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MANUFACTURE OF SOAPS

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This invention relates to the manufacture of soaps, the invention being concerned with a novel type soap product, and with a process for producing such soap.

For various reasons which need not be considered in detail herein, production of good quality solid soaps from fatty oils has not been practicable heretofore except, possibly, by addition of relatively large amounts of other fatty materials and/or employment of relatively complicated processes.

It is an object of the present invention to produce such good quality solid soaps from fatty oils, in accordance with an extremely simple and inexpensive process.

The cost of the soap produced in accordance with this invention may be very low, not only because the ingredients used may be quite inexpensive, but also because of the simplicity of the manufacturing process.

Briefly summarized, the preferred mode of carrying out the present process contemplates employment of an oil and treatment of that oil with sodium carbonate, at elevated temperatures.

It is now known (see, for example, my Patent 1,957,437) that fatty oils may be solidified by heating in the presence of certain water-free alkaline agents, such as alkali metals, or oxides or hydroxides thereof, sodium hydroxide being a specific example.

I have found, however, that a better quality soap may be produced by the employment of alkali metal carbonates, especially sodium carbonate, the reasons being pointed out more fully hereinafter in connection with the following more specific description of the process.

It is here first noted that soaps in accordance with this invention may be produced from starting materials, particularly fatty oils such as those listed just below:

Tung oil
Oiticica oil
Linseed oil
Perilla oil
Sunflower oil
Poppyseed oil
Soya bean oil
Walnut oil
Rapeseed oil
Pineseed oil
Olive oil
Corn oil
Cottonseed oil
Coconut oil
Hydroxylated oils such as castor oil, etc.
Fish oils (train oils)
Acids of any of the above oils
Red oil (oleic acid)

From the list it will be seen that not only drying oils are suitable but also semi-drying and non-drying oils.

Solid fats (such as tallow and lard) and acids thereof may also be used, either alone or in admixture with oils, for instance, those listed above.

It is to be understood that reference in the claims to treatment of fatty oils comprehends various of the oil and fat materials of the types mentioned above.

In connection with products produced from fatty oils or fats in accordance with the present process, it is to be noted that, in contrast to many soaps, the product of the present invention is thermoplastic. For various purposes the product produced in accordance with the present invention may be further treated, for instance as disclosed in my copending application, Serial No. 455,132, filed concurrently herewith, in order to change the physical characteristics of the product.

The alkali metal and ammonium (which latter acts similarly to the former) carbonates, as a class, have characteristics suitable for use in the present process. Carbonates of lithium, sodium, potassium and ammonium are especially suitable and are all capable of producing solid soaps. Lithium carbonate will normally produce the hardest soap, other conditions being equal. Carbonates of sodium, potassium and ammonium yield progressively softer soaps, in the order listed, although all are capable of producing a solid soap product. Sodium carbonate is preferred for at least most purposes, for the reason that sodium carbonate is not only readily available but also is capable of producing a soap of good texture and hardness. By using potassium carbonate a good quality potassium soap is obtained.

Bicarbonates may also be used instead of the carbonates, although for most purposes I prefer the carbonates, particularly since when using bicarbonates the quantity of the reagent required is considerably increased. In some instances it may be advantageous to employ materials in addition to the carbonate, for instance, sodium hydroxide or other alkali metals, their oxides or hydroxides.

In accordance with the preferred practice, the treatment is as follows:

The oil is mixed and heated with sodium carbonate. The temperature is desirably between about 150° C. and 350° C., preferably from 200° C. to 300° C., and the treatment time preferably at least one-half hour. In most cases temperatures between 260° C. and 300° C. are best.

The sodium carbonate is preferably added to the oil after some pre-heating, for instance, between about 160° C. and 220° C., the treating agent being added in the form of a fine powder. Moreover, I have found that the tendency to foam, which occurs at least with some oils, may be quite

effectively controlled if the quantity of treating agent to be added to a batch is divided into a number of parts, for instance, ten parts, and the several parts are added successively, each one after the foam of any preceding part has subsided. The tendency to foam is usually more pronounced with the initial additions of the treating agent, the tendency markedly diminishing with later additions.

During the heating, carbon dioxide is given off, and for most purposes the preferred practice contemplates treatment conditions utilizing the carbon dioxide to exclude air from contacting the surface of the batch. By way of example, the treatment may be effected in any suitable vessel having a relatively small outlet open to atmosphere. When the conditions are properly controlled, it will be found that a protective layer of carbon dioxide can be maintained on the surface of the batch, thereby substantially completely shielding the batch from contact with the air.

Exclusion of the air prevents oxidation and, still further, is of considerable importance in avoiding discoloration of the soap being produced. Indirect heating and evenness of heating are also important in preventing discoloration or darkening of the soap.

The use of sodium or other carbonates for the purposes above mentioned is further desirable since release of the carbon dioxide throughout the reaction mass serves to agitate the mass, thereby accelerating and facilitating the saponification. Additional agitation, such as mechanical agitation, may be employed, if desired, to further accelerate and facilitate the saponification.

Under some conditions and for some purposes, variations in the above process may be adopted. For instance, superatmospheric pressure or subatmospheric pressure may be employed and reaction vessels of other types may also be used, including an open vessel in which the air is not excluded to the degree contemplated in the preferred practice, as above described.

Carbon dioxide (in addition to that developed by the reaction) may be introduced either by bubbling the same through the reaction mass or by delivering the gas to the surface of the batch as an aid in blanketing the surface. Moreover, for certain purposes other inert gases, as for instance, nitrogen, may be employed either by bubbling through the mass or by blanketing the surface.

Glass lined reaction vessels are desirably used and in the event of use of vacuum, it will be found that a glass lined autoclave constitutes a highly satisfactory reaction vessel. Where reduced pressure is used, it may, for example, be taken down to 400 mm. or 100 mm. of mercury or even down to about 10 to 12 mm.

With further reference to the treatment conditions, it is mentioned that most commercially available oils contain at least some water, and further than (unless anhydrous sodium carbonate is employed) the water of crystallization of the sodium carbonate will be present in the reaction mass. I have found that production of solid soap can only be achieved if the treatment temperature and time be such as to drive off most or substantially all of the water present or formed. In most cases it will be found that a temperature of 200° C. or higher, and a treatment time of at least thirty minutes, is advisable for the purposes just mentioned. Increase of temperature and/or time increases the hardness of the soap product.

The quantity of sodium carbonate used should preferably aproximate that required for substantially complete saponification of the particular oil being treated. Some leeway may be permissible as, for instance, when producing superfatted or alkaline soaps, but, in general, I prefer adhering fairly close to neutrality.

It will be understood, of course, that the exact quantity of sodium carbonate required in accordance with the foregoing will vary, depending upon the particular fatty oil being treated. In other words, some fatty oils require more reagents than others, to effect complete saponification.

The quantity of sodium carbonate required in any particular instance may be determined by experimentation or may be calculated from the saponification number or value of the particular oil (the number of milligrams of potassium hydroxide required to completely saponify one gram of oil).

In all of the following examples anhydrous sodium carbonate was used.

Example 1

300 parts of linseed oil were heated with 70 parts of sodium carbonate to a temperature of 250° C. and for about 4½ hours.

The product was a soft solid.

Example 2

300 parts of linseed oil were heated with 70 parts of sodium carbonate, the temperature being raised over a period of time up to about 270° C. The reaction vessel was then placed under vacuum and the temperature further raised to 300° C. Upon cooling, the product was of a brownish color and very hard.

Example 3

300 parts of fish oil fatty acids were heated with 74 parts of sodium carbonate, the temperature being gradually raised under vacuum up to about 280° C. The reaction required about 3½ hours, and when poured out the product was very hard and of dark color, yielding a very good lather.

The starting material may contain certain other ingredients in addition to fatty oils. Thus, for example, for many purposes it may be desirable to incorporate rosin in accordance with the disclosure of my copending application above mentioned. Some examples of the use of sodium carbonate where rosin is also present are as follows:

Example 4

250 parts of oleic acid, 75 parts of rosin and 74 parts of sodium carbonate were heated together to a temperature of 280° C. and for a period of 5 hours. The product was very hard.

Example 5

1400 parts of sunflower oil, 150 parts of rosin and 350 parts of sodium carbonate were heated together at a temperature of 290° C. for 5 hours at atmospheric pressure.

The product was a brownish hard solid.

Example 6

300 parts of cod liver oil, 90 parts of rosin and 85 parts of sodium carbonate were heated together under vacuum at 260° C. for 3 hours. The

product was quite hard and the odor was good, even after standing for a number of months.

Example 7

250 parts of cod liver oil, 100 parts of rosin and 102 parts of potassium carbonate were heated together under vacuum, the temperature being maintained at about 250° C. for 1¾ hours and at 270° C. for 2 hours. The product had a light color and was capable of forming a good lather.

Example 8

375 parts of fish oil, 125 parts of rosin were initially heated up to about 200° C. and then small portions of sodium carbonate and sodium hydroxide mixed together were slowly added, the total quantity of these two materials being 93 parts of sodium carbonate and 10 parts of sodium hydroxide. The temperature was raised to about 280° C. and the batch maintained at that temperature for about four hours under vacuum, although it appeared that the reaction was completed in about two and a half or three hours. The product contained about .56% of free alkali.

Although various of the oils mentioned in the foregoing examples are capable of producing good quality soaps, in addition other oils will also produce good soaps, such for example as corn oil and cotton seed oil, both of which are readily available and inexpensive.

When ground or powderized, the soaps of this invention yield first class washing powders.

I claim:

1. The process for producing a solid soap from materials of the group consisting of fatty oils and fats, which process comprises heating a batch of fatty material in the presence of substantially anhydrous sodium carbonate (substantially free of crystal water) to a temperature of between 150° C. and 350° C., the sodium carbonate being present in an amount at least sufficient to effect approximately complete saponification of said fatty material, and the heating being effected under conditions maintaining the released carbon dioxide in intimate contact with the surface of the batch.

2. A process in accordance with claim 1 in which the treatment time is at least one-half hour.

3. The process for producing a solid soap from materials of the group consisting of fatty oils and fats, which process comprises heating a batch of fatty material in the presence of a substantially anhydrous carbonate to a temperature of between 150° C. and 350° C. for a period of time of at least one-half hour, in a manner to ensure substantially anyhdrous conditions the carbonate being present in an amount sufficient to effect approximately complete saponification of said fatty material, the cation of said carbonate being a member of the class which consists of the ions of the alkali metals and of ammonium.

4. A process in accordance with claim 3 in which the temperature is maintained between about 260 C. and 300° C.

5. A process in accordance with claim 3 in which said carbonate is sodium carbonate.

6. A process in accordance with claim 3 in which said carbonate is potassium carbonate.

7. A process in accordance with claim 3 in which said carbonate is ammonium carbonate.

8. A process in accordance with claim 3 in which said fatty oil is a drying oil.

9. A process in accordance with claim 3 in which said fatty oil is a semi-drying oil.

10. A process in accordance with claim 3 in which said fatty oil is a non-drying oil.

11. A process in accordance with claim 3 in which said fatty oil is fish oil.

12. A process in accordance with claim 3 in which said fatty oil is corn oil.

13. A process in accordance with claim 3 in which said fatty oil is cottonseed oil.

14. A process in accordance with claim 3 in which said alkali metal carbonate is sodium carbonate, and further in which additional sodium hydroxide is also present during the reaction.

15. The process for producing a solid soap from fatty oils, which process comprises heating a batch of fatty oil in the presence of a substantially anhydrous carbonate to a temperature of between 150° C. and 350° C. for a period of time of at least one-half hour, in a manner to ensure substantially anhydrous conditions the carbonate being present in an amount sufficient to effect approximately complete saponification of said oil, the cation of said carbonate being a member of the class which consists of the ions of alkali metals and of ammonium.

16. A process in accordance with claim 15 and further in which an additional alkali metal hydroxide is also present during the reaction.

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