

Improvements in or relating to
The Production of Sodium Alpha Olefin Sulphonate

This invention relates to a method of manufacturing sodium alpha olefin sulphonate from fatty oil and sodium alpha olefin sulphonate obtained thereby.

5 Our co-pending patent application 8621277 is a divisional to this present application and is directed to a method of manufacturing a laundry detergent product containing sodium alpha olefin sulphonate obtained from fatty oil.

10 When ordinary soap is used in hard water, the calcium and magnesium ions in the hard water react

with the soap and form insoluble calcium and magnesium soaps, which have no detergency and no lathering property. Thus, a part of the soap is wasted when used in hard water. In sea water, ordinary laundry
5 soaps cannot be used at all. The amount of wastage depends on the hardness of water. Assuming the weighted average hardness of water as 200ppm calcium carbonate, and considering the weighted average use of laundry soap in India, one-third of the soap used is
10 wasted outright, and the remaining two-thirds does not function efficiently. Thus, 100,000 tons per annum of fatty matter in laundry soap is wasted outright. Reckoning rice bran oil at Rs. 7500 per ton, the annual wastage is of the magnitude of at least Rs.75
15 crore.

The term 'fatty oil' wherever referred to in this specification includes a fatty oil or a mixture of fatty oils such as Rice Bran Oil, Coconut Oil, Palm Oil or Tallow.

20 According to the present invention in one aspect, there is provided a method of manufacturing sodium alpha olefin sulphonate from fatty oil, which method comprises the steps of converting the fatty oil into fatty acid, converting the fatty acid into saturated
25 fatty alcohol, dehydrating the saturated fatty alcohol at an elevated temperature in the presence of a catalyst to remove a molecule of water and form an

olefin having a double bond in the alpha position, and sulphonating, neutralising and hydrolysing the alpha olefin to produce sodium alpha olefin sulphonate.

The fatty oil may be converted into fatty acid by
5 splitting at high pressure and temperature, for example, at about 32 kg/sq cm pressure and 230 °C temperature. The fatty acid may be converted into saturated fatty alcohol by hydrogenation at high pressure and temperature, for example, at about
10 250kg/sq cm and 300 °C temperature. The saturated fatty alcohol is preferably dehydrated at an elevated temperature of between 300 °C and 400 °C and the catalyst may be alumina. The alpha olefin is sulphonated in known manner, for example, with sulphur
15 trioxide.

The sulphonated alpha olefin is neutralised in known manner, for example, with sodium hydroxide. The neutralised sulphonated alpha olefin is hydrolysed in known manner, for example, at a temperature of
20 about 150 °C.

The sodium alpha olefin sulphonate obtained by said method has, preferably, C₁₂ to C₁₈ chainlength and still preferably C₁₆ to C₁₈ chainlength.

The invention further provides sodium alpha
25 olefin sulphonate obtained by the method described in the preceeding paragraphs.

As mentioned previously, our co-pending patent application 8621277 concerns a method of manufacturing a laundry detergent product containing sodium alpha olefin sulphonate obtained from fatty oil, as an active detergent, which method comprises mixing said sodium alpha olefin sulphonate with a detergency builder to form a homogeneous mixture and, if required, further processing said homogeneous mixture to obtain a laundry detergent product which is resistant to hard water and sea water.

The sodium alpha olefin sulphonate used for the manufacture of said laundry detergent product has, preferably, a C₁₂ to C₁₈ chainlength and still preferably a C₁₆ to C₁₈ chainlength.

The detergency builders are, for example, borax, tetrasodium pyrophosphate, potassium pyrophosphate, trisodium phosphate, sodium silicate, sodium carbonate, sodium bicarbonate, sodium tripolyphosphate, polyvinyl pyrrolidone and carboxymethyl cellulose.

The fillers are, for example, sodium sulphate, sodium chloride, talc, china clay and starch.

The additives are, for example, optical brighteners, colouring agents, perfumes, emollients, preservatives, pearlescent agents and/or foam boosters.

Optical brighteners are, for example, "Ranipal"
(R.T.M.)
and "Photine C". Colouring agents are, for example,
phthalocyanine blue and phthalocyanine green.

Perfumes are, for example, jasmine, rose, lavender and
5 citronella. Emollients are, for example,
hydrogenated castor oil and paraffin wax.

Preservatives are, for example, methyl paraben and
propyl paraben. Pearlescent agents are, for example,
ethylene glycol monostearate. Foam boosters are, for
10 example, coconut monoethanolamide and coconut
diethanolamide.

Examples of soap are as follows:

- 1 Soap made from a mixture of Palm Oil,
Coconut Oil and Groundnut Oil.
- 15 2 Soap made from a mixture of Tallow, Coconut
Oil and Groundnut Oil.

Examples of said other detergents are alkyl
benzene sulphonate, sodium lauryl ether sulphate and
sodium lauryl sulphate.

20 The proportion of said sodium olefin sulphate is
preferably 5 to 30% by weight.

The proportion of said other synthetic
detergent(s) and/or soap(s) is up to 10% by weight.

In case the hard water and sea water resistant
25 laundry detergent product is required in the form of
bars, said mixing is carried out at ambient
temperature or at an elevated temperature, for

example, between 50 to 55 °C, preferably, in a mixer such as sigma mixer and the resulting homogeneous mixture is further processed, for example, by milling in a triple roll mill into ribbons and extruding the ribbons into bars through an extrusion plodder. In case said mixing is carried out at elevated temperature the time for carrying out said mixing can be reduced.

In case the hard water and sea water resistant laundry detergent product is required in the form of powder, said mixing is carried out with water preferably, in a crutcher and the resulting homogeneous mixture in the form of a slurry is further processed by spray drying into powder.

In case the hard water and sea water resistant laundry detergent product is required in the form of a clear liquid or a peralescent liquid, said mixing is carried out with water preferably in a mixer.

The advantages of the laundry detergent product containing sodium alpha olefin sulphonate obtained from fatty oil are as follows:-

- 1 possesses excellent detergency in hard water (well water) and sea water;
- 2 easy to extrude;
- 25 3 non-irritant and mild to skin;
- 4 with sodium alpha olefin sulphonate little or no STPP (sodium tripolyphosphate) is required. STPP

causes eutrophication;

- 5 good quality bar can be made without matter
insoluble in water and such bar is not mushy;
- 6 better for dish washing and petroleum stain
5 removal;
- 7 compatible with other laundry detergent products
in any desired proportion;
- 8 completely biodegradable.

10 The invention will now be described further with
reference to the following Examples I to III, these
being merely exemplary of the invention. Examples IV
to XI are directed to subject matter contained in our
co-pending patent application 8621277.

15 EXAMPLE I

Crude palm oil was split in an autoclave using
32kg/sq cm pressure and 230 C temperature.

The split fatty acids were straight distilled in
a distillation apparatus, under reduced pressure of
20 3-4mm.

The distilled fatty acids were hydrogenated in a
high pressure autoclave at 250 kg/sq cm pressure and
300 C temperature using 0.6% copper chromite as
catalyst. The fatty acids got converted into fatty
25 alcohols, at the same the time double bonds were also
saturated.

The crude fatty alcohols were straight distilled in a distillation apparatus, under reduced pressure of 3-4mm.

5 The distilled fatty alcohols were dehydrated in a reactor by a continuous vapour phase dehydration process over a fixed bed of alumina catalyst, at 400 °C temperature and atmospheric pressure. The alcohols lost one molecule of water and were converted into olefins having a double bond in the alpha (terminal) position.

10 The crude alpha olefins were distilled to remove impurities. The yield of distilled alpha olefins was 73% of the starting crude palm oil. The alpha olefins had the following chainlength:

15	C		1.2%
	14		
	C		45.0%
	16		
	C		53.8%
	18		

20 The alpha olefins were sulphonated with sulphur trioxide gas in sulphonating equipment. The sulphonated alpha olefins were neutralised with sodium hydroxide, using excess quantity of sodium hydroxide. The neutralised sulphonated alpha olefins were hydrolysed in an autoclave at a temperature of 150 °C. The yield of sodium alpha olefin sulphonate was 133% of the alpha olefins on 100% basis.

EXAMPLE II

Crude rice bran oil was split in an autoclave using 32 kg/sq cm pressure and 230 °C temperature.

5 The split fatty acids were straight distilled in a distillation apparatus, under reduced pressure of 3-4mm.

The distilled fatty acids were hydrogenated in a high pressure autoclave at 250 kg/ sq cm pressure and 300 °C temperature using 0.6% copper chromite as
10 catalyst. The fatty acids got converted into fatty alcohols, at the same time the double bonds were also saturated.

The crude fatty alcohols were straight distilled in a distillation apparatus, under reduced pressure of
15 3-4mm.

The distilled fatty alcohols were dehydrated in a reactor by a continuous vapour phase dehydration process over a fixed bed of alumina catalyst, at 400 °C temperature and atmospheric pressure. The alcohols
20 lost one molecule of water and were converted into olefins having a double bond in the alpha (terminal) position.

The crude alpha olefins were distilled to remove impurities. The yield of distilled alpha olefins
25 was 66% of the starting rice bran oil. The alpha olefins had the following chainlength:

C		0.3%
14		
C		20.5%
16		
C		79.2%
18		

5 The alpha olefins were sulphonated with sulphur trioxide gas in sulphonating equipment. The sulphonated alpha olefins were neutralised with sodium hydroxide, using excess quantity of sodium hydroxide. The neutralised sulphonated alpha olefins were
10 hydrolysed in an autoclave at a temperature of 150 °C. The yield of sodium alpha olefin sulphonate was 132% of the alpha olefins, on 100% basis.

EXAMPLE III

Crude tallow was split in an autoclave using 32
15 kg/sq cm pressure and 230 °C temperature.

The split fatty acids were straight distilled in a distillation apparatus, under reduced pressure of 3-4mm.

The distilled fatty acids were hydrogenated in a
20 high pressure autoclave at 250 kg/sq cm pressure and 300 °C temperature using 0.6% copper chromite as catalyst. The fatty acids got converted into fatty alcohols, at the same time the double bonds were also saturated.

25 The crude fatty alcohols were straight distilled in a distillation apparatus, under reduced pressure of 3-4mm.

The distilled fatty alcohols were dehydrated in a reactor by a continuous vapour phase dehydration process over a fixed bed of alumina catalyst, at 400 °C temperature and atmospheric pressure. The alcohols
5 lost one molecule of water and were converted into olefins having a double bond in the alpha (terminal) position.

The crude alpha olefins were distilled to remove impurities. The yield of distilled alpha olefins was
10 72% of the starting tallow. The alpha olefins had the following chainlength:

C		2.5%
14		
C		32.5%
16		
C		65.0%
18		

15 The alpha olefins were sulphonated with sulphur trioxide gas in sulphonating equipment. The sulphonated alpha olefins were neutralised with sodium hydroxide, using excess quantity of sodium hydroxide in an autoclave at a temperature of 150 °C. The yield
20 of sodium alpha olefins sulphonate was 133% of the alpha olefins, on 100% basis.

EXAMPLE IV

The following ingredients were taken in the
25 proportion (by weight) indicated:

			<u>Parts</u>
	1	Sodium alpha olefin sulphonate (containing 90% anhydrous sodium alpha olefin sulphonate) ...	25
5	2	Sodium tripolyphosphate ...	25
	3	Sodium bicarbonate ...	25
	4	Sodium carbonate ...	6
	5	Neutral sodium silicate (containing 40% anhydrous neutral sodium silicate) ...	22
10	The ingredients were mechanically mixed in a sigma mixer for 20 to 30 minutes at an elevated temperature of 50 C - 55 C. The mixed dough formed was then milled in a triple roll mill into a ribbon form and the ribbons were extruded in bar form through an extrusion plodder.		
15			

A moisture loss of about 3% occurred during the entire operation. The final composition of the extruded bar was as follows:

	1	Sodium alpha olefin sulphonate (on anhydrous basis) ...	22 - 23%
20	2	Sodium tripolyphosphate ...	25%
	3	Sodium bicarbonate ...	25%
	4	Sodium carbonate ...	6%
	5	Neutral sodium silicate (on anhydrous basis) ...	9%
25	6	Water ...	12 - 13%

EXAMPLE V

The following ingredients were taken in the proportion (by weight) indicated:

			<u>Parts</u>
5	1	Sodium alpha olefin sulphonate (containing 90% anhydrous sodium sodium alpha olefin sulphonate)	... 25
	2	Sodium bicarbonate	... 6
	3	Sodium carbonate	... 6
	4	Neutral sodium silicate (containing 40% anhydrous neutral sodium silicate)	... 25
10			
	5	Sodium sulphate	... 40
	6	Paraffin Wax	... 2
	7	CMC	... 1
	8	Ranipal	... 0.2
15	9	Jasmine	... 0.2

The ingredients were mechanically mixed in a sigma mixer for 20 to 30 minutes at an elevated temperature of 50 C - 55 C. The mixed dough formed was then milled in a triple roll mill into a ribbon form and the ribbons extruded in bar form through an extrusion plodder.

A moisture loss of about 3% occurred during the entire operation. The final composition of the extruded bar was as follows:

25	1	Sodium alpha olefin sulphonate (on anhydrous basis)	... 22-23%
	2	Sodium bicarbonate	... 6%

	3	Sodium carbonate	...	6%
	4	Neutral sodium silicate (on anhydrous basis)	...	10%
	5	Sodium sulphate	...	40%
5	6	Paraffin Wax	...	2%
	7	CMC	...	2%
	8	Ranipal	...	0.2%
	9	Jasmine	...	0.2%
	10	Water	...	12-13%

10 EXAMPLE VI

The following ingredients were taken in the proportion (by weight) indicated:

			<u>Parts</u>
	1	Sodium alpha olefin sulphonate (containing 90% anhydrous sodium alpha olefin sulphonate)	... 18
15	2	Sodium tripolyphosphate	... 15
	3	Sodium bicarbonate	... 25
	4	Sodium carbonate	... 5
	5	Neutral sodium silicate (containing 40% anhydrous neutral sodium silicate)	... 25
20	6	Sodium sulphate	... 16
	7	CMC	... 1
	8	Ranipal	... 0.2
	9	Rose	... 0.2

25 The ingredients were mechanically mixed in a sigma mixer for 20 to 30 minutes at an elevated

temperature of 50° C-55° C. The mixed dough formed was then milled in a triple roll mill into a ribbon form and the ribbons extruded in bar form through an extrusion plodder.

5 A moisture loss of about 3% occurred during the entire operation. The final composition of the extruded bar was as follows:

	1	Sodium alpha olefin (on anhydrous basis)	...	15 - 16%
10	2	Sodium tripolyphosphate	...	15%
	3	Sodium bicarbonate	...	25%
	4	Sodium carbonate	...	5%
	5	Neutral sodium silicate (on anhydrous basis)	...	10%
15	6	Sodium sulphate	...	16%
	7	CMC	...	1%
	8	Ranipal	...	0.2%
	9	Rose	...	0.2%
	10	Water	...	12 - 13%

20 EXAMPLE VII

The following ingredients were taken in the proportion (by weight) indicated:

			<u>Parts</u>
25	1	Sodium alpha olefin sulphonate (containing 90% anhydrous sodium alpha olefin sulphonate)	... 18

2	Sodium soap flakes (containing 90% anhydrous sodium soap)	...	5.5
3	Sodium tripolyphosphate	...	15
4	Sodium bicarbonate	...	20
5	Sodium carbonate	...	6
5	6 Neutral sodium silicate (containing 40% anhydrous neutral sodium silicate)	...	25
7	Sodium sulphate	...	17
8	Photine C	...	0.2
9	Rose	...	0.2

10 The ingredients were mechanically mixed in a sigma mixer for 20 to 30 minutes at an elevated temperature of 50°C-55°C. The mixed dough formed was then milled in a triple roll mill into a ribbon form and the ribbons extruded in bar form through an extrusion plodder.

15 A moisture loss of about 3% occurred during the entire operation. The final composition of the extruded bar was as follows:

1	Sodium alpha olefin sulphonate	...	15 - 16%
20	(on anhydrous basis)		
2	Sodium soap (anhydrous)	...	5%
3	Sodium tripolyphosphate	...	15%
4	Sodium	...	20%
5	Sodium carbonate	...	6%
25	6 Neutral sodium silicate (on anhydrous basis)	...	10%
7	Sodium sulphate	...	16%

8	Photine C	...	0.2%
9	Rose	...	0.2%
10	Water	...	12-13%

EXAMPLE VIII

5 The following ingredients were taken in a crutcher in the proportion (by weight) indicated:

1	Sodium alpha olefin sulphonate	...	60%
	(containing 40% anhydrous sodium alpha olefin sulphonate)		
2	Sodium carbonate	...	15%
10	3 Alkaline sodium silicate	...	32%
	(containing 48% anhydrous alkaline sodium silicate)		
4	Sodium sulphate	...	35%
5	CMC	...	1%
6	Photine C	...	0.2%
15	7 Lavender	...	0.2%
	8 Phthalocyanine blue paste	...	0.2%
	9 Water	...	10%

After mixing, the slurry was spray-dried. The final composition of the spray-dried powder was as

20 follows:-

1	Sodium alpha olefin sulphonate	...	24%
	(on anhydrous basis)		
2	Sodium carbonate	...	15%
3	Alkaline sodium silicate	...	15%
	(on anhydrous basis)		
25	4 Sodium sulphate	...	35%

5	CMC	...	1%	
6	Photine C	...	0.2%	
7	Lavender	...	0.2%	
8	Phthalocyanine blue colour	...	0.05%	
5	9	Water	...	10%

EXAMPLE IX

The following ingredients were taken in a crutcher in the proportion (by weight) indicated:

	1	Sodium alpha olefin sulphonate	...	50%
		(containing 40% anhydrous sodium alpha olefin sulphonate)		
10	2	Sodium tripolyphosphate	...	15%
	3	Sodium carbonate	...	15%
	4	Alkaline sodium silicate	...	32%
		(containing 48% anhydrous alkaline sodium silicate)		
15	5	Sodium sulphate	...	24%
	6	CMC	...	1%
	7	Ranipal	...	0.2%
	8	Jasmine	...	0.2%
	9	Phthalocyanine blue paste	...	0.1%
20	10	Water	...	15%

After mixing, the slurry was spray-dried. The final composition of the spray-dried powder was as follows:-

	1	Sodium alpha olefin sulphonate	...	20%
		(on anhydrous basis)		
25	2	Sodium tripolyphosphate	...	15%

	3	Sodium carbonate	...	15%
	4	Alkaline sodium silicate	...	15%
		(on anhydrous basis)		
	5	Sodium Sulphate	...	24%
	6	CMC	...	1%
5	7	Ranipal	...	0.2%
	8	Jasmine	...	0.2%
	9	Phthalocyanine blue colour	...	0.05%
	10	Water	...	10%

10 EXAMPLE X

The following ingredients were mixed in the proportion (by weight) indicated:

	1	Sodium alpha olefin sulphonate	...	50%
		(containing 40% anhydrous sodium alpha olefin sulphonate)		
	2	Coconut diethanolamide	...	2%
15	3	Sodium chloride	...	2%
	4	Potassium phrophosphate	...	5%
	5	Methyl paraben	...	0.2%
	6	Polyvinyl pyrrolidine	...	0.5%
	7	Ranipal	...	0.1%
20	8	Rose	...	0.2%
	9	Water	...	40%

The final liquid detergent contained 20% sodium alpha olefin sulphonate (on anhydrous basis).

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EXAMPLE XI

The following ingredients were mixed in the proportion (by weight) indicated:

	1	Sodium alpha olefin sulphonate	...	25%
		(containing 40% anhydrous sodium alpha olefin sulphonate		
5	2	Sodium lauryl ether sulphate	...	18%
		(containing 28% anhydrous sodium lauryl ether sulphonate)		
	3	Coconut diethanolamide	...	2%
	4	Ethylene glycol monostearate	...	2%
10	5	Potassium pyrophosphate	...	3%
	6	Sodium chloride	...	1.5%
	7	Polyvinyl pyrrolidone	...	0.5%
	8	Ranipal	...	0.1%
	9	Methyl paraben	...	0.2%
15	10	Rose	...	0.5%
	11	Water	...	47.2%

The pH of the product was adjusted to 7.5 with citric acid.

The final pearlescent liquid detergent contained
 20 10% sodium alpha olefin sulphonate (on anhydrous basis) and 5% sodium lauryl ether sulphate (on anhydrous basis).

CLAIMS

- 1 A method of manufacturing sodium alpha olefin
sulphonate from fatty oil, which method comprises the
steps of converting the fatty oil into fatty acid,
converting the fatty acid into saturated fatty
5 alcohol, dehydrating the saturated fatty alcohol at an
elevated temperature in the presence of a catalyst to
remove a molecule of water and form an olefin having a
double bond in the alpha position, and sulphonating,
neutralising and hydrolysing the alpha
10 olefin to produce sodium alpha olefin sulphonate.
- 2 A method as claimed in claim 1 wherein the
catalyst is alumina.
- 3 A method as claimed in claim 1 wherein the
saturated fatty alcohol is distilled and dehydrated by
15 a continuous phase dehydration process in the presence
of an alumina.
- 4 A method as claimed in any one of claims 1 to 3
wherein the saturated fatty alcohol is dehydrated at a
temperature of between 300^o C and 400^o C.
- 20 5 A method as claimed in any one of claims 1 to 4
wherein the fatty oil is split into fatty acid at
a pressure of the order of 32kg/cm² and a temperature
of the order of 230^o C.



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6 A method as claimed in claim 5 wherein the fatty acid is hydrogenated at a pressure of the order of 250kg/cm² and a temperature of the order of 300° C.

7 A method as claimed in claim 6 wherein the
5 sulphonated alpha olefin is neutralised with sodium hydroxide.

8 A method as claimed in any one of claims 1 to 7 wherein the sodium olefin sulphonate has a chainlength of from C₁₂ to C₁₈.

10 9 A method as claimed in any one of claims 1 to 7 wherein the sodium olefin sulphonate has a chainlength of from C₁₆ to C₁₈.

10 Sodium alpha olefin sulphonate whenever
15 manufactured by a method as claimed in any one of claims 1 to 9.

11 A method of manufacturing sodium alpha olefin sulphonate from fatty oil substantially as herein described particularly with reference to Examples I to III.



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