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# PROCESS FOR PRODUCING ALUMINUM SOAPS

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This invention relates to an improved process for producing aluminum soaps. More particularly the invention relates to a process for producing aluminum soaps free from or containing only small predetermined controlled amounts of aluminum hydroxide and/or free fatty acid.

Pure aluminum di-soaps ($AlOHR_2$) disperse readily in non-polar liquids and rapidly form clear, stable, strong gels. However, if the soap is contaminated by aluminum hydroxide [$Al(OH)_3$], the solubility of the soap is reduced and the gels formed are relatively weak, cloudy and unstable, the reduction in solubility and the weakness, cloudiness and instability of the gel being determined by the amount of the contaminating aluminum hydroxide associated with the soap. If free fatty acid HR is present in the soap, it peptizes solutions of the soap in non-aqueous or oily solvents and adversely affects the gel strength, to an extent corresponding to the amount of free fatty acid in the soap. If both aluminum hydroxide and free fatty acid exist in the soap, the adverse effects of both contaminants reduce the desirability of the soaps, at least for many purposes.

In the usual, known methods for making aluminum soaps, an aluminum salt solution is added to an aqueous sodium soap solution. In those known methods, the initial excess alkali to sodium hydroxide in the sodium soap solution results in complex interactions which influence both the composition of the product soap and its particle size and agglomeration characteristics. The aluminum soaps not only contain aluminum hydroxide and free fatty acid but the proportions of those contaminants in the soap cannot be controlled and predetermined but varies from batch to batch. The contaminated soaps dissolve relatively slowly and the gels formed are weak, cloudy and unstable.

In most mases, pure aluminum di-soaps are desired, although the presence of small, predetermined and controlled amounts of aluminum hydroxide and/or free fatty acid may not be objectionable in some cases.

In the known method, the practice has been to add the aluminum salt solution to the sodium soap solution (which may or may not contain excess alkali) until a "strike" occurs, i.e., until the latex formed during the addition separates into a floating precipitate and a clear subnatant solution. Under those conditions, the particle size of the aluminum soap cannot be directly controlled and is, rather, dependent on the composition of the soap. The composition is controlled by the excess alkali in the sodium soap solution. Thus, if there is no excess alkali, a "tri-soap" is formed which is self-gelled and agglomerated by the fatty acid present; 50% excess alkali yields a "di-soap" the properties and composition of which varies for no apparent reason, and 200% excess alkali yields a "mono-soap" of high melting point, limited solubility and marked syneretic properties.

One object of this invention is to produce pure aluminum di-soaps or mixtures of aluminum di-soaps substantially free from both aluminum hydroxide [$Al(OH)_3$] and free fatty acid (HR).



Another object is to prepare aluminum soaps which contain a predetermined controlled amount of aluminum hydroxide but which do not contain free fatty acid.

A further object is to prepare aluminum soaps which contain a predetermined controlled amount of free fatty acid but which are free from aluminum hydroxide.

An additional object is to prepare aluminum soaps (di-soaps) containing both aluminum hydroxide and free fatty acid, but in controlled predetermined amounts.

Another object is to provide a method of making aluminum soaps of predetermined, controlled particle size and agglomerating characteristics.

An additional object is to provide a method of reliably and reproducibly producing aluminum soaps of desired particle size and agglomerating characteristics.

These and other objects are accomplished by this invention in accordance with which the aluminum salt solution is added to the sodium soap solution in the usual way, and the addition is interrupted at predetermined stages of precipitation or latex formation but before the "strike" occurs and the floating precipitate occurs. The "strike" generally occurs after the neutralization point of the soap solution has been passed and an amount of aluminum salt solution chemically equivalent to the sodium soap solution has been run in. In the present method, the addition of the aluminum salt solution may be terminated at any stage in the process prior to the "strike."

The composition and properties of the aluminum soap produced by the present method depend on the stage at which addition of the aluminum salt solution is stopped. If the addition is interrupted at a very early stage (*a*) it is possible to separate substantially pure aluminum hydroxide from the reaction solution by filtration through a fine-pore filter. If the interruption takes place at a later stage (*b*) in the process, for instance during the early latex stage, the precipitate filtered off is the aluminum di-soap containing aluminum hydroxide as contaminant. When the addition of the aluminum salt solution is interrupted at a still later stage (*c*) but before the "strike," the precipitate is the di-soap containing both aluminum hydroxide and free fatty acid.

Thus, the addition of the aluminum salt solution is interrupted at one stage, early in the process, to separate aluminum hydroxide from the solution of the soap which is then obtained free of the hydroxide, at an earlier stage to obtain a so-called "mono-soap" which is, however, a two-component [$AlOHR_2$ and $Al(OH)_3$] soap instead of the three-component

$$[AlOHR_2+Al(OH)_3+HR]$$

soap usually obtained.

By fixing the stage at which addition of the aluminum salt solution is interrupted in advance, the composition of the product separated from the reaction mass can be predetermined.

After interrupting the addition of the aluminum salt solution and removing the intermediate product by filtration, additional amounts of the aluminum salt solution may be added to the filtrate. This addition can be continued until the "strike" occurs, or it may be interrupted at any stage. Continuing the addition up to the "strike" results in the production of:

(*a*) A "di-soap" containing less of the contaminating $Al(OH)_3$ than of free fatty acid HR;

(*b*) A "di-soap" containing free fatty acid (HR) only—i.e., a two-component soap.

If, in the addition of aluminum salt solution to the filtrate obtained by removing the primary product formed,

the addition is interrupted before the "strike" occurs, a pure aluminum soap ($AlOHR_2$) is formed.

Two different excess alkali ratios may be used to produce two soaps, corresponding with respect to component content but having different particle properties.

It is also within the scope of the invention, to use one or two different excess alkali ratios to produce two soaps of identical R:Al molar ratio but having different component properties. For example, there may be produced two soaps both having an R:Al ratio of 2:1, but one of which is the di-soap $AlOHR_2$ and the other of which is a soap comprising the three components

$$Al(OH)_3+10AlOHR_2+2HR$$

The two soaps may have the same or different particle (crystallite) sizes or size distributions.

It is possible, by the present method, to provide soaps having a variety of predetermined compositions each having a specific, predetermined particle size.

In carrying out the method, the addition of the aluminum salt solution to the sodium soap solution may be interrupted more than once, to effect fractionation of the aluminum soap being formed into several fractions. The following fractions may be obtained in that manner:

(A) Component fractions:
- (*a*) $Al(OH)_3$ or $AlOHR_2$ or HR (one component take-out)
- (*b*) $Al(OH)_3$ and $AlOHR_2$ or $AlOHR_2$ and HR (two component take-out)
- (*c*) Three component take-out

(B) R/Al=*n* fractions:
- (*a*) $n<1$ "sub-mono" soaps
- (*b*) $n=1$ "mono-soaps" $\frac{1}{2}Al(OH)_3+\frac{1}{2}AlOHR_2$
- (*c*) $n=1+(1$ to $1.5)$ "mono-soaps"

$$Al(OH)_{3-n}R_n$$

  - (1) 2 phase or 1 phase; two components
  - (2) 1, 2 or 3 phase; three components
- (*d*) $n=1.5$ to 2.5 "di-soap" $Al(OH)_{3-n}R_n$

$$(n<2)$$

$$AlOHR_2+n2HR(n<2)$$

  - (1) 2 component, no HR present
  - (2) 2 component, HR present
  - (3) 3 component
- (*e*) $n=2.0$ "di-soap"
  - (1) 3 component, 2 phase
  - (2) 3 component, 1 phase
- (*f*) $n=2.0$ "di-soap"; 1 component, 1 phase
- (*g*) $n=2.5$ and up; $AlOHR_2+n2HR$ "Tri-soap"
  - 3.2 component; 3,2, 1 phase
  - 3.2 component; 3,2 phase
  - 3.2 component; 1 phase
- (*h*) $n=3.0$ "Tri-soap"
  - (1) $AlOHR_2+HR$
  - (2) $AlOHR_2+HR$
- (*i*) $n>2.0$ "Supra-di" soap
- (*j*) $n>3.0$ "Supra-tri" soap

} as *g* and *h*

These various fractions comprising soap or mixed soaps, the soap plus aluminum hydroxide, the soap plus fatty acid, the soap plus alumium hydroxide and fatty acid, or aluminum hydroxide can be obtained by interrupting addition of the aluminum salt solution at stages determined experimentally and thereafter the products can be reliably reproduced as desired.

In the tabulation of fractions given "take-out" refers to the product removed from the reaction solution by filtration; *n* represents the degree of substitution of the soap, i.e., proportion of aluminum in the fatty acid.

The addition of an excess of $Al^{+++}$ to any of the filtrates remaining after removal of the fractions results in production of a pure di-soap.

The results obtained by utilized the present method are explainable, theoretically, as follows: An alkaline



sodium soap solution contains $OH^-$ and $R^-$ anions and the addition of $Al^{+++}$ thereto can yield either (*a*) $Al(OH)_3$ or (*b*) $AlOHR_2$, (*a*) being favored at higher $OH^-$ concentrations, (*b*) at lower $OH^-$ concentrations; with the result that as $Al^{+++}$ is added to the solution $Al(OH)_3$ forms first and $AlOHR_2$ forms only as the addition is continued, making it possible, by interrupting the addition, to remove $Al(OH)_3$ as an intermediate precipitate; and as the addition of $Al^{+++}$ to the original solution, or to an aliquot thereof obtained as filtrate from an "interruption take-out" continues, the decreasing alkalinity favors formation of $AlOHR_2$ while the acidity becomes sufficient for the resolution of $Al(OH)_3$, producing internal $Al^{+++}$ which reprecipitates as $AlOHR_2$, the addition of still further $Al^{+++}$ solution leading to acid hydrolysis of $AlOHR_2$, with formation of free acid, HR.

The following examples illustrate specific embodiments of the invention, it being understood that these examples are not intended as limitative:

*Example I*

Fifty pounds of propionic acid were saponified with 50% excess sodium hydroxide. The solution was split into two equal portions. One portion of the solution was "struck" by adding 7% alum solution to it, and the floating precipitate consisting of the aluminum propionate "mono-soap" was recovered.

To the second portion there was added only 75% of the alum solution required to effect a "strike." Aluminum propionate "sub-mono" soap was recovered.

*Example II*

Fifty pounds of valeric acid were saponified with 50% excess sodium hydroxide and the solution was divided into two equal portions. A 7% alum solution in an amount equal to 75% of the amount required for a "strike" was added to one portion of the sodium soap solution. Aluminum valerate "mono-soap" was recovered by filtration of the solution.

As a comparison, 7% alum solution was added to the remaining portion of the sodium soap solution until a "strike" occurred. The floating precipitate consisted of the aluminum propionate "di-soap."

*Example III*

Fifty pounds of caproic acid were saponified with 50% excess sodium hydroxide and the solution was divided into two equal portions. One portion was "struck" by adding 7% alum solution thereto. The "struck" soap was the aluminum caproate "di-soap."

A 7% alum solution was added to the remaining portion of the sodium solution but the addition was interrupted when the amount of the alum solution mixed with the soap solution was only 75% of that required for "strike." The product recovered by filtration of the solution was the aluminum caproate "mono-soap."

*Example IV*

The procedure of Examples I–III was repeated using, in the respective runs, the following acids:

Caprylic
Isovaleric
Isocaproic
2-ethylhexanoic
Isocaprylic

The soaps recovered by filtration after adding to the sodium soap solution 75% of the 7% alum solution required for "strike" were, respectively, the "mono-soaps":

Aluminum capryloate
Aluminum isovalerate
Aluminum isocaproate
Aluminum 2-ethylhexanoate
Aluminum isocapryloate

The "struck" soaps were the corresponding "di-soaps."

*Example V*

The procedure of Examples I–IV was repeated, using in the respective runs, lauric and isobutyric acids. The soaps recovered by filtration after the addition to the sodium soap solution of 75% of 7% alum solution required for "strike" were, respectively, the "mono-soaps" aluminum laurate and aluminum isobutyrate. Those "mono-soaps" were free from acid, whereas the "struck" soaps obtained by adding sufficient 7% alum solution to the soap solution to effect "strike" were di-soaps contaminated with free lauric acid isobutyric acid, respectively.

*Example VI*

Five hundred pounds of 2-ethylhexanoic acid were saponified with a 32.5% excess of 96% sodium hydroxide in 40% solution. The sodium soap solution was divided into four equal portions (A–D), to each of which there was added an aqueous 10% alum solution.

*Portion A.*—The alum solution was added until "strike" took place. A three-component soap having an R/Al ratio of 2.28 determined by ignition analysis for Al, was recovered.

*Portion B.*—Only 75% of the alum solution required for "strike" was added. The precipitate recovered by filtration had an R/Al ratio of 1.83. Alum solution of 10% concentration was added to the filtrate to effect "strike." The "struck" soap had an R/Al ratio of 2.45.

*Portion C.*—The alum solution was added in an amount equal to one-half that required for "strike." The precipitate recovered by filtration had an R/Al ratio of 1.24. The 10% alum solution was added to the filtrate in an amount equal to one-half the amount added to Portion C of the soap solution. The precipitate filtered off (a two-component soap) had an R/Al ratio of 2.27. The second filtrate was "struck" by addition of aqueous 10% alum solution thereto. The two-component soap recovered had an R/Al ratio of 2.56.

*Portion D.*—The 10% aqueous alum solution was added in an amount equal to 25% that required for "strike." The precipitate recovered by filtration was pure aluminum hydroxide ($Al(OH)_3$.

(*a*) The 10% alum solution was added to filtrate A in an amount equal to 25% of that required for strike. A two-component soap having an R/Al ratio of 2.24 precipitated and was removed by filtration.

(*b*) The addition of the alum solution in an amount equal to 25% of that required for strike to the filtrate B resulted in the precipitation of a two-component soap having an R/Al ratio of 2.45.

The soaps obtained differed in solubility and in particle size.

The aluminum salt solution is added to the sodium salt solution in a constant stream. The time required to form "take-out" precipitate depends on the rate of addition, which can be ascertained and fixed to insure reproducible production of a precipitate of the desired composition in a given time period.

Any fatty acid the sodium salt of which reacts with the aluminum salt by a process of double decomposition to form the corresponding aluminum soap may be used. Similarly, any of the water-soluble aluminum salts commonly reacted with sodium soaps to produce the aluminum soap may be employed.



In carrying out the invention, the concentration of the aluminum salt solution being fixed and being generally between 5% and 10%, the amount thereof required to be added to the sodium soap solution to effect "strike" can be readily determined. From that, it can be determined, very readily, the proportion of the solution to be added before the "strike" takes place to form a precipitate of the desired composition. The amount of the 5% to 10% aqueous aluminum salt solution added to precipitate the desired product may be 10% to 90% of the amount thereof required to effect the "strike." Generally, the amount of aluminum salt solution mixed with the sodium soap solution before the addition is interrupted is between 25% and 75%.

The addition of the aluminum salt solution to the sodium soap solution may be made under the conditions usual in aluminum soap-making and may be made at room temperature.

In practicing the invention as illustrated in the examples given, various changes may be made with respect to the sodium soap and aluminum salt used and in other conditions.

Among the changes that may be made are the use of other alkali soaps such as lithium, potassium, rubidium or cesium soaps, the nature of the fatty acid used being such as to render the soap solutions of the appropriate concentration non-isotropic. The soaps of these other alkali metals are less prone to form thick phases at practical reaction concentrations than are the sodium soaps.

It will also be apparent that, in addition to the acids exemplified herein, there may be used, in making the alkali soap, any other straight-chain or branched-chain saturated or unsaturated fatty acid, or any acid containing alicyclic, benzenoid or heterocyclic groups capable of yielding an aluminum soap.

Since these various changes may be made without departing from the spirit and scope of the invention, it is to be understood that it is not intended to limit the invention except as defined in the appended claims.

What is claimed is:

1. A process for making aluminum soaps which comprises adding an aqueous aluminum salt solution to aqueous sodium soap in incremental amounts each predetermined to produce, by reaction between the aluminum salt and sodium soap, an aluminum soap of predetermined composition which precipitates and recovering the precipitate after each of the aluminum salt solution increments has been added.

2. A process for making aluminum soaps which comprises adding an aqueous aluminum salt solution to aqueous sodium soap and until free aluminum hydroxide precipitates, filtering off the aluminum hydroxide, adding sufficient of the aluminum salt solution to the filtrate to form an aluminum soap substantially free of aluminum hydroxide which soap precipitates, and recovering the aluminum soap.

**References Cited** in the file of this patent

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