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DOUBLE DECOMPOSITION SOAP PROCESS

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8 Claims. (Cl. 260-414)

This invention relates to double decomposition preparation of heavy metal soaps.

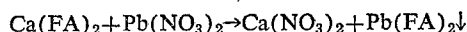
Metallic soaps are commonly prepared by a double decomposition process involving formation of a sodium soap followed by the replacement of the sodium cation to form a heavy metal insoluble soap.

While a double decomposition process as indicated above is suitable for the preparation of certain water insoluble metallic soaps, it has not been found to be suitable for the preparation of certain other heavy metal soaps.

It is an object of this invention to prepare heavy metal soaps by a double decomposition process.

It is a further object of this invention to prepare pure heavy metal soaps by means of a double decomposition process wherein the product may be easily washed free of undesirable contaminants.

I have now discovered that it is possible to produce heavy metal soaps by a double decomposition process comprising slurring an insoluble calcium soap of a fatty acid in water and then adding a water soluble salt having an anion which has the ability to form water soluble calcium salts and preferably is selected from the group consisting of nitrate anion, chloride anion, iodide anion, bromide anion and acetate anion, and a metallic cation of a metal selected from groups III, IV, V, VI-A, and VIII cations. The fatty acid is preferably a fatty acid having a carbon chain length of from 9 to 22 atoms and more specifically a fatty acid selected from the group consisting of azelaic acid, stearic acid, lauric acid, myristic acid, hydroxy-stearic acid, palmitic acid, erucic acid, pelargonic acid, eleostearic acid, oleic acid, and linolenic acid. The cation of the salt is preferably a cation selected from the group consisting of cations of the rare earth metals, aluminum, titanium, zirconium, tin, lead, vanadium, antimony, chromium and iron. It should be understood that in order to maintain certain of the fatty acids and more specifically eleostearic acid, oleic acid and linolenic acid in a solid state, it is necessary to cool these acids below room temperature. The reaction may be illustrated as follows, wherein FA represents a fatty acid radical:



In general, stoichiometric quantities of reactants are preferred although quantities of from 1.0 to 1.1 equivalents of water soluble salt may be employed per equivalent of calcium soap.

It has been found that reaction of the calcium soap with a water soluble heavy metal salt occurs under the most favorable circumstances where a dispersion of the calcium soap has been obtained by passing the soap through a homogenization unit. The homogenization operation appears to have the effect of exposing sufficient surface area of the calcium soap to insure reaction of the water soluble heavy metal salt. The intimate mixture which can be had with the homogenizer also results in the reduction of the amount of water which must be used in the slurry. The term homogenizer as employed herein means homogenization apparatus well known in the chemical industry and of a type wherein the material to be homogenized is passed through a very small opening under high pressure. It is preferred to force the material to be homogenized through the homogenizer against sufficient back pressure to reduce the particle size suffi-

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ciently to disperse the reaction product. This can be done by one or a plurality of passes of the material through the homogenizer.

The following specific examples of preparations prepared according to the process of this invention are given for purposes of illustration and are not to be considered as limiting the spirit and scope thereof:

Example I

500 grams of calcium stearate were dispersed in 2000 ml. of water by passing through a Manton-Gaulin homogenizer. 180 grams of FeCl_3 were then dissolved in 500 ml. of water. The temperature of the calcium stearate slurry was raised to about 150° F. and the FeCl_3 solution at room temperature was added over a period of 30 minutes with moderate agitation. The slurry mixture became thick as the FeCl_3 was added, but eventually thinned out on completion of the addition. Agitation was continued for about 30 to 40 minutes after completion of FeCl_3 addition. The product was then filtered on a water Buchner filter, the filtration being quite rapid. The product, which was ferric stearate, was dried at 140° F.

Example II

150 grams of calcium stearate were slurried in 500 ml. of water by means of a Manton-Gaulin homogenizer. Two or three drops of Stearox A.J. were employed in order to wet the calcium stearate. 29 grams of ZrCl_4 were added with rapid agitation over a 10 to 12 minute period. The temperature of the slurry mixture rose from 79° F. to 102° F. The resultant product, which was zirconium stearate, was filtered and washed with about 500 ml. of water.

Example III

500 grams of calcium stearate were slurried in 2000 ml. of water by means of passage through a Manton-Gaulin homogenizer. 142 grams of $\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$ were dissolved in 500 ml. of water. Calcium stearate slurry was then heated to about 160° F. and the $\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$ solution added thereto with good agitation. Agitation of the slurry was continued for approximately thirty minutes after completion of the $\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$ addition. The product, which was aluminum stearate, was filtered on a Buchner funnel, washed with about 400 cc. of water and dried at 140° F.

Example IV

2500 ml. of water, 111 grams of lime, 6 grams of Stearox A.J. and 658 grams of stearic acid were mixed with agitation. 710 grams of the resultant calcium stearate slurry were weighed into a 1500 ml. beaker. 38 grams SbCl_3 were then dissolved in 150 ml. of water. The SbCl_3 solution was then added to the calcium stearate slurry. A temperature rise of from 86° F. to 99° F. was observed. Agitation was continued for one hour after completion of the addition. The resultant product, which was antimony stearate, was filtered and dried at 140° F.

Example V

2500 ml. of water, 111 grams of lime, 6 grams of Stearox A.J., and 658 grams of stearic acid were mixed with agitation. 710 grams of the resultant calcium stearate slurry were weighed into a 1500 ml. beaker. 29 grams of ZrCl_4 were then added to slurry. A temperature rise of 86° F. to 108° F. was observed. Agitation was continued for about one hour after completion of the addition of ZrCl_4 . The product, which was zirconium stearate, was filtered, washed with 500 ml. of water and dried at about 140° F.

Example VI

2500 ml. of water, 111 grams of lime, 6 grams of

Stearox A.J., and 658 grams of stearic acid were mixed with agitation. 710 grams of the resultant calcium stearate slurry were then weighed into a 1500 ml. beaker. 44 grams of SnCl_4 were then dissolved in 100 ml. of water. The SnCl_4 solution was added to the calcium stearate slurry; a temperature rise of from 86° F. to 92° F. being noted. The slurry was agitated for approximately one hour after completion of the addition of the SnCl_4 solution. The resultant product, which was tin stearate, was then filtered, washed with 500 ml. of water and dried at 140° F.

Example VII

2500 ml. of water, 111 grams of lime, 6 grams of Stearox A.J. and 658 grams of stearic acid were mixed with agitation. 710 grams of the resultant calcium stearate slurry were added to a 1500 ml. beaker. 30 grams of SbCl_5 were then added to the slurry; a temperature rise of from 86° F. to 113° F. being noted. The slurry was agitated for approximately one hour after completion of the addition of the SbCl_5 . The resultant product, which was antimony stearate, was filtered, washed with 500 ml. of water and dried.

Example VIII

500 grams of calcium stearate were dispersed in 200 ml. of water. 238 grams of ferric nitrate were dissolved in 500 ml. of water. The calcium stearate dispersion was then heated to about 140° F. and the ferric nitrate solution added thereto with agitation. The agitation was continued for a period of about thirty minutes, during which time the reaction went to completion. The product, which was ferric stearate, was filtered on a Buchner funnel and air dried at 140° F.

The process of this invention results in a superior heavy metal soap product. It is believed that this product is superior to the heavy metal soap products produced by the sodium soap reactant double decomposition process of the prior art in that calcium soap is not hydrolyzed to the extent that the sodium soap is by the presence of certain salts of heavy metals. The fact that hydrolysis is not produced in a calcium soap results in a product being produced which may be easily washed free of the resultant soluble calcium salts without being hindered by the presence of large amounts of metal hydrate and free fatty acid.

What I claim is:

1. A process for the preparation of a metallic soap comprising dispersing in water a calcium soap of a fatty acid having from 9 to 22 carbon atoms and then adding

a water soluble salt having an anion which has the ability to form water soluble salts with calcium, and a metallic cation selected from cations of the group III, IV, V, VI-A and VIII elements.

2. The method of claim 1 wherein said anion is an anion selected from the group consisting of nitrate anion, chloride anion, iodide anion, bromide anion and acetate anion.

3. The process of claim 1 wherein said fatty acid is a fatty acid selected from the group consisting of azelaic acid, stearic acid, lauric acid, myristic acid, hydroxystearic acid, palmitic acid, erucic acid, pelargonic acid, eleostearic acid, oleic acid and linolenic acid.

4. The method of claim 1 wherein the said dispersion is carried out by passing said calcium soap through a homogenizer.

5. The process for the preparation of a metallic soap comprising dispersing in water a calcium soap of a fatty acid having a carbon chain length from 9 to 22 atoms, and then adding a salt having an anion selected from the group consisting of nitrate, chloride, iodide, bromide and acetate, and a cation selected from the group consisting of cations of rare earth metals, aluminum, titanium, zirconium, tin, lead, vanadium, antimony, chromium and iron, washing free the resultant soluble calcium salt and recovering the soap of said cation.

6. The method of claim 5 wherein said fatty acid is a fatty acid selected from the group consisting of azelaic acid, stearic acid, lauric acid, myristic acid, hydroxystearic acid, palmitic acid, erucic acid, pelargonic acid, eleostearic acid, oleic acid and linolenic acid.

7. The method of claim 5 wherein said slurring is carried on by passing said calcium soap through a homogenizer.

8. A process for preparing a metal soap comprising dispersing in water a calcium soap of a fatty acid having from 9 to 22 carbon atoms in the molecule, adding to the resulting dispersion a salt having as its anion one which will form a highly soluble calcium salt and having as its cation one selected from cations from rare earth metals, aluminum, titanium, zirconium, tin, lead, vanadium, antimony, chromium and iron and separating the liquid phase of the reaction product whereby to produce a solid, insoluble metallic soap.

References Cited in the file of this patent

UNITED STATES PATENTS

2,890,232 Rogers et al. _____ June 9, 1959

UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,078,288

February 19, 1963

Harold M. Olson

It is hereby certified that error appears in the above numbered patent requiring correction and that the said Letters Patent should read as corrected below.

Column 4, line 3, strike out "the" same line, for "group" read -- groups --; line 40, for "wil" read -- will --; line 41, for "from" read -- of --.

Signed and sealed this 3rd day of September 1963.

(SEAL)
Attest:

ERNEST W. SWIDER
Attesting Officer

DAVID L. LADD
Commissioner of Patents