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METHOD FOR THE REFINING OF TALL OIL SOAP

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This invention relates to a method for the refining of an aqueous tall oil soap containing undesirable non-saponifiable components and color bodies to effect a substantial removal of such undesirable constituents.

Tall oil soap (skimmings) is obtained in quantity in the manufacture of pine wood pulp by the sulfate process, and is composed primarily of an aqueous solution of the sodium salts of various fatty and resin acids, together with a minor but appreciable proportion of non-saponifiables and color bodies. For the tall oil released by the acidification of such a tall oil soap to be of the greatest utility in the manufacture of esters, alkyl resins, etc., it is necessary that the tall oil be substantially free from the aforementioned non-saponifiables and color bodies. Many processes have been suggested for the refining of tall oil as such (e. g., distillation, contacting with adsorbent or mineral acids, etc.), but such processes do not result in an effective separation of the aforementioned undesirable components from the tall oil. For example, distillation at temperatures in excess of about 100° C. of a tall oil containing non-saponifiables results in the formation of non-distillable esters by a combination of the tall oil fatty acids and the non-saponifiables, thus reducing the amount of commercially useful fatty acids in such distilled tall oil.

The foregoing being briefly the state of the art, it is an object of this invention to provide a method for the refining of a crude tall oil soap containing non-saponifiable components and color bodies to effect a substantial removal of such undesirable materials.

It is also an object of this invention to provide a tall oil soap from which there may be released by acidification a tall oil which may be more advantageously processed in accordance with known procedures to provide an improved and further refined tall oil.

The foregoing and other objects are accomplished in accordance with this invention by dissolving in certain ketones an aqueous solution of a tall oil soap containing undesirable non-saponifiables and color bodies, and thereafter diluting the ketone solution with water to form a ketone layer and an aqueous tall oil soap layer containing reduced quantities of non-saponifiables and color bodies.

The examples which follow illustrate specific embodiments of the method of this invention, and are to be considered not limitative thereof.

Example I

139 parts by weight of a crude tall oil soap containing 65% solids was dissolved in 839 parts by

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weight of methyl isobutyl ketone while the solvent was being refluxed. The solution was then refluxed for 15–20 minutes, and was then cooled to 60° C., after which 800 parts by weight water was added. The mixture separated into two layers after a period of about 15 minutes. Analyses of the tall oil soap before and after having been subjected to the foregoing treatment showed that the non-saponifiable content thereof had been reduced from 5.4 to 0.9%, based upon the total solids content of the tall oil soap, and also that the color of the tall oil soap had been substantially decreased.

The aqueous tall oil soap layer prepared as just described contains a small amount of methyl isobutyl ketone, which assists in the retention of the aforementioned 0.9% of non-saponifiables in that layer. However, such residual non-saponifiable content may be easily further reduced by contacting the aqueous tall oil soap layer with fresh portions of the ketone. Thus, by mixing the aqueous tall oil soap layer at reflux temperature with an equal volume of methyl isobutyl ketone, cooling the mixture, separating the aqueous tall oil soap layer from the methyl isobutyl ketone layer, and repeating this process three times more, the non-saponifiable content of the aqueous tall oil soap layer was further reduced to 0.3%, based upon the total solids content of the tall oil soap.

Example II

10 parts by weight of a crude tall oil soap containing 65% solids was dissolved in 200 parts by weight of methyl isobutyl ketone by stirring them together at room temperature for a period of about 30 minutes. The insoluble portion of the tall oil soap, representing 1.7% of the total solids content thereof, was filtered off. To 252 parts by volume of the filtered methyl isobutyl ketone solution was added at room temperature 125 parts by volume of water. Upon standing, the mixture separated into two layers, viz., an upper methyl isobutyl ketone layer amounting to about 235 parts by volume and containing a substantial proportion of the non-saponifiable constituents of the crude tall oil, and a lower aqueous layer amounting to about 141 parts by volume and containing the refined tall oil soap, which was substantially lighter in color than the crude tall oil soap subjected to the aforementioned refining treatment.

The foregoing examples illustrate the method of this invention for the reduction of the content of non-saponifiables and color bodies in an aqueous solution of a tall oil soap by dissolving the solution in a ketone, and thereafter diluting the ketone solution with water to form a ketone

layer, and an aqueous tall oil soap layer containing reduced quantities of non-saponifiables and color bodies. Although methyl isobutyl ketone was employed as a solvent for the aqueous tall oil soap in the examples, there may be substituted for such ketone any ketone which is a liquid at ordinary temperatures and which at ordinary temperatures is soluble in water to the extent of about 0.2 to about 10% by weight, such as diethyl ketone, methyl n-propyl ketone, methyl n-butyl ketone, methyl n-amyl ketone, etc. Methyl isobutyl ketone is the preferred ketone, however, because of its cheapness and availability. As the examples show, the aqueous tall oil soap solution may be dissolved in the ketone over a wide range of temperatures, for example at room temperature (Example II) or at the boiling point of the ketone (Example I). Furthermore, although the aqueous tall oil soap used in the examples contains 65% by weight of solids, this percentage is not critical. In general, the aqueous tall oil soap to be refined in accordance with the method described herein should preferably contain from about 30 to about 75% by weight of solids in order that the refining may be effected most economically. The upper limit of concentration of the tall oil soap in the aqueous solution treated is determined by the ease of handling such solution as a liquid.

In addition, the amount of ketone used to dissolve the aqueous solution of the tall oil soap will depend upon various factors, such as the particular ketone used, the concentration of tall oil soap in the aqueous solution thereof, upon the temperature at which the solution is effected, etc. However, the volume of ketone employed is generally from about 8 to about 60, and preferably from about 10 to about 20, times the volume of the water contained in the aqueous tall oil soap solution to be refined. Solution of the aqueous tall oil soap in the ketone results in a precipitation of the sodium salts of certain acidic color bodies and a portion of the sodium salt of lignoceric acid contained in the tall oil soap being treated, and the precipitate thus formed should preferably be separated from the remainder of the ketone solution at this stage of the procedure.

As is seen from the examples, the addition of sufficient water to the solution of aqueous tall oil and ketone results in the formation of two layers, one of which is the ketone substantially free from tall oil soap but containing a substantial proportion of the non-saponifiables and color bodies originally present in the crude tall oil soap, and the other of which is an aqueous solution of the tall oil soap having a reduced content of non-saponifiables and color bodies. The partition constant greatly favors the ketone phase with respect to the non-saponifiable materials. When a crude aqueous tall oil soap containing 65% of solids is refined, the most effective separation of the two layers is in general accomplished by the addition to the solution of aqueous tall oil soap in the ketone of at least four volumes of water, based upon the volume of crude aqueous tall oil soap present, followed by a settling period of at least 15 minutes. Residual amounts of the ketone dissolved in the aqueous phase may be removed therefrom and recovered for further use by heating such aqueous phase and distilling the ketone therefrom.

Both the aqueous layer and the ketone layer produced in accordance with the foregoing procedure are valuable compositions of matter.

Thus, acidification of the aqueous layer results in the separation of a tall oil of improved properties for the preparation of esters, alkyd resins, etc. The tall oil produced by such acidification may also be further refined in accordance with known methods, such as distillation, contacting with adsorbents, etc. The ketone layer, on the other hand, may be stripped of solvent, leaving a residue having a high content of beta-sitosterol.

We claim:

1. The method of refining an aqueous solution of a tall oil soap comprising 25% to 70% water and also containing undesirable non-saponifiables and color bodies, which method comprises dissolving said aqueous solution to form a single phase in a dialkyl ketone which is liquid at ordinary temperatures and which, at ordinary temperatures, is soluble in water to the extent of about 0.2% to about 10% by weight and thereafter separating the ketone solution into a ketone layer containing the major portion of the non-saponifiables and color bodies and an aqueous layer containing the major portion of the tall oil soap by adding further water to the said ketone solution.

2. The method of refining an aqueous solution of a tall oil soap comprising from about 30% to about 75% by weight of solids and containing undesirable non-saponifiables and color bodies, which method comprises dissolving said aqueous solution to form a single phase in a dialkyl ketone which is liquid at ordinary temperatures and which, at ordinary temperatures, is soluble in water to the extent of 0.2% to about 10% by weight and thereafter separating the ketone solution into a ketone layer containing the major portion of the non-saponifiables and color bodies and an aqueous layer containing the major portion of the tall oil soap by adding further water to the said ketone solution.

3. The method of refining an aqueous solution of a tall oil soap comprising 25% to 70% water and also containing undesirable non-saponifiables and color bodies, which method comprises dissolving said aqueous solution in methyl isobutyl ketone to form a single phase and thereafter separating the ketone solution into a ketone layer containing the major portion of the non-saponifiables and color bodies and an aqueous layer containing the major portion of the tall oil soap by adding further water to such ketone solution.

4. The method of refining an aqueous solution of tall oil soap comprising from about 30% to about 75% by weight of solids and also containing undesirable non-saponifiables and color bodies, which method comprises dissolving said aqueous solution in methyl isobutyl ketone to form a single phase and thereafter separating the ketone solution into a ketone layer containing the major portion of the non-saponifiables and color bodies in an aqueous layer containing the major portion of the tall oil soap by adding further water to the said ketone solution.

5. The method of refining an aqueous solution of a tall oil soap comprising 25% to 70% water and also containing undesirable non-saponifiables and color bodies, which method comprises dissolving said aqueous solution to form a single phase in a dialkyl ketone which at ordinary temperature is soluble in water to the extent of 0.2% to about 10% by weight, at a temperature within the range from about room temperature to the boiling point of the ketone and thereafter separating the ketone solution into a ketone layer

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containing the major portion of the non-saponifiables and color bodies and an aqueous layer containing the major portion of the tall oil soap by adding further water to the said ketone solution.

6. The method of refining an aqueous solution of a tall oil soap comprising 25% to 70% water and also containing undesirable non-saponifiables and color bodies, which method comprises dissolving said aqueous solution in a dialkyl ketone which at ordinary temperature is soluble in water to the extent of about 0.2% to about 10% by weight, at a temperature within the range from about room temperature to the boiling point of the ketone, separating any precipitated solids from the solution and thereafter separating the ketone solution into a ketone layer containing the major portion of the non-saponifiables and color bodies and an aqueous

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layer containing the major portion of the tall oil soap by adding further water to the said ketone solution.

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