and neutral or alkaline acting salt. Where the initial consistency is too thick, it will be understood that water may be added to give the composition the consistency required for milling with shearing forces. Where the initial consistency of the composition is too thin due to too low a solids content, as in some of the examples hereinafter set forth, it will be understood that the excess moisture will be rapidly evaporated during the initial stages of the milling operation whereby the composition then being milled will have the required 60 to 85% solids content. The usual proportions by weight of dry ingredients in the dyestuff composition are about 40 to 60 parts of the dyestuff, 60 to 40 parts of dispersing agent and 0 to 10 parts of neutral or alkaline acting salt. The initial milling operation usually takes about 6 to 8 hours, after which vacuum is applied to the mill to remove the remaining water in the mix while continuing the milling action. The vacuum treatment normally takes about 2 to 4 hours, at the completion of which the composition is in the form of a dry powder ready for standardization.

As dispersing agents any of the usual solid dispersing agents may be employed as for example, sodium lignin sulfonate, oleoyl methyltaurine sodium salt, naphthalene sodium sulfonate, sodium alkylnaphthalene sulfonate, sodium oleyl sulfate, N,N-diethyl-N'-oleoyl-ethylene diamine hydrochloride, and the like.

As neutral or alkaline acting salts there may be employed sodium carbonate, sodium sulfate, sodium chloride, potassium pyrophosphate, sodium phosphite, potassium chloride, sodium pyrophosphate, magnesium sulfate, magnesium chloride, calcium sulfate, and the like. In general, such salts are alkali metal and alkaline earth metal salts of strong or weak acids. However, salts having an alkaline reaction are preferably employed. These are generally alkali metal salts of weak acids, for example, sodium carbonate. Such salts increase the dispersing
efficiency of the dispersing agent, prevent the formation of dispersed dyestuff compositions which are low melting and tend to sinter, regulate pH and act as diluents or bulking agents in the final composition.

While the process of this invention may be employed to treat any dyestuff in particulate form to thereby yield a finely divided dispersible powder, it is particularly advantageous when employed for treating those insoluble dispersible dyestuffs used in dyeing acetate rayon and the synthetic polymeric fibers such as nylon, Orlon, Acrilan, Dynel, Dacron and the like. The following dyestuffs which have been found particularly amenable to treatment in accordance with the instant invention are listed for illustrative purposes only.

The following specific examples, in which parts are by weight unless otherwise indicated, are given for illustrative purposes only.

**Example 1**

50 parts dye as a paste direct from the filter press (60-65% solid content) of the formula:

5 parts sodium lignin sulfonate and 2½ parts sodium car-
bonate. The mix is viscous milled for about 6 hours. A vacuum then is applied and milling continued until the powder is dry. It is removed from the mixer and standardized as usual.

**Example 2**

50 parts dye as a paste direct from the filter (70-75% solid content), of the formula:

\[
\text{O} \quad \text{NH}_2 \quad \text{O} \quad \text{CH}_3
\]

47½ parts oleoyl methyl taurine sodium salt and 2½ parts sodium pyrophosphate are viscous milled in a Werner and Pfleiderer mixer for 6 hours. A vacuum is then applied and milling continued until the powder is dry. It is removed from the mixer and standardized as usual.

**Example 3**

60 parts dye as a paste direct from the filter (35-40% solid content) of the formula:

\[
\text{NH}_2 \quad \text{N} \quad \text{N} \quad \text{OH}
\]

and 40 parts sodium alkylphthalenesulfonate are viscous milled in a Werner and Pfleiderer mixer for 8 hours. A vacuum is then applied and milling continued until the powder is dry. It is removed from the mixer and standardized as usual.

**Example 4**

50 parts dye as a paste direct from the filter (35-40% solid content) of the formula:

\[
\text{C}_6\text{H}_5\text{O} \quad \text{N} \quad \text{O} \quad \text{H} \quad \text{H}
\]

are mixed in a Werner and Pfleiderer mill with 47½ parts sodium lignin sulfonate and 2½ parts sodium carbonate for 6 hours. A vacuum is then applied and milling continued until the powder is dry, after which the powder is removed from the mixer and standardized.

**Example 5**

50 parts dye as a paste direct from the filter (45-50% solid content) of the formula:

\[
\text{NH}_2 \quad \text{N} \quad \text{N} \quad \text{OH}
\]

are mixed in a Werner and Pfleiderer mill with 47½ parts sodium lignin sulfonate and 2½ parts sodium carbonate. The charge is viscous milled for 6 hours. A vacuum is then applied and milling continued until the powder is dry. It is removed from the mixer and standardized as usual.

**Example 6**

50 parts dye as a paste direct from the filter (45-50% solid content) of the formula:

\[
\text{O} \quad \text{NH}_2 \quad \text{N} \quad \text{OH}
\]

are viscous milled in a Werner and Pfleiderer mill for 8 hours, with 50 parts sodium alkylphthalenesulfonate. A vacuum is then applied and milling continued until the powder is dry. It is removed from the mixer and standardized as usual.

**Example 2**

50 parts dye as a paste direct from the filter (70-75% solid content), of the formula:

\[
\text{O} \quad \text{NH}_2 \quad \text{O} \quad \text{CH}_3
\]

47½ parts oleoyl methyl taurine sodium salt and 2½ parts sodium pyrophosphate are viscous milled in a Werner and Pfleiderer mixer for 6 hours. A vacuum is then applied and milling continued until the powder is dry. It is removed from the mixer and standardized as usual.

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60 parts dye as a paste direct from the filter (35-40% solid content) of the formula:

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\text{NH}_2 \quad \text{N} \quad \text{N} \quad \text{OH}
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50 parts dye as a paste direct from the filter (35-40% solid content) of the formula:

\[
\text{C}_6\text{H}_5\text{O} \quad \text{N} \quad \text{O} \quad \text{H} \quad \text{H}
\]

are mixed in a Werner and Pfleiderer mill with 47½ parts sodium lignin sulfonate and 2½ parts sodium carbonate for 6 hours. A vacuum is then applied and milling continued until the powder is dry, after which the powder is removed from the mixer and standardized.

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\[
\text{NH}_2 \quad \text{N} \quad \text{N} \quad \text{OH}
\]

are mixed in a Werner and Pfleiderer mill with 47½ parts sodium lignin sulfonate and 2½ parts sodium carbonate. The charge is viscous milled for 6 hours. A vacuum is then applied and milling continued until the powder is dry. It is removed from the mixer and standardized as usual.

**Example 6**

50 parts dye as a paste direct from the filter (45-50% solid content) of the formula:

\[
\text{O} \quad \text{NH}_2 \quad \text{N} \quad \text{OH}
\]

are viscous milled in a Werner and Pfleiderer mill for 8 hours, with 50 parts sodium alkylphthalenesulfonate. A vacuum is then applied and milling continued until the powder is dry. It is removed from the mixer and standardized as usual.

Various modifications and variations of this invention will be obvious to a person skilled in the art and such variations and modifications are to be regarded as within the purview of this application and the spirit and scope of the appended claims.

We claim:

1. A process comprising milling with forces predominantly shearing in nature a composition having a total solids content of about 60 to 85 percent comprising an insoluble, dispersible, acetate type dyestuff, a solid dispersing agent, and water in an amount sufficient to produce a thick magma, and then drying the milled composition in the mill by subjecting it to a vacuum treatment without the addition of heat while continuing the milling action.

2. A process comprising milling with forces predominantly shearing in nature a composition having a total solids content of about 60 to 85 percent comprising an insoluble, dispersible, acetate type dyestuff, a solid dispersing agent, a compound of the group consisting of the inorganic salts of alkali metals and alkaline earth metals, and water in an amount sufficient to produce a thick magma, and then drying the milled composition in the mill by subjecting it to a vacuum treatment without the addition of heat while continuing the milling action.

3. The process of claim 2 in which the said compound is an alkaline acting salt.

4. The process of claim 3 in which said compound is sodium carbonate.

5. A process comprising milling with forces predominantly shearing in nature a composition having a total solids content of about 60 to 85 percent and comprising, by weight, 50 parts of a dyestuff of the formula

\[
\text{O} \quad \text{NH}_2 \quad \text{O} \quad \text{CH}_3
\]

47½ parts of sodium lignin sulfonate and 2½ parts of sodium carbonate for about 6 hours and then drying the milled composition in the mill by subjecting it to a vacuum treatment without the addition of heat while continuing the milling action.

6. A process comprising milling with forces predominantly shearing in nature a composition having a total solids content of about 60 to 85 percent and comprising, by weight, 50 parts of a dyestuff of the formula

\[
\text{O} \quad \text{NH}_2 \quad \text{O} \quad \text{CH}_3
\]

47½ parts of oleoyl methyl taurine sodium salt and 2½ parts of sodium pyrophosphate for about 6 hours, and then drying the milled composition in the mill by subjecting it to a vacuum treatment without the addition of heat while continuing the milling action.

7. A process comprising milling with forces predominantly shearing in nature, a composition having a total solids content of about 60 to 85 percent and comprising, by weight, 60 parts of a dyestuff of the formula

\[
\text{O} \quad \text{NH}_2 \quad \text{O} \quad \text{CH}_3
\]

and 40 parts of sodium alkylphthalenesulfonate for about 8 hours, and then drying the milled composition in the mill by subjecting it to a vacuum treatment without the addition of heat while continuing the milling action.

8. A process comprising milling with forces predominantly shearing in nature a composition having a total solids content of about 60 to 85 percent and comprising, by weight, 70 parts of a dyestuff of the formula

\[
\text{O} \quad \text{NH}_2 \quad \text{O} \quad \text{CH}_3
\]
solids content of about 60 to 85\% and comprising, by weight, 50 parts of a dyestuff paste of the formula

\[
\begin{align*}
\text{O} & \text{N} \\
\text{NO}_2 & \text{NH} \\
\text{OH} & \text{C}_6\text{H}_4\text{O} \\
\text{H} & \text{H} \\
\text{H} & \text{H} \\
\end{align*}
\]

47\% parts of sodium lignin sulfonate and 2\% parts of sodium carbonate for about 6 hours and then drying the milled composition in the mill by subjecting it to a vacuum treatment without the addition of heat while continuing the milling action.

9. A process comprising milling with forces predominantly shearing in nature a composition having a total solids content of about 60 to 85\% and comprising, by weight, 50 parts of a dyestuff of the formula

\[
\begin{align*}
\text{ONO} & \text{N} \\
\text{H} & \text{H} \\
\text{H} & \text{H} \\
\text{H} & \text{H} \\
\end{align*}
\]

47\% parts of sodium lignin sulfonate and 2\% parts of sodium carbonate for about 6 hours and then drying the milled composition in the mill by subjecting it to a vacuum treatment without the addition of heat while continuing the milling action.

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