

## UNITED STATES PATENT OFFICE

IVAN GUBELMANN, HENRY J. WEILAND, AND CLYDE O. HENKE, OF SOUTH MILWAUKEE, WISCONSIN, ASSIGNORS, BY MESNE ASSIGNMENTS, TO E. I. DU PONT DE NEMOURS & COMPANY, A CORPORATION OF DELAWARE

## TREATING BATH AND PROCESS OF TREATING TEXTILE FIBERS

No Drawing.

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This invention relates to a process for increasing the wetting out and penetrating capacity of treating liquids employed in the textile and leather industries.

It is an object of this invention to provide an improved method of increasing the efficiency of aqueous baths for treating textile fibers in conjunction with a variety of treating operations of which the following are typical: dyeing of fabrics or fibrous materials, both animal and vegetable; washing and scouring of fibers preliminary to the dyeing operation; dyeing of leather; carbonizing, fulling and softening of wool; sizing, dressing and the like. This invention is applicable to the wet treatment of textile fibers in substantially all cases where it is advantageous that a quick wetting and penetrating effect of the treating liquid be obtained.

Other and further important objects of this invention will become apparent from the following description and appended claims.

We have found that sulfonated products which may be derived from sulfonation of the products obtainable by the pyrogenic decomposition of rosin, abietic acid or abietyl chloride have in aqueous solution remarkable wetting and penetrating properties.

Due to variations in the source of the abietic acid containing material (rosin), the resulting product produced therefrom will vary slightly in its composition, but we believe that, independent of the source of the abietic acid containing material, the product obtained will contain a large proportion of a specific hydrocarbon, abietene or abietine. A number of these various products are described by Ruzicka, Helvetica Chimica Acta, volume 6, pages 838 to 840. We have sulfonated many of these products and find that they are all useful for our purpose. However, exceptionally outstanding in this respect is the sulfonation product of a rosin-decomposition product obtainable according to U. S. Patent No. 1,853,353 issued April 12, 1932. This procedure, briefly, consists of refluxing rosin in the presence of iron and at a temperature of about 350 to 375° C. until its acidity has been practically destroyed, and

then distilling to recover a fraction boiling below 450° C. The hydrocarbon product thus obtained is characterized by a specific gravity of about 0.99 (at 20° C.) and its sulfonation product is characterized by exceptionally high wetting powers, and by yielding a sodium salt which is non-hygroscopic.

The methods of preparing these sulfonic acids are more specifically described and claimed in U. S. Patents Nos. 1,853,352 and 1,853,353. Briefly, these methods consist of treating the above defined hydrocarbons of the abietene family with strong sulfonating agents, such as, sulfuric acid monohydrate, at temperatures between about 0 to 50° C.

We have also found that the sulfonation products of abietane, which are more specifically described and claimed in U. S. Patent No. 1,853,348 can be used in the same way as the sulfonation products of abietene. We believe the general ring structure of abietene and abietane to be the same, the specific difference being, that part of the unsaturation linkages of abietene are absent in abietane.

The sulfonic acids of the above products may be transformed into water soluble salts such as potassium, sodium and ammonium and in any of these forms may be used as assistants in acid, neutral or alkaline treating baths.

These new products even in dilute aqueous solutions exhibit remarkable wetting and penetrating effects upon fibrous materials. Our preferred concentrations for use are from 0.1% up to about 5% of the treating bath, the latter concentration being sufficiently high for most purposes without waste of material.

The following examples, given in parts by weight, serve to illustrate the nature of our invention.

*Example 1*

10 parts of cotton yarn are added to a solution prepared from 70 parts of water and 0.7 parts of abietene sulfonic acid sodium salt. The cotton is soaked for some time at a temperature of about 40° C. It is then removed from the bath, rinsed and dried. The cotton yarn is then dyed with Anthrene



Violet BNX as follows. A dye bath is made up by vatting 1 part of Anthrene Violet BNX double paste (Color Index 1163) in 25 parts of water and 0.6 parts of a 10% solution of NaOH and 0.6 parts of sodium hydrosulfite at 60° C. The dye vat is then diluted to a total volume of 200 parts with water at 60° C. and then 20 parts of a 10% solution of Glauber's salt are added. The cotton yarn is now immersed. The dyeing operation exhausts the dye bath. Upon oxidation of the skein in air, followed by rinsing and soaping, clear, even, deep shades are obtained. In case a similar dyeing is made upon untreated cotton the shades obtained are uneven and weak. As an alternative method in the dyeing process, the abietene sulfonic acid sodium salt may be incorporated directly in the dye vat, thus eliminating the extra step of treating the fiber outside the dye bath. This latter method gives practically the same results as the first method.

#### Example 2

10 parts of woolen yarn are placed in a solution containing 70 parts of water and 0.7 parts of abietene sulfonic acid. The woolen yarn is wetted in a few seconds. This procedure may be incorporated as a step in any process involving the wetting of wool, such as cleansing, degreasing, dyeing, fulling, carbonizing or the like.

#### Example 3

The procedure is the same as in Example 1 except that abietane sulfonic acid is used instead of abietene sulfonic acid. The results are substantially the same.

#### Example 4

2.5 parts of a dyestuff mixture containing 20% of 1-amino-2-methyl-anthraquinone, 75% sugar and 5% abietene-sulfonic acid sodium salt are added to 3000 parts of water at 60° C. The dyestuff is immediately dispersed. 100 parts of celanese silk are then immersed in the dyebath and dyed in the usual manner. The silk is dyed in an even and clear orange shade.

We are aware that numerous details of the process may be varied through a wide range without departing from the principles of this invention, and we, therefore, do not purpose limiting the patent granted hereon otherwise than necessitated by the prior art.

We claim as our invention:

1. The process of treating fibrous material, which comprises subjecting fibrous material to an aqueous treating bath containing water soluble sulfonation products of a member of the group consisting of abietene, abietine and abietane.

2. The process of treating fibrous material, which comprises subjecting fibrous material

to an aqueous treating bath containing water soluble sulfonation products of abietene.

3. The process of treating fibrous material, which comprises subjecting fibrous material to an aqueous treating bath containing an alkali metal salt of abietene sulfonic acid.

4. The process of treating fibrous material which comprises subjecting such material to an aqueous treating bath containing a water soluble sulfonation derivative of a rosin decomposition product obtainable by refluxing rosin in the presence of iron until its acidity has been substantially reduced and distilling to recover a fraction boiling below 450° C.

5. As a new composition of matter, an aqueous treating bath for fibrous material containing a sulfonation product of a member of the group consisting of abietene, abietine and abietane.

6. As a new composition of matter an aqueous treating bath for fibrous material containing a sulfonation product of abietene.

7. As a new composition of matter, an aqueous treating bath for fibrous material containing a water soluble sulfonation derivative of a rosin decomposition product obtainable by refluxing rosin in the presence of iron until its acidity has been substantially reduced and distilling to recover a fraction boiling below 450° C.

8. As a new composition of matter, an aqueous treating bath for fibrous material containing an alkali metal salt of abietene sulfonic acid.

In testimony whereof, we have hereunto subscribed our names at Carrollville, Milwaukee County, Wisconsin.

IVAN GUBELMANN.  
HENRY J. WEILAND.  
CLYDE O. HENKE.