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METHOD OF PREPARATION OF A SOAP

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This invention relates to an improved method for the production of soap made from a gasoline-insoluble resin derived from pine wood.

Heretofore, it has been known to produce a dry size from a gasoline-insoluble pine wood resin. However, the saponification has been carried out at temperatures above the melting point of the resin, requiring the use of superatmospheric pressure inasmuch as the gasoline-insoluble pine wood resin is solid at the boiling point of water at atmospheric pressure. The use of pressure equipment is expensive and hazardous.

Heretofore, it has also been known to produce a soap by saponification of rosin with an alkali in liquid, aqueous medium at temperatures of approximately 100° C. When such a procedure is used with the gasoline-insoluble pine wood resin a precipitate generally settles out from the size solution presumably due to the effect of heat on the resin. Such a precipitate presents obvious disadvantages in uses of the soap, such as in sizing of paper.

Now, in accordance with my invention, I have found that I may obviate the disadvantages above cited and produce a soap from a gasoline-insoluble pine wood resin without the use of heat. More particularly I have found a method by which an essentially neutral soap may be produced from gasoline-insoluble pine wood resin without the use of expensive and hazardous pressure procedures and without formation of a water-insoluble precipitate. Furthermore, my improved procedure presents economies over prior art soap-making procedures in that it requires no heating.

The gasoline-insoluble pine wood resin I employ in my improved method of preparation of soap therefrom is hereby defined as the resin described in U. S. Patent No. 2,102,122, to Fred H. Lane, and further described and claimed in application for U. S. patent, Serial No. 61,745, filed January 31, 1936, by Lucius C. Hall, which resin may be produced by extracting resinous wood with coal tar hydrocarbon, removing said hydrocarbon by evaporation, extracting the residue with a petroleum hydrocarbon and removing a gasoline-insoluble resin. To further identify the gasoline-insoluble pine wood resin which I employ in my invention, I may point out that by using the process described above, 50-150 pounds of the above resin per ton of wood may be produced, depending on the details (time, temperature, etc.) of the separation of FF rosin from the gasoline-insoluble resin. A typical example of the gasoline-insoluble resin will have

the following characteristics: melting point, A. S. T. M. drop method, 125° C., acid No. 100, naphtha-insoluble matter 98%, petroleum ether-insoluble matter 98%, gasoline-soluble matter 8%.

In carrying out my improved procedure for making soap from the gasoline-insoluble pine wood resin I prefer to use the resin in pulverized form, preferably above 100 mesh. The process is carried out in two stages.

In the first stage the pulverized resin is dispersed in cold water. Dispersion may be accomplished in any convenient manner, such as by addition of a small amount of alkali to the water, say about 10 per cent of the alkali required for saponification of the resin. Likewise, the pulverized resin may be dispersed in the water with any suitable dispersing agent, such as, for example, a solution of casein in sodium hydroxide, sodium oleate, sodium alginate, sulphonated higher alcohols, sulphonated naphthalenes, bentonite, etc. The pulverized resin may also be dispersed in water mechanically without the use of agents as above. Such mechanical dispersion may be accomplished by making a heavy paste of about 60 per cent or more pulverized resin and water in a suitable type of mixer, adding water and mixing until a smooth paste results. Dilution of the smooth paste to lower solids content is then preferable for convenience of handling in the subsequent operation.

The above dispersion of the resin in water may be carried out over a wide range of concentration. I have prepared satisfactory dispersions in concentrations of about 10 per cent to about 60 per cent but prefer to carry out the dispersion with about 20 per cent to about 30 per cent solids.

In the second stage the pulverized gasoline-insoluble pine wood resin dispersed in water as above described is saponified with alkali without the use of heat. The saponification is carried out by addition of a concentrated alkali solution to the dispersion with mechanical agitation.

Although I have described my procedure as involving first the dispersion of the pulverized gasoline-insoluble pine wood resin in water then the saponification of the resin by addition of alkali, I find that other procedures may be used to produce the same result. Thus, I may saponify the resin by adding the latter in dry form to the caustic solution, thereby dispersing and saponifying the resin in one operation.

As specific examples of my improved method

of preparation of soap from gasoline-insoluble pine wood resin the following are illustrative:

Example 1

Seventy pounds of pulverized gasoline-insoluble pine wood resin are added with agitation to a solution of 0.8 pound of sodium hydroxide dissolved in 23½ gallons of water. After agitation has continued for about 10 minutes a solution of 6½ pounds of sodium hydroxide dissolved in 2½ gallons of water is added to the dispersion at room temperature with agitation. After agitation has continued for about 30 minutes the soap is ready for use, for example, as a sizing agent. A typical analysis is as follows:

	Percent
Free resin	2.5
Free alkali as Na ₂ CO ₃	0.0
Total solids	27.5

Example 2

Thirty pounds of pulverized gasoline-insoluble pine wood resin are added with agitation to a solution of 0.3 pound sodium hydroxide dissolved in 10½ gallons of water. After agitation has continued for about 10 minutes a solution of 4 pounds of sodium hydroxide dissolved in 1¼ gallons of water is added at room temperature to the dispersion with agitation. After agitation has continued for about 30 minutes the preparation is complete. A typical analysis is as follows:

	Percent
Free resin	0.0
Free alkali as Na ₂ CO ₃	1.15
Total solids	23.9

Example 3

Thirty-one pounds of pulverized gasoline-insoluble pine wood resin are added to a solution of 0.3 pound of a solution of casein in sodium hydroxide in 10 gallons of water with vigorous agitation. To this dispersion a solution of 3 pounds of sodium hydroxide dissolved in 1.2 gallons of water is added and agitation continued for 30 minutes. A typical analysis of a soap prepared in this manner is as follows:

	Percent
Free resin	3.4
Free alkali as Na ₂ CO ₃	0.2
Total solids	25.5

Example 4

One hundred and nineteen pounds of pulverized gasoline-insoluble pine wood resin are added with agitation to a solution of 0.17 pound sodium hydroxide dissolved in 10 gallons of water. After agitation has continued for about 10 minutes a solution of 10.8 pounds of sodium hydroxide dissolved in 2.3 gallons of water is added to the dispersion at room temperature with agitation. After agitation has continued for about 30 minutes the soap is ready for use for example as a sizing agent. A typical analysis is as follows:

	Percent
Free resin	1.1
Free alkali as Na ₂ CO ₃	0.1
Total solids	55

Example 5

Thirty-five pounds of pulverized gasoline-insoluble pine wood resin are added with agitation to a solution of 0.2 pound of potassium hydroxide dissolved in 10 gallons of water. After agitation has continued for about 10 minutes a solution of 4.2 pounds of potassium hydroxide dis-

solved in 2.1 gallons of water is added to the dispersion at room temperature with agitation. After agitation has continued for about 30 minutes the finished soap has an analysis as follows:

	Percent
Free resin	4.7
Free alkali as K ₂ CO ₃	0.12
Total solids	34.3

In the above examples potassium hydroxide and sodium hydroxide are considered as equivalents, substitution of one for the other being on the basis of equivalent amounts.

In practicing my invention I prefer to use a concentrated solution of alkali to effect the saponification, as the reaction is more rapid when the concentration of alkali is high. My preferred strength of alkali is about 20 to about 50 per cent. It will be appreciated, however, that the concentration of alkali is not considered critical, the lower limit being determined by the reactivity with the resin and the upper limit being dependent on the solution strength which can be conveniently prepared and handled.

The gasoline-insoluble pine wood resin soap solution produced in accordance with this invention may be used as a sizing material, such as for sizing of paper and board.

Where, in the claims, I refer to a gasoline-insoluble pine wood resin I refer to the residue produced by extracting resinous wood with coal tar hydrocarbon, removing said hydrocarbon by evaporation, extracting the residue, with a petroleum hydrocarbon, and recovering a gasoline-insoluble resin.

It will be appreciated that the details and proportions set forth in the examples herein are illustrative only, and that the invention as broadly described is in no way limited thereby.

What I claim and desire to protect by Letters Patent is:

1. A method of preparing a soap solution which comprises dispersing in water a gasoline-insoluble pine wood resin, produced by extracting resinous wood with coal tar hydrocarbon, removing said hydrocarbon by evaporation, extracting the residue with a petroleum hydrocarbon and recovering a gasoline-insoluble resin, carrying out said dispersion in water with about 10 to about 60 per cent solids concentration, and saponifying at room temperature with an aqueous alkali metal hydroxide solution of about 20 to about 50 per cent concentration to give a substantially neutral soap.

2. A method of preparing a soap solution which comprises dispersing in water a gasoline-insoluble pine wood resin, produced by extracting resinous wood with coal tar hydrocarbon, removing said hydrocarbon by evaporation, extracting the residue with a petroleum hydrocarbon and recovering a gasoline-insoluble resin, carrying out said dispersion in water with about 20 to about 30 per cent solids concentration, and saponifying at room temperature with an aqueous alkali metal hydroxide solution of about 20 to about 50 per cent concentration to give a substantially neutral soap.

3. A method of preparing a soap solution which comprises dispersing in water a gasoline-insoluble pine wood resin, produced by extracting resinous wood with coal tar hydrocarbon, removing said hydrocarbon by evaporation, extracting the residue with a petroleum hydrocarbon and recovering a gasoline-insoluble resin, carrying out said dispersion in water with about 10 to about

60 per cent solids concentration by addition of alkali, and saponifying at room temperature with an aqueous alkali metal hydroxide solution of about 20 to about 50 per cent concentration to give a substantially neutral soap.

4. A method of preparing a soap solution which comprises dispersing in water a gasoline-insoluble pine wood resin, produced by extracting resinous wood with coal tar hydrocarbon, removing said hydrocarbon by evaporation, extracting the residue with a petroleum hydrocarbon and recovering a gasoline-insoluble resin, carrying out said dispersion in water with about 10 to about 60 per cent solids concentration with a dispersing agent, and saponifying at room temperature with an aqueous alkali metal hydroxide solution of about 20 to about 50 per cent concentration to give a substantially neutral soap.

5. A method of preparing a soap solution which comprises dispersing in water a gasoline-insoluble pine wood resin, produced by extracting resinous wood with coal tar hydrocarbon, removing said hydrocarbon by evaporation, extracting the resi-

due with a petroleum hydrocarbon and recovering a gasoline-insoluble resin, carrying out said dispersion in water with about 10 to about 60 per cent solids concentration, and saponifying at room temperature with an aqueous sodium hydroxide solution of about 20 to about 50 per cent concentration to give a substantially neutral soap.

6. A method of preparing a soap solution which comprises dispersing in water a gasoline-insoluble pine wood resin, produced by extracting resinous wood with coal tar hydrocarbon, removing said hydrocarbon by evaporation, extracting the residue with a petroleum hydrocarbon and recovering a gasoline-insoluble resin, carrying out said dispersion in water with about 10 to about 60 per cent solids concentration, and saponifying at room temperature with an aqueous potassium hydroxide solution of about 20 to about 50 per cent concentration to give a substantially neutral soap.

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