Patented Mar. 2, 1943

2,312,864

UNITED STATES PATENT OFFICE

2,312,864

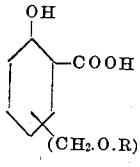
DERIVATIVES OF o-HYDROXYBENZENE-CARBOXYLIC ACIDS AND PROCESS OF MANUFACTURE

Jakob Bindler, Basel, Switzerland, assignor to the firm J. R. Geigy A. G., Basel, Switzerland

No Drawing. Application February 3, 1940, Serial No. 317,214. In Switzerland February 8, 1939

10 Claims. (Cl. 260—521)

This invention relates to a process of imparting to natural and regenerated cellulose a soft and flowing feel by treating it with a soluble salt of a compound having the configuration



wherein R is an aliphatic or alicyclic radical having more than 10 carbon atoms which may also be unsaturated and n is 1 or 2. The free positions of the benzene nucleus may carry any desired groups or atoms, for instance CH_3 or halogen. The compound used may be applied to the fibre together with direct dyestuffs. It is known that salts of aromatic hydroxycarboxylic acids substituted in the aromatic nucleus by radicals of high molecular weight, which radicals may be interrupted by hetero atoms, may be used as substitutes for soap. That compounds of this class corresponding with the above formula have substantive properties is surprising, since this is not only undesirable for material having soapy properties, but forbids the use of such material as a soap.

The soluble salts of alkyloxymethyl compounds having the foregoing general formula are obtained by causing a chloromethyl or hydroxymethyl substitution product of a hydroxycarboxylic acid of the benzene series or of the corresponding acid anhydride to react with an aliphatic or alicyclic alcohol having more than 10 carbon atoms and converting the product into a soluble salt by neutralisation. The condensation is carried out in the usual manner for example in an organic solvent such as benzene or chlorobenzene. The sodium salts of the alkali-soluble condensation products are bright yellow powders or waxy masses; they yield with water solutions which foam strongly and have pronounced softening action.

It has been proposed in British Patent No. 17,118 of 1899 to react chloromethyl derivatives of aromatic hydroxy carboxylic acids with alcohols, such as methyl and ethyl alcohol. The products obtained by the present invention with the aid of alcohols having more than 10 carbon atoms are distinguished from the products obtained with the aid of the lower alcohols by their valuable softening effects on natural and regenerated cellulose.

The following examples illustrate the invention, the parts being by weight:

Example 1

A hank of artificial silk is dyed in a liquor ratio of 1:25 in usual manner with a substantive dyestuff in a boiling bath which contains Glauber's salt. Together with the dyestuff there is added 1–4 per cent of a soluble salt of an ether of the above formula. After dyeing the goods are rinsed cold as usual and dried. The finished hank has a soft flowing feel. Thus the dyeing of viscous yarn may be carried out as follows:

10 grams of viscous yarn are dyed for instance in 250 cc of water with 2 per cent of diphenyl blue black GHS (Colour Index 1924, No. 402) with the addition of 2 per cent of sodium salt of 5-octadecyloxymethyl-2-oxybenzoic acid and 20 per cent of Glauber's salt, the latter being added only after dyeing has proceeded for about ¼ hour. The whole dyeing operation occupies about 1 hour.

Example 2

A foulard or a padding machine is charged with a solution of 2 per cent strength of sodium salt of 5-octadecyloxymethyl-2-oxybenzoic acid and 100 parts by weight of cotton material are treated therein and subsequently pressed to 200 parts by weight and then dried. The goods acquire a very good soft feel.

The sodium salt of 5-octadecyloxymethyl-2-oxybenzoic acid is made in the following manner:

27 parts of octadecyl alcohol and 25 parts of 5-hydroxy- or 28 parts of 5-chloromethyl-2-hydroxybenzene-1-carboxylic acid of 84 per cent strength are dissolved in 100 parts of chlorobenzene and condensed over night at 100 to 120° C. The solution is then freed by filtration from impurities and the chlorobenzene is distilled in a vacuum. The semi-solid residue is washed several times with hot water and then dissolved in caustic soda to produce the sodium salt and the solution is evaporated. There is obtained a bright powder.

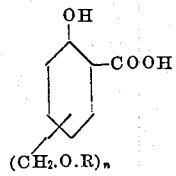
If in the above prescription there are used 22 parts of 5-chloromethyl-2-hydroxy-3-methylbenzene-1-carboxylic acid or 3-chloromethyl-2-hydroxy-5-methylbenzene-1-carboxylic acid of 100 per cent strength and 100 parts of chlorobenzene and 27 parts of octadecyl alcohol or of a technical mixture of octadecyl and hexadecyl alcohol or 24.6 parts of hexadecyl alcohol or 26.8 parts of octadecenyl alcohol there is obtained by the same method of condensation a light yellowish solid or waxy neutralised condensation product. A similar compound is also obtained when 37 parts of commercial dodecyl-alcohol are condensed with 26 parts of 3,5-di-(chloromethyl) 2-hydroxybenzene-1-carboxylic acid of 100 per cent strength in 200 parts of chlorobenzene and the product is treated in the manner indicated. Instead of the commercial dodecyl alcohol equivalent parts of technical octadecyl alcohol (containing hexadecyl alcohol), of hexadecyl or hex-

adecenyl alcohol may be used; the resulting products possess similar properties.

Instead of the above named alcohols other alcohols especially mixtures which are obtained by the reduction of vegetable or animal fats, of resins, of waxes, of naphthenic acids or of behenic acid may be used. There may be named by way of examples the alcohol mixtures from palm kernel fatty acid, from fish oil fatty acid, from cotton seed oil fatty acid, from ricinoleic acid, from sperm oil etc. The alkyloxymethyl compounds obtained have quite similar properties. Halogen substituted chloromethyl compounds, for instance 5-chloromethyl-3-chloro- or bromo-2-hydroxybenzoic acid are also very suitable for the reaction with all of the above mentioned alcohols or alcohol mixtures.

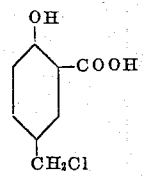
What I claim is:

1. A process for the manufacture of soluble salts of derivatives of o-hydroxybenzene-carboxylic acids having the general formula:



wherein n stands for one of the figures 1 and 2 and R stands for a radical of the aliphatic and alicyclic series having more than 10 carbon atoms, by causing a halgeno-methyl-o-hydroxy-benzene-carboxylic acid of the benzene series to react with an alcohol selected from the group consisting of aliphatic and alicyclic alcohols having more than 10 carbon atoms and converting the acid into a soluble salt by neutralisation.

2. A process for the manufacture of soluble salts of derivatives of o-hydroxybenzene-carboxylic acids, comprising causing chloromethyl-salicylic acid of the formula:



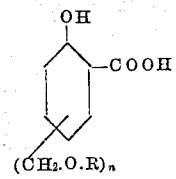
to react with an alcohol selected from the group consisting of aliphatic and alicyclic alcohols having more than 10 carbon atoms and converting the acid into a soluble salt by neutralisation.

3. A process for the manufacture of a soluble salt of a derivative of a o-hydroxybenzene-carboxylic acid, comprising causing chloromethyl-salicylic acid to react with octadecyl alcohol and converting the product into the sodium salt by neutralisation.

4. A process for the manufacture of a soluble salt of a derivative of a o-hydroxybenzene-carboxylic acid, comprising causing chloromethyl-salicylic acid to react with hexadecyl alcohol and converting the product into the sodium salt by neutralisation.

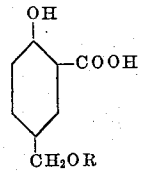
5. A process for the manufacture of a soluble salt of a derivative of a o-hydroxybenzene-carboxylic acid, comprising causing chloromethyl-salicylic acid to react with dodecyl alcohol and converting the product into the sodium salt by neutralisation.

6. The o-hydroxy-benzene-carboxylic acids having the following formula



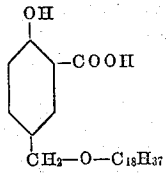
wherein n stands for one of the figures 1 and 2 and R stands for a radical of the aliphatic and alicyclic series having more than 10 carbon atoms, said compounds being after neutralisation with caustic soda and evaporation bright powders or waxy masses well soluble in water, imparting to natural and regenerated cellulose a soft and flowing feel.

7. The o-hydroxy-benzene-carboxylic acids having the following formula



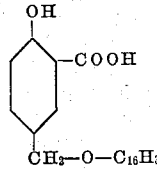
wherein R stands for a radical of the aliphatic and alicyclic series having more than 10 carbon atoms, said compounds being after neutralisation with caustic soda and evaporation bright powders or waxy masses well soluble in water, imparting to natural and regenerated cellulose a soft and flowing feel.

8. The o-hydroxy-benzene-carboxylic acid having the following formula



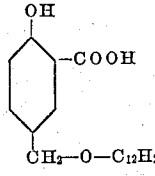
said compound being after neutralisation with caustic soda and evaporation a bright powder well soluble in water, imparting to natural and regenerated cellulose a soft and flowing feel.

9. The o-hydroxy-benzene-carboxylic acid having the following formula



said compound being after neutralisation with caustic soda and evaporation a bright powder well soluble in water, imparting to natural and regenerated cellulose a soft and flowing feel.

10. The o-hydroxy-benzene-carboxylic acid having the following formula



said compound being after neutralisation with caustic soda and evaporation a waxy mass well soluble in water, imparting to natural and regenerated cellulose a soft and flowing feel.

JAKOB BINDLER.