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METHOD OF REFINING TALL OIL

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The present invention relates to an improved method of refining tall oil.

It is well known that crude tall oil is the dark colored, ill smelling mixture obtained from the sodium soap known as sulfate soap formed in the process of producing paper pulp commonly known as the kraft or sulfate process. This soap is skimmed off on evaporation and cooling of the cooking liquor and on treatment with acid the sodium salt of the acid is formed and the organic acids are set free as an odorous dark colored material which is an oil at ordinary temperature and known as crude tall oil. This oil consists essentially of a mixture of fatty acids of the oleic and linoleic series and rosin acids. There is also generally 10 to 15% of materials not of acid character present, of which phytostearin is probably the most important though present to the extent of only 2 to 3%.

The presence of this soap in the alkaline liquor which has been used to cook the wood chips is due to the fact that the coniferous woods which are generally used contain small amounts of fats, resins, etc., and that during the cooking process these products are saponified and go into the liquor as the alkali soaps of the organic acids. The average composition of tall oil may be taken as somewhat more than 40% each of fatty acids and of rosin acids and 10 to 15% of inert substances, i. e., substances that do not have an organic acid character.

The sulfate soap and the tall oil itself are available in large quantities but up to the present have few industrial applications and none of these is of large tonnage. An important reason why it is not used is because of the dark color and disagreeable odor which prior methods of refining have not been successful in removing. For better and more acceptable commercial use, the refined tall oil must be free of both odor and dark color. Although many attempts have been made to provide an acceptable method, none as far as I am aware, has been wholly satisfactory and successful in practical and commercial operation, particularly on an industrial scale.

I have discovered that the color-carrying material is at least largely due to oxidized or oxidizable material. The odor producing compounds are mainly sulfur compounds and are largely of the mercaptan type. By proceeding in accordance with the principles of my invention, the objectionable color and odor can be removed from tall oil whereby large prospective uses may be satisfied with my refined tall oil.

It is an object of the present invention to pro-

vide a method of refining tall oil which inexpensively removes the undesirable color and odor.

A further object of the invention is to provide a method of refining tall oil which can be carried into practice on an industrial scale with standard equipment.

The present invention also contemplates the provision of a method of effectively eliminating color and odor with an inexpensive and universally available solvent of a hydrocarbon type.

It is likewise within the contemplation of the invention to provide an improved method of refining tall oil with a solvent of the gasoline type which has the multiple function of dissolving the desirable constituents in a recoverable solution, of being inert with respect to a major portion of the coloring material and allowing said portion to remain as an insoluble residue, of diluting the tall oil to permit the treatment with other reagents, and of carrying away the odorous substances when distilled and leaving the refined tall oil substantially free from color and odor.

Generally speaking the method contemplates the use of an inexpensive solvent, gasoline, which serves the triple purpose of dissolving the desirable constituents but leaving behind a large portion of the coloring material, of diluting the oil so that it can be conveniently treated with other reagents, and when the gasoline is distilled off from the oil by means of heat or steam of carrying away the odorous materials and leaving the oil essentially free from odor. While in solution in the gasoline the tall oil may be further decolorized by treatment, preferably warm, with air and filtering. As a third step in removing color, if it is desired to have the oil as light as possible, the gasoline solution of tall oil may be mixed with adsorbent carbon and, after remaining in contact with it for a period of time and preferably kept hot, filtered. In place of adsorbent carbon, fuller's earth may be used or the solution may be shaken with furfural at a temperature of about 50° C. The furfural dissolves the residual coloring material but does not dissolve any substantial amount of the gasoline or tall oil. The gasoline is then distilled from the filtrate, thus leaving refined tall oil which is essentially free from color and odor and which is ready for shipment for commercial uses.

For the purpose of giving those skilled in the art a better understanding of the invention, the following illustrative examples are given:

Example No. 1

About 500 kilograms crude tall oil are stirred

with about 4000 liters of heavy gasoline and decanted or filtered from the gasoline insoluble portion which will vary from 30 to 125 kilograms of material depending on the particular sample of crude tall oil taken. To the gasoline solution is added an adsorptive material, such as a decolorizing carbon, and the whole agitated at about 60° C. for half an hour and then filtered. The filtrate is steam distilled to remove the gasoline and to leave the tall oil in a commercially useful form, i. e. with only a light color and a mild pleasant odor. The odorous materials which pass over with the gasoline vapors can be destroyed in the gasoline by treatment with a hypochlorite, a process that could not be used with the original tall oil without injuring the valuable constituents thereof. The gasoline is then ready to be reused in the process.

Example No. 2

About 500 kilograms of crude tall oil are treated with about 4000 liters of heavy gasoline and the gasoline insoluble portion filtered off. The filtrate is stirred with about 1000 liters of furfural preferably in batches, at a temperature of about 50° C. for about 15 minutes. The batch is then permitted to stand and cool. The furfural with the dissolved colored materials settles to the bottom and is drawn off and distilled to recover the furfural for reuse. The gasoline is distilled from the decolorized tall oil-gasoline solution and there remains a light yellow mild odored tall oil ready for commercial use.

Example No. 3

About 500 kg. crude tall oil are heated to approximately 100° C. by a steam coil and a slow current of air blown through the mass for about 8 hours. It is then cooled and stirred with about 4000 liters of gasoline and filtered from the gasoline insoluble portion. The gasoline solution of purified tall oil is then steam distilled to remove the gasoline and the residual tall oil heated to remove any condensed water. On cooling it is found to be nearly odorless, of light color, and satisfactory for making soap and for other uses.

In a modification of this process the viscous sulfate soap-water mixture is given a similar air treatment, then cooled, partially dissolved in water, acidified, the tall oil separated, treated with gasoline, filtered, and the gasoline solvent removed.

It is desirable to remove as much of the coloring material as possible by rendering it insoluble in gasoline rather than by taking it out with fuller's earth, absorbent carbon, or furfural. The coloring materials may be rendered insoluble in gasoline by oxidation methods. For example, by drying the sulfate soap to complete dryness and heating it to 100 or 110° C. in contact with air, then dissolving in water, acidifying, and treating as before. A second method consists in heating the viscous sulfate soap water mixture for a considerable period of time with air passing continuously through it and then setting free the tall oil from the soap. A third method consists in treating the sulfate soap with a small amount of a chemical oxidizing agent such as hydrogen peroxide, say 1 to 2% of the dry weight of the soap and then setting free the tall oil from the soap. A fourth method consists in blowing the hot tall oil with air, either by itself or in the hot tall oil solution in gasoline.

In carrying the invention into practice, gaso-

line having the following characteristics has given satisfactory results:—

Average distillation point 125° C., free from the lead tetraethyl commonly present in motor fuel, low in unsaturated hydrocarbons, a straight run gasoline. In other words, a cheap gasoline of the older type now not so much wanted as a motor fuel.

It has also been found that lower boiling gasolines and less saturated gasolines will serve but they have the disadvantage that the extraction with furfural is not so easily carried out, while higher boiling gasolines have the disadvantage of requiring a higher distillation temperature for their recovery.

Benzol and toluol may likewise be substituted for the preferred gasoline but have the disadvantages of higher price per gallon and that they are somewhat better solvents for the oxidized coloring matter and hence do not give so satisfactory a separation of the material to which the undesirable color is due.

I claim:

1. The process of refining tall oil which comprises oxidizing crude tall oil to render as much of the coloring material insoluble in gasoline as possible, then treating the treated tall oil with gasoline to dissolve tall oil to form a solution and to leave an insoluble residue, separating said insoluble residue from the gasoline containing dissolved tall oil, and distilling the tall oil-gasoline mixture to remove the gasoline and odorous substances whereby a refined tall oil is produced.

2. The process of refining tall oil which comprises oxidizing crude tall oil to render as much of the coloring material insoluble in gasoline as possible, then treating the treated tall oil with gasoline to dissolve tall oil to form a solution and to leave an insoluble residue, separating said insoluble residue from the gasoline containing dissolved tall oil, adding adsorbent carbon to said gasoline to remove coloring substances, separating the carbon from the gasoline, and distilling the tall oil-gasoline mixture to remove the gasoline and odorous substances whereby a refined tall oil is produced.

3. The process of refining tall oil from sulfate soap which comprises drying sulfate soap to complete dryness, heating the said soap to 100 to 110° C. in contact with air, dissolving in water, acidifying to liberate crude tall oil, treating the crude tall oil with gasoline to dissolve tall oil to form a solution and to leave an insoluble residue, separating said insoluble residue from the gasoline containing dissolved tall oil, and distilling the tall oil-gasoline mixture to remove the gasoline and odorous substances whereby a refined tall oil is produced.

4. The process of refining tall oil from sulfate soap which comprises heating viscous sulfate soap mixture with water for a considerable period while passing air therethrough, acidifying to liberate crude tall oil, treating the crude tall oil with gasoline to dissolve tall oil to form a solution and to leave an insoluble residue, separating said insoluble residue from the gasoline containing dissolved tall oil, and distilling the tall oil-gasoline mixture to remove the gasoline and odorous substances whereby a refined tall oil is produced.

5. The process of refining tall oil which comprises treating crude tall oil with gasoline to dissolve tall oil to form a solution and to leave an insoluble residue, blowing air through said solution while hot, separating said insoluble resi-

due from the gasoline containing dissolved tall oil, and distilling the tall oil-gasoline mixture to remove the gasoline and odorous substances whereby refined tall oil is produced.

5 6. The process of refining tall oil from sulfate soap which comprises treating sulfate soap with a small amount of a chemical oxidizing agent of the hydrogen peroxide type, dissolving in water, acidifying to liberate crude tall oil, treating the
10 crude tall oil with gasoline to dissolve tall oil to form a solution and to leave an insoluble residue, separating said insoluble residue from the gasoline containing dissolved tall oil, and distilling the tall oil-gasoline mixture to remove the
15 gasoline and odorous substances whereby a refined tall oil is produced.

7. The process of refining tall oil which comprises oxidizing crude tall oil to render as much of the coloring material insoluble in gasoline as
20 possible, treating the treated tall oil with gasoline to dissolve tall oil to form a solution and to leave an insoluble residue, separating said insoluble residue from the gasoline containing dissolved tall oil, subjecting the gasoline-treated

tall oil to the action of furfural to dissolve coloring material, removing said furfural containing coloring matter from the gasoline containing tall oil, and distilling the tall oil-gasoline mixture to remove the gasoline and odorous substances whereby a refined tall oil is produced.

8. The process of refining tall oil from sulfate soap which comprises heating viscous sulfate soap mixture with water for a considerable period while passing air therethrough, acidifying to liberate crude tall oil, treating the crude tall
10 oil with gasoline to dissolve tall oil to form a solution and to leave an insoluble residue, separating said insoluble residue from the gasoline containing dissolved tall oil, subjecting the gasoline-treated tall oil to the action of furfural to dissolve coloring material, removing said furfural containing coloring matter from the gasoline
15 containing tall oil, and distilling the tall oil-gasoline mixture to remove the gasoline and odorous substances whereby a refined tall oil is produced.
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