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PUMPABLE POTASSIUM SOAPS

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This invention relates to soap making. More particularly, this invention relates to resin acid soaps and to methods of preparing the same.

As ordinarily produced, the potassium soaps derived from mixtures of resin acids and fatty acids occur in the form of a viscous, stiff gel and cannot be flowed or poured in a high solids form (i. e., greater than 30% solids) except at relatively high temperatures. Pumping of the soap will not take place except at temperatures substantially in excess of 150° F.

Because of these unusual characteristics, it is difficult to remove the soap from containers, especially containers having restricted access openings. The problem is seen more clearly in connection with the tank car shipment of potassium soap derived from fatty and resin acid mixtures. In this connection, it is highly impractical to unload the potassium soap in solid form. Instead, it is usually satisfactory to convert the soap to a fluid form so that it can be pumped from the tank car. According to the usual method of unloading cars, an enclosed steam line is introduced into the tank and steam is blown through the line until the soap contained in the tank is fluid. The fluid soap is then pumped out of the tank while hot and is allowed to cool. Refrigeration is sometimes employed to facilitate the cooling of the soap.

This practice of unloading the soap has several disadvantages. First, the practice requires high capacity heating facilities at the point of unloading. Such facilities are often unavailable, in which case unloading of the soap is impractical. Also, the practice is time consuming and involves relatively large costs in heating and refrigeration. Further, soap losses occur to some extent due to caking and burning at the heat transfer surface. Not only is the soap damaged due to caking and burning but by reason of the burned soap forming in an insulative layer, the heat is prevented from diffusing properly into the soap mass. Of course, it will be apparent that the above disadvantages apply to containers in general and are not confined alone to tank cars and the like.

Therefore, at the present time it would be desirable to have a potassium soap of fatty and resin acid mixtures which could be readily pumped, poured and otherwise transferred without the foregoing disadvantages.

An object of this invention is to provide an economical method of producing readily pumpable potassium soap of mixtures of fatty and resin acids. Another object is to provide potassium soap of mixed fatty and resin acids which is capable of being pumped at a temperature of 150° F. or lower. Another object of this invention is to provide a fatty and resin acid soap which may be readily transferred from containers. Still another object of this invention is to provide a potassium soap of fatty and resin acids which may be heated to a fluid pumpable condition without burning or discoloring the soap. Other specific objects and advantages will appear as this specification proceeds.

This invention is based on the discovery that when the moisture and free acid content of potassium soap derived from fatty and resin acid mixtures are maintained within critical limits, the objectionable gelling characteristics are eliminated and the soap is readily pumpable at 150° F. or lower.

The present invention may be more fully illustrated by the following preferred method of producing potassium soap, employing a mixture of 60% by weight resin acids (calculated as abietic acid), 32% fatty acids (calculated as oleic acid) and 8% unsaponifiable material. The mixture employed has a saponification value of 175.

Approximately 2000 pounds of the mixture just mentioned are mixed continuously in a crutcher with 725 pounds at 45% aqueous potassium hydroxide. The run is started at about 90° F., the temperature being allowed to rise during saponification to a finishing temperature of about 200 to 210° F. The finished soap contains 17% moisture and 5% free acid calculated as oleic, and consists largely of mixed potassium salts of resin acids and fatty acids. The soap flows at room temperature and may be readily pumped. At 120° F. the soap is a smooth gel with highly fluid properties.

Soaps produced in accordance with the present invention may be readily flowed and pumped, it being required in some cases to heat the soaps moderately to about 70° F. or higher, but in no case higher than 150° F. The present soaps not only are readily pumpable but also have a pronounced resistance to burning when brought into contact with heated surfaces. It has been determined, upon heating the present soaps to a fluid condition, that burning and caking are largely eliminated.

The present invention is applicable to mixtures of resin acids and fatty acids, particularly mixtures containing abietic acids, principally Steele's abietic acid, dehydroabietic acid, dihydroabietic acid, etc., and fatty acids containing 18 carbon atoms such as oleic, linoleic, linolenic, etc. Suitably the resin acids for these mixtures may be obtained from gum rosin, wood rosin, crude or refined tall oil, or other source of resin acids. Similarly, the fatty acids may be obtained from animal or vegetable oils, tall oil, or other suitable sources. Mixtures containing about 20 to 90% resin acids will generally be satisfactory. Preferably, the mixtures will contain from 40 to 70% resin acids, and for optimum advantage, from 60 to 70%. Usually a small amount of non-saponifiable material such as sterols, abietenes, etc., is associated with resin acids. The presence of a small proportion, such as 5 to 15% by weight, of such material is not objectionable for present purposes although it is preferred to eliminate such material altogether or at least to maintain such material at a minimum, as for example 5% or lower. The remaining portion of the applicable mixtures consists essentially of fatty acids having 18 carbon atoms, particularly oleic, linoleic and linolenic acids. Small amounts of higher and lower fatty acids (as for example adjacent homologs) may also be present. The saponification value of the mixtures employed may be from 150 to 200 and preferably from 160 to 185.

Since certain of the acids contemplated by the present invention are unstable and tend to break down when subjected to heat, air, etc., it is considered desirable in some cases to convert the acids to a stable acid form prior to saponification. Steele's abietic acid, in particular, is relatively unstable, and it is usually desirable therefore to convert Steele's acid to a more stable form prior to saponification. It will be convenient in effecting stabilization, to subject the mixture of fatty and resin acids to heat treatment in the presence of a stabilizing catalyst such as platinum or iodine. While desirable, stabilization is not necessary to the practice of the present invention, since satisfactory soaps may be prepared from mixtures which have not been stabilized.

I have found it important in the composition of the

present soaps, to maintain the proportions of moisture and free acid within narrow limits. For satisfactory results, the free acid content (calculated as oleic acid) may be from about 4 to 10% of the total weight of the soap and, preferably, from 4 to 6%. The moisture content may be from about 15 to 22% of the total weight and, preferably, from 16 to 18%. For example, a preferred soap will contain 17% moisture by weight, the balance being solids, of which 5% consists of free acid calculated as oleic acid. Proportions of moisture or free acid greater or lesser than those set forth result in a stiff gel that can be pumped only at temperatures substantially in excess of 150° F. For example, a potassium soap prepared from mixed fatty and resin acids (resin acid, 60%; fatty acid, 35%; unsaponifiable, 5%) and containing 3% moisture is a stiff gel that cannot be pumped. Addition of moisture to such a soap makes it fluid and pumpable in the range of 15 to 22% moisture. Further addition of moisture causes the soap to set up into a non-fluid gel which cannot be pumped. By way of example, a soap of this type containing 30% moisture is an extremely heavy, unpumpable gel.

As a saponifying or neutralizing agent, any potassium alkali may be used, especially potassium hydroxide (caustic potash), potassium carbonate (pearl ash), and mixtures thereof. The use of potassium hydroxide is preferred. Preferably, for acid materials having a saponification value of about 175, the potassium hydroxide (KOH) and acid material are employed in the weight ratio of 1:6, these proportions serving to provide in the finished soap a free acid content of from 4 to 10% by weight. Of course, it is understood that where the saponification value of the acid material is higher (or lower) than 175, correspondingly higher (or lower) proportions of KOH are necessary in order to provide in the finished soap the desired amount (i. e., 4 to 10% by weight) of free acid. It is advantageous to employ the saponifying agent in the form of an aqueous solution or slurry of a concentration sufficient to provide the desired saponifying action and to further provide the moisture content desired in the finished soap. For example, it is convenient to employ a 45 to 50% aqueous potassium hydroxide solution in an amount sufficient to neutralize about 95% of the acid content, thereby leaving the desired content of acid in the finished soap. In such a case, the amount of moisture escaping from the crutcher due to evaporation is such that the final moisture content in the finished soap ordinarily falls within the desired range. Of course it will be apparent that in a case where the moisture or acid content of the soap does not fall within the desired limits, it will be satisfactory to make adjustment to within these limits by reducing or increasing the moisture or adding acid or alkali, as the case may be. In any case, provision of a satisfactory soap in accordance with the present invention may be considered as involving the step of neutralizing and the step of adjusting the acid content and moisture content, but of course it will be more convenient to provide the correct proportions of ingredients (alkali, acid and water) prior to neutralization so that adjustment of the proportions of these ingredients is accomplished automatically in connection with neutralization.

In the use of pearl ash, it is found that a considerable amount of foaming is produced during saponification. If desired, the foaming may be reduced through the use of any suitable anti-foaming agent.

The temperature of saponification is not at all critical. The reaction will proceed at room temperature. Generally, however, it will be preferred to supply moderate heat at the start of a run in order to initiate the reaction and, since the reaction is exothermic, it is not necessary to supply further heat once the reaction has started. Usually a run is started by heating the charge to about 90 to 125° F. and, owing to heat of saponification, the temperature rises to about 200 to 210° F. before the soap

finishes. If saponification is found to be incomplete, additional saponifying agent may be charged to the crutcher, care being taken to assure a final free acid content of from 4 to 10%. Likewise, the moisture may be adjusted to assure a final moisture content of from about 15 to 22%.

Thorough mixing or agitation of the soap materials during saponification is important. The mixing serves to dissipate the violent heat of reaction produced, especially in cases where potassium hydroxide is employed as a saponifying agent. If localized overheating occurs, the soap is often produced in the form of a very heavy gel, or in the form of hard beads. The mixing action provided by conventional soap crutchers will generally be satisfactory.

A preferred method of preparing a pearl ash soap in accordance with the present invention may be set forth as follows:

1000 pounds of a tall oil fraction (60% resin acids, 35% fatty acids, and 5% unsaponifiable materials; saponification value, 180) and 258 pounds of pearl ash in aqueous solution, are charged to a crutcher at about 175° F. Mixing is started, and 0.02 pound of anti-foaming agent (Dow-Corning Anti-Foam A) are added. Mixing is continued until saponification is 85% complete. An excess of pearl ash is added to complete the saponification to 95%, the final moisture being 18% by weight. The resulting soap is a soft solid at 85° F. and is fluid and pumpable at 150° F.

While in the foregoing specification various details of this invention have been set forth for the purpose of illustration, it will be apparent that such details may be varied widely by those skilled in the art without departing from the spirit of the invention.

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1. A soap-making process, comprising heating a mixture of resin acids and higher fatty acids with potassium alkali at a saponifying temperature in the presence of water to cause substantial neutralization of said acids thereby providing a potassium soap, said mixture containing about 20 to 90% by weight resin acids and having a saponification value of about 150 to 200, and adjusting the free acid content, calculated as oleic acid and moisture content of said soap to about 4 to 10% and bout 15 to 22% by weight respectively.

2. In the method of producing a pumpable potassium soap, the steps of agitating at a saponifying temperature a mixture of resin acids and higher fatty acids, said mixture having a saponification value of about 175 and containing about 20 to 90% by weight resin acids, and potassium hydroxide in the weight ratio of about 6 to 1 in the presence of water to cause neutralization of said acids, thereby providing a soap containing about 4 to 10% free acid calculated as oleic acid, and adjusting the moisture content of said soap to about 15 to 22% by weight moisture.

3. A method of producing soap comprising mixing refined tall oil containing higher fatty acids and 20 to 90% by weight resin acids and having a saponification value of about 175 with caustic potash in the weight ratio of about 6 to 1 in the presence of water at a temperature of at least 70° F. to cause neutralization of the caustic potash and to provide an excess of free acid of about 4 to 10 weight per cent, calculated as oleic acid, and adjusting the water content of the mixture to about 15 to 22% by weight.

4. A pumpable potassium soap, comprising potassium salts of resin acids and higher fatty acids contained in a tall oil fraction having a saponification value of 160 to 185 and a resin acid content of from 20 to 90% by weight, in admixture with 4 to 10% free acid, calculated as oleic acid and 15 to 22% moisture.

5. A fluid soap material comprising potassium salts of resin and higher fatty acids contained in a mixture of resins and higher fatty acids having a saponification value

of about 150 to 200 and a resin acid content of 20 to 90 per cent by weight, in admixture with 4 to 10 per cent by weight of free acid calculated as oleic acid and 15 to 22 per cent moisture.

6. A fluid soap material comprising potassium salts of resin and higher fatty acids contained in a mixture of resin and higher fatty acids having a saponification value of about 150 to 200 and a resin acid content of 40 to 70 per cent by weight, in admixture with 4 to 10 per cent by weight of free acid calculated as oleic acid and 15 to 22 per cent moisture.

7. A fluid soap material comprising potassium salts of resin and higher fatty acids contained in a mixture of

resin and higher fatty acids having a saponification value of about 180, a resin acid content of about 60 per cent by weight, a higher fatty acid content of about 35 per cent by weight, and an unsaponifiable content of 5 per cent by weight, in admixture with 5 per cent free acid calculated as oleic acid and 18 per cent moisture.

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