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PRODUCTION OF DISPERSIONS

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This invention relates to the production of dispersions and has particular reference to the production of dispersions of the type wherein a bitumen-pitch type base of a solid or semi-solid consistency, or even a more fluid product of this general type, is dispersed in an aqueous vehicle, the latter constituting the external phase of the dispersion, while the binder comprises the internal or dispersed phase thereof.

This application is in part a continuation of applications previously filed by me, 32,839, filed May 25th, 1925, and 64,698, filed October 24th, 1925.

Manifestly it is desirable, and in many instances essential, that a dispersion of the character here referred to should, when spread or otherwise applied in the form of a thin film, and the aqueous content thereof permitted to evaporate, result in the formation of a highly water-resistant and impermeable film.

Accordingly, one of the objects of the present invention resides in the treatment of the dispersion with materials which function to modify the character of the dried film to the extent of increasing its impermeability and improving its power to resist water action as soon as it has dried.

Another object of the present invention is to induce conditions favorable to, and which do facilitate, dispersion of the selected base in either larger quantities or in a finer state of subdivision, or both, by the dispersing agent.

It has been found in accordance with the present invention that it is possible to facilitate the dispersing action as well as modify the character of the dried film by carrying on the dispersion in the presence, or with the addition, of certain substances, hereinafter to be enumerated. These substances, or modifying agents, as they may be termed, which after long experimentation have shown themselves capable of producing the effects mentioned, may be generally described as acidic, and may comprise either acidic powders, acids, or acid salts, or combinations of these, which act with or upon the dispersive medium or upon the dispersion without, however, breaking the same and causing particle agglomeration.

Included within the class of materials above indicated by the term acidic, are mixtures of basic substances with substances of acid character, in quantities so proportioned that the resultant combination is acidic. A modifying agent thus produced by the admixture of basic with acidic substances has been found to be suitable, for the purposes indicated, where employed with the dispersive agent in proportions sufficient to maintain the modified system well within the acid range.

The bases which may be dispersed in accordance with the present invention may include asphalt, either natural or artificial, coal tar pitch, vegetable or animal pitches, paraffine, resin or the like, or suitable admixtures of these bases as desired. Rubber prepared for dispersion mechanically or with plasticizing agents may be dispersed and/or treated in the manner described.

As the dispersive media there is preferably selected a clay-like material such as bentonite, although it should be understood that other materials, such as clay, colloidal oxides, hydroxides, powdered metallic silicates, casein and the like, or suitable combinations of these materials, may be employed in a similar capacity as the dispersive media.

The dispersion is carried out preferably in a continuous manner; the base, liquefied by heat, if necessary, being fed into any suitable emulsifying instrumentality simultaneously with the introduction of the aqueous dispersive media. The entire mass undergoing dispersion is thoroughly and continuously agitated, while being circulated within the emulsifying apparatus under controlled temperature conditions preferably approximating the melting point of the selected base.

In practicing the present invention the procedure may be directed either toward the modification of the dispersive media either prior to or during the dispersing operation whereby to facilitate the dispersion, or, the principles hereof may be utilized to treat a finished dispersion, for the purpose of modifying the character of the dried film, or, if desired, both the aforesaid purposes may be

effectually accomplished in accordance with the intention, as hereinafter to be described.

For the purpose of facilitating the dispersing operation, so that the base may be dispersed in larger quantities or in a finer state of subdivision, or both, without regard to the character of the resultant film, there may be first prepared an aqueous mixture of the modified dispersive media. This may be accomplished by adding a modifying agent to an aqueous suspension of the dispersing media, and agitating the entire mass. If the suspension or mixture exhibits a tendency for its pH value to increase, more of the modifying agent may be added and the procedure continued until the mixture has attained a substantially constant pH value of say, 3.5, more or less. The modified dispersing agent thus formed, is then employed to disperse the selected base.

As an illustration, an aqueous suspension of bentonite or similar clay-like material, may be modified with an acidic substance, oxalic acid, the latter being added in sufficient quantities to bring the pH of the dispersive media down to a constant value of say, 3.5. The thus modified dispersing agent may then be employed to disperse asphalt, pitch, or a similar material, which tends to increase the pH value of the dispersive media.

Illustrative of acidic salts which have been found satisfactory and efficient as modifying agents for the purpose indicated, there may be named aluminum sulphate, sodium acid phosphate, and calcium acid phosphate.

Furthermore, in lieu of the acidic salts above enumerated, and especially in those cases where the dispersive media is highly basic in its nature, and would require excessive amounts of an acid salt in order to be brought down to within the acid range aforementioned, resort may be had to acids. Certain of these materials, being more highly acid than the salts, are capable of use in much smaller quantities to lower the pH of the dispersing agent and avoid the presence of an excessive quantity of soluble salt in the final film. This is important for the reason that an excess of such soluble salts may harmfully affect the resultant film and destroy its water-resistant capacity.

Illustrative of acids which may be employed with satisfactory results, as modifying agents for the dispersive media, there may be named sulphuric, oxalic, phosphoric, chromic, hydrochloric, and benzoic. For most purposes, however, acid salts are quite satisfactory and are used in preference to the acids.

Instead of using solely acidic substances as the modifying agent, mixtures of such substances with basic materials may also be adopted in carrying out the principles of the present invention. When resorting to such mixtures or combinations, the selected acidic substance may be first pre-mixed with the selected basic substance. Or the dispersive means may be treated with one or the other substance in succession. Thereafter the resultant product is employed to modify the selected dispersing media, and the modified system is then utilized in carrying out the dispersion of the selected base.

When using combinations of acidic and basic substances, the former may preferably take the form of an acid for the reasons above stated. For example, a mixture of magnesium oxide and phosphoric acid, or a similar mixture may be used with oxalic acid to advantage. In place of either of the aforenamed, mixtures of chromic oxide with acetic acid, or lime with chromic acid, may be used, and in fact numerous other mixtures will readily suggest themselves as capable of use in this connection.

While the materials which have been above described as modifying agents may, in addition to facilitating the dispersing action, exert a beneficial effect on the film, in that the water-resistance of the latter is somewhat accelerated in point of time, nevertheless, it is requisite to the use of such dispersions in certain arts that the dried film be characterized by an ability to more rapidly attain its maximum powers of water resistance. The second principle as embodied in the present invention, therefore, involves the treatment of the final dispersion whereby to accelerate to a maximum degree the water-resisting powers of its dried film. It has been found, in carrying out this principle of the present invention, that when a dispersion is treated in such manner as to bring the pH value thereof down to, say 4.0, more or less, its water-free film will more rapidly attain maximum water-resistant properties than does a film produced from dispersions not subjected to the aforesaid modification.

The treatment of the final dispersion with a view to modifying the properties of its water-free film may, if desired, follow the production of the dispersion with a modified dispersing agent, as heretofore described, or such treatment may be utilized to improve the character of the film of a dispersion produced with an unmodified dispersing agent.

In those cases where modifying agents are resorted to in the production of the dispersion, the same materials as those which comprised the modifying agent may also be used to modify the character of the water-free film, the required additional amount of the reagent being added after the dispersion is complete. Inasmuch as it may occur to some, that the additional quantity of the reagent may be incorporated with the dispersive media at the same time that the latter is subjected to the modifying action heretofore described, or during dispersion, it may be stated that such procedure in some cases, is inadvis-

able and is to be avoided for the reason that the presence of the additional amount of the reagent has a tendency to bring the pH of the system too low for proper use and thereby render efficient dispersion more difficult of attainment, and hence destroys the very effect which is sought to be induced by the modifying agent when used in proper quantities. Thus, while the same material may serve the two-fold purpose of facilitating the dispersion and improving the character of the film, the better operation and more successful results are realized when part of the reagent is added to the dispersive media, the dispersion carried out with the thus modified dispersive material, and the remainder of the reagent added to the completed dispersion.

Where the dispersion has been formed by means of a dispersing media modified with a salt which is weakly acid, the final dispersion is preferably subjected to treatment with a substance more pronounced in its acid character than the said acid salt. Thus, for example, using bentonite, modified as above described with 10% of its weight (on the dry basis) of potassium dichromate, and dispersing asphalt with the modified material, the final dispersion may contain asphalt in quantities as high as 50–75 times the weight of bentonite. This dispersion may then be modified to substantially improve its water-free film by adding to the dispersion an acid, such as phosphoric, instead of using further quantities of the potassium dichromate. Whereas, for example, when using 120 penetration Mexican asphalt with a mixture of one-half bentonite and one-half New Jersey pottery clay, 0.3% of phosphoric acid, or 0.4% of chromic acid, or 0.1% hydrochloric acid, on the basis of the final dispersion, is sufficient to bring down the pH of this system to a point within the acid range where the water-free film of the product rapidly achieves maximum water-resisting properties, i. e. pH, 1.5 to 4.0, the full effect cannot be attained by the use of potassium dichromate, since the acidity is not sufficient and the introduction of increased quantities of this soluble salt may diminish rather than improve the final film.

In those cases where dispersions are produced with unmodified dispersing agents, the procedure obviously involves first, the dispersion of the selected base with such dispersing agent as may be found suitable and then the subjection of the finished dispersion to the action of the modifying agent. The latter may take the form of an acid, an acid salt, or a mixture of basic and acidic substances, as hereinabove enumerated, the selection being based upon the ability of the respective agents to materially lower the pH of the particular dispersion and the effect of the modifying agent in altering the characteristics of the dispersion itself. That is, specific ions affect in a different manner the suspendability of the dispersed phase, the water content of the emulsion, its fluidity and body. The selection of the particular modifying agent is therefore made in view of these other effects which are entailed in the emulsion product as well as its water-free film.

As another illustration of this type of operation, but one which is peculiarly characterized in the initial treatment in that the binder is treated with an alkali rather than with acid, coal tar may be dispersed with bentonite, provided the pH value of the dispersion means is maintained well within the alkaline range. Though bentonite is itself alkaline, having a pH value of about 9.0 the coal tar, being generally of an acid character continually tends to bring the pH of the mass too low and hence it becomes necessary in such case to add an alkali, such as sodium or ammonium carbonate, or tri sodium phosphate, to maintain the pH well on the alkaline side, under which conditions the dispersion of the coal tar may be readily effected. The addition of the alkali is, however, not always necessary, and where the coal tar is not of such highly acid character the dispersion thereof may be carried on without the use of the alkali. The desired amount of coal tar having been dispersed, the system may then be treated either with the acid salts, acids, or mixtures of basic and acidic substances, whereby to modify the character of film in accordance with the principles heretofore described.

The final product after treatment in accordance with either of the methods herein described, may be of a thick viscous consistency. In such state the product of the present invention finds very little use, and hence, in order to be suitable for application in various arts, it is generally necessary to convert the material to a more fluid consistency. Inasmuch as the product is very stable, and is further characterized by the fact that the aqueous constituent is in the external phase, its resultant miscibility with water affords opportunity for thinning the same to any desired degree merely by the addition of water in quantities sufficient to bring the consistency of the product to the desired point.

Instead of thinning the final dispersion solely by the addition of water, it may be subjected to a rapid beating or shipping action, as described in my Patent No. 1,616,904, dated February 8th, 1927. This method of treatment is especially useful in connection with the present invention, inasmuch as the use of electrolytes, such as the acidic substances herein described, tends to cause the particles of the dispersion to flocculate and thicken, so that even larger quantities of water are required to bring the dispersion to the proper consistency than is the case where such substances are not present. The beating or whipping action accordingly

affords a means for decreasing or eliminating the use of such additional quantities of water, as well as for reducing the quantity ordinarily required where the dispersion is not subjected to the beating operation.

By the expression "bitumen-pitch type base" as used in the claims, I intend to embrace those materials hereinbefore specifically listed.

It is to be understood that I do not wish to limit myself to the specific materials described herein, but intend to embrace the general range of materials having similar characteristics.

I claim as my invention:

1. In the production of dispersions, steps which comprise modifying the dispersive media by combining acidic substances therewith, dispersing a bitumen pitch-type base with the modified dispersive media and thereafter incorporating in the dispersion acidic material in quantities sufficient to substantially accelerate the water resisting powers of its dried film.

2. In the production of dispersions, steps which comprise modifying the dispersive media by combining acidic substances therewith, dispersing a bitumen pitch-type base with the modified dispersive media and thereafter incorporating in the dispersion acidic material in quantities sufficient to lower its pH value to less than 4.0.

3. In the production of dispersions, steps which comprise modifying the dispersive media by combining acidic substances therewith, dispersing a bitumen pitch-type material with the modified dispersive media, and thereafter incorporating in the dispersion material of more pronounced acid character than said first named acidic substance.

4. A process of producing a dispersion in which the dried film is characterized by increased ability to resist water action, which comprises dispersing a bitumen pitch-type material with dispersive media including a dispersing agent and an electrolyte and after dispersion, adding acidic substances to bring the pH value of the dispersion to 4.0 or less.

5. A process of producing a dispersion in which the dried film is characterized by increased ability to resist water action, which comprises dispersing a bitumen pitch-type material with modified dispersive media in the alkaline range, and after dispersion adding acidic substances to bring the pH value of the dispersion to 4.0 or less.

6. In the production of aqueous dispersions of bitumen-pitch type bases, the step which comprises adding acidic material to the dispersion in quantities less than 2% by weight of the dispersion, said acidic material being capable of reducing the pH value of the dispersion to less than 4.0 when used in such quantities.

7. A dispersion of a bitumen-pitch type base in water, said dispersion containing acidic material capable of imparting to the dispersion a pH value less than 4.0 when used in quantities less than 2% by weight of the dispersion.

8. A dispersion of a bitumen-pitch type base in water containing less than 2% by weight thereof, of acidic material, adapted to impart a pH value less than 4.0 to said dispersion.

9. A dispersion of bitumen-pitch type base in water and containing a mineral dispersing agent, said dispersion having a pH value less than 4.0 and containing less than 2% by weight of acidic material.

10. A dispersion of bitumen-pitch type base in water and containing a clay-like dispersing agent, said dispersion having a pH value less than 4.0 and containing less than 2% by weight of acidic material.

11. A dispersion of a bitumen-pitch type base in water, said dispersion having a mineral powder dispersing agent and containing water soluble acidic material in the continuous phase thereof in quantities sufficient to impart a pH value less than 4.0 to the dispersion.

12. A dispersion of a bitumen-pitch type base in water, said dispersion containing less than 1% by weight thereof of a mineral acid and having a pH value less than 4.0.

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