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[54] **DETERGENT INTERMEDIATE AND PROCESS THEREFOR**

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[51] **Int. Cl.** **C11d 3/02**

[58] **Field of Search** 252/89, 92; 134/7

[56] **References Cited**

UNITED STATES PATENTS

3,329,616	7/1967	Feierstein et al.	252/DIG. 3
1,692,996	11/1928	Richardson	252/DIG. 3

FOREIGN PATENTS OR APPLICATIONS

511,415	3/1955	Canada	252/89

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[57] **ABSTRACT**

A detergent intermediate for use in phosphate-free detergent formulations is provided by absorbing a liquid surfactant onto a coarse light soda ash utilizing a pan pelletizer to effectuate absorption.

4 Claims, No Drawings

DETERGENT INTERMEDIATE AND PROCESS THEREFOR

BACKGROUND OF THE INVENTION

1. Field of the Invention

The present invention concerns phosphate-free detergents and ingredients therefor. More particularly, the present invention concerns a biodegradable, free-flowing detergent intermediate suitable for use in phosphate-free detergents and which is prepared by absorbing, in a pan pelletizer, a liquid nonionic surfactant onto a carrier of coarse light soda ash.

2. Prior Art

The great concern surrounding eutrophication and pollution of our waterways, as a resultant from the use of phosphates in detergents and the like, has given rise to a groundswell of activity to provide suitable replacements therefor.

To enable achievement of this end result the present invention, as above-noted, provides a free-flowing biodegradable surfactant-loaded soda ash intermediate for use in phosphate-free detergent formulations.

The absorption of surface active agents or surfactants onto inorganic carriers has been known. See, for instance, U.S. Pat. Nos. 3,533,942, 3,306,858 and 3,329,616. However, the present product and process therefor is distinct from the art in that the amount of surfactant absorbed on the soda ash has heretofore been unrealized. Moreover, the process utilized herein has not heretofore been disclosed or suggested for the preparation of such products.

Thus, it will become apparent to those skilled in the art that a major stride in the preparation of phosphate-free detergents is provided by the invention disclosed herein.

SUMMARY OF THE INVENTION

In accordance with the present invention a biodegradable, free-flowing product is prepared in a pan pelletizer by the absorption of a liquid, nonionic surfactant onto a base carrier consisting essentially of a coarse light soda ash. The resulting, graulated product contains from about 15 to 30 percent by weight of surfactant.

For a more comprehensive discussion of the present invention, reference is made to the following detailed description of the preferred embodiment and accompanying drawing which depicts the pan pelletizer employed in accordance with the present invention.

DESCRIPTION OF THE PREFERRED EMBODIMENT

In accordance with the present invention, a surfactant-loaded soda ash product useful as a detergent intermediate is prepared by absorbing onto coarse light soda ash a liquid nonionic surfactant utilizing a pan pelletizer to effectuate absorption.

A pan pelletizer substantially comprises an open-ended cylinder or disc having a cylindrical sidewall mounted on a motor-driven rotatable shaft disposed at an inclined angle to the horizontal plane. As the disc rotates, a solid feed is fed thereinto, the solid feed being sprayed with a liquid absorbent as it is rotated. The construction of a pan pelletizer is more completely shown in FIG. 5 on page 92 of Chemical Engineering, Feb. 5, 1962.

It has been found that the use of a pan pelletizer gives a more uniform, free-flowing product than that obtained with other spray apparatus, such as, ribbon blenders, screw feeders or the like. Moreover, by utilizing the pan pelletizer there is substantially no breakdown of the soda ash particles. Hence, the use of a pan pelletizer to the preparation of a product is considered to be of significant criticality.

The soda ash or sodium carbonate used herein must conform to certain standards in order to be practicable within the scope of the instant invention. It has been found that soda ash "fines" deleteriously affect the properties of the resulting product, thus, it is essential that fines be substantially eliminated as the surfactant carrier. Also, the generation of fines by attrition of the soda ash must be minimized during mixing of the product. The soda ash which is employed herein is a "coarse light soda ash."

As used herein and in the appended claims, the term "coarse light soda ash" means particles of sodium carbonate substantially one hundred percent of which has a mesh size of −20 +100, U.S. Standard Sieve number, and preferably a mesh size of from −10 +80, U.S. Standard Sieve number.

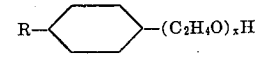
Also, as used herein, the term "absorption" implies that the surfactant is diffused into the crystal lattice structure of the soda ash as well as some of the surfactant remaining on the surface of the soda ash.

Within this mesh size range it is advantageous that the soda ash particles have a distribution of about:

Mesh Size	% Soda Ash, by weight
On 10	0.0–7.0
On 20	1.0–35.0
On 40	5.0–70.0
On 60	10.0–75.0
On 80	5.0–43.0
On 100	0.0–6.0
On Pan	0.0–4.0

The nonionic surfactant which is absorbed onto the soda ash can be selected from any of the well-known liquid nonionic surfactants, such as alkoxylated alkyl phenols, Alkoxylated linear aliphatic alcohols, the conjugated alkylene oxide adducts of hydrophobic bases, and the like.

Suitable alkoxylated alkyl phenols are the ethylene oxide adducts of alkyl phenols represented structurally as:



wherein R is an alkyl group having from 10 to 20 carbon atoms and x is an integer sufficiently large to ensure that the ethylene oxide groups constitute from about 25 to 80 percent by weight of the surfactant. These surfactants are widely known and commercially available.

Another useful class of surfactants are the conjugated alkylene oxide adducts of hydrophobic bases which correspond to the formula:

$$Y[(C_3H_6O)_a(C_2H_4O)_bH]_x$$

wherein Y is the residue of an organic compound containing from two to six carbon atoms and having x reactive hydrogens, x being at least two, a has a value such that the molecular weight of the polyoxypropylene base is at least about 900 and not in excess of about

4000, and *b* has a value such that the oxyethylene content of the molecule is from about 10 to about 50 percent by weight. Compounds defined by Y include, for example, propylene glycol, glycerine, pentaerythritol, trimethylolpropane, ethylene diamine, triethanolamine, triisopropanolamine, butylamine and the like. These surfactants are more particularly described in U.S. Pat. Nos. 2,674,619 and 2,979,528.

Preferred within this group of conjugated compounds are those which correspond to the formula:

$$H(C_2H_4O)_b(C_3H_6O)_a(C_2H_4O)_bOH$$

wherein *a* and *b* have the values described above and Y is propylene glycol, and which are more particularly defined in the above-referred to U.S. Pat. No. 2,674,619.

Another useful class of nonionic surfactants are polyoxyalkylene compounds having heteric polyoxyethylene chains, and which are represented structurally as:

$$Y(-P-E-H)_x$$

wherein Y is the residue of an organic compound having *x* reactive hydrogens and up to six carbon atoms, P is a hydrophobic polyoxyalkylene chain having an oxygen/carbon atom ratio of not more than 0.40, the molecular weight of P and the value of *x* being such that the molecule, excluding E, has a molecular weight of at least about 400 to 900 and up to about 25,000, and E is a hydrophilic polyoxyalkylene chain which (1) contains oxyethylene groups and at least 5 percent by weight of higher molecular weight oxyalkylene groups having at least three carbon atoms in their structure, and (2) has an average oxygen/carbon atom ratio of greater than 0.40, E being present in the composition in an amount sufficient to constitute from about 5 to 90 percent by weight of the total composition. These surfactants are more particularly described in U.S. Pat. No. 3,101,374.

Still another useful class of liquid nonionic surfactants are the alkoxylates of linear aliphatic alcohols. These surfactants can be generally designated as:

$$R-O(A)_nH$$

wherein R is the organic residue of a linear aliphatic alcohol or mixtures thereof having from eight to 20 carbon atoms in the aliphatic portion, A represents oxyalkylene groups, and *n* is an integer such that the oxyalkylene groups constitute from 55 percent to 80 percent by weight of the compound. Generally, A represents oxyethylene groups, oxypropylene groups or mixtures thereof. These surfactants are generally prepared either by using a random mixture of oxyalkylene groups or in sequential addition thereof. When both oxyethylene groups and oxypropylene groups are employed they are present in a respective weight ratio of from about 1:2 to 7:1. These surfactants are more particularly described in U.S. Pat. Nos. 3,340,309, and 3,504,041 and Canada Pat. No. 770,664.

Other useful surfactants include the propylene oxide adducts of polyoxyethylene glycol such as described in U.S. Pat. No. 3,036,118.

Although any conventional liquid nonionic surfactant can be employed herein, it is preferred to employ the two alkoxylates of linear aliphatic alcohols, since these are completely biodegradable. It should be noted, also, that it is contemplated herein that surfactants which are not conventionally liquid in nature can be used herein if they are liquid at the temperatures employed in the present process which are more fully defined hereinafter.

In preparing the detergent intermediate or builder of the present invention, the coarse light soda ash is loaded into the pan pelletizer and the liquid nonionic surfactant is sprayed thereonto. The surfactant is sprayed onto the soda ash at a temperature ranging from about 15° to 65° C. and, preferably, at a temperature of from about 20° to 35° C. The soda ash is nominally at a temperature of about 15° to 95° C., and preferably, at about 20° to 70° C., and in an amount ranging in parts by weight, from about 15 to 30 of surfactant per 85 to 70 of coarse light soda ash.

By operating within these parameters maximum absorption of soda ash is achieved. Neither the angle of inclination nor the speed at which the shaft rotates are critical to preparing the product.

The product obtained by the process of the present invention, is a free-flowing, biodegradable, detergent intermediate having from 15 to 30 percent by weight of surfactant absorbed onto the coarse light soda ash. This is a distinct improvement over the prior art where such products contain only minimal amounts of surfactants.

For a more complete understanding of the present invention, reference is made to the following illustrative examples.

EXAMPLE I

This example illustrates the preparation of a detergent intermediate in accordance with the present invention.

Using a three-foot inclined pan pelletizer rotating at 15 rpm and set at an angle of rotation of 22.5° from the horizontal plane about 870 lbs./hr. of a coarse light soda ash at a temperature of about 22° C. were continuously fed into the pelletizer. As the soda ash rotated, about 292 lbs./hr. of a liquid nonionic surfactant at a temperature of about 22° C. was sprayed thereonto with a fan spray. After about 7.5 hours, 7,600 lbs. of product were obtained. Random samplings of the resulting product showed that an average of 25.4 percent, by weight, of surfactant had been absorbed onto each particle of soda ash.

The product had an average flowability of about 63.4 percent as determined by a laboratory prepared test funnel that is fitted onto a half-gallon jar (the flowability is expressed as the ratio of the time in seconds required to empty the jar filled with a standard reference material, such as furnace grade sand, to the time required to empty the jar filled with the product being evaluated), and the following mesh size distribution:

U.S. Standard Sieve No.	Amt. of Soda Ash, %
On 20	4.4
On 40	9.8
On 60	71.2
On 80	14.2
On 100	0.4

The coarse light soda ash employed herein had the following particle size distribution:

U.S. Standard Sieve No.	Amt. of Soda Ash, % by weight
On 20	3.1
On 40	4.6
On 60	36.1
On 80	48.4
On 100	4.4
On Pan	3.4

The surfactant utilized herein was the condensation product of a mixture of C_{12}–C_{15} alcohols and a mixture of ethylene oxide and propylene oxide in a respective weight ratio of about 3:1, the resulting product containing about 60 percent by weight of ethylene oxide; about 20 percent by weight of propylene oxide, and about 20 percent by weight of alcoholic residues. The starting mixture of alcohols contained, by weight, about 20% C_{12}, 30% C_{13}, 30% C_{14}, and 20% C_{15} fatty alcohols.

EXAMPLE II

Following the procedure of Example I, substantially similar results, i.e. a free-flowing product containing 25 percent surfactant, were obtained utilizing the following surfactants:

1. the condensation product of a mixture of C_{12}–C_{15} alcohols, as described in Example I and a mixture of ethylene oxide and propylene oxide in a respective weight ratio of about 1.5:1. The resulting product containing about 50 percent oxyethylene groups, about 30 percent oxypropylene groups and about 20 percent alcoholic residues;

2. the condensation product of a mixture of C_{12}–C_{15} alcohols, as described in Example I, and a mixture of ethylene oxide and propylene oxide in a respective weight ratio of about 1:2, the resulting product containing about 25 percent oxyethylene groups, about 50 percent oxypropylene groups and about 25 percent alcoholic residues;

3. the ethylene oxide adduct of a 1750 molecular weight polyoxypropylene glycol containing about 20 percent by weight of ethylene oxide;

4. a propylene oxide adduct of a 780 molecular weight polyoxyethylene glycol containing about 75 percent by weight of propylene oxide, and more particularly described in U.S. Pat. No. 3,036,118; and

5. a surfactant similar to Example 42 of U.S. Pat. No. 3,101,374 but having a total molecular weight of about 3,900 with the hydrophilic chain constituting about 30 percent of the total molecular weight of the surfactant.

The percent flowabilities of the products produced, by loading 25 percent of each surfactant onto the coarse light ash were as follows:

Surfactant	% Flowability
1	71.7
2	71.0
3	76.8
4	77.7
5	76.8

The coarse light ash used in this example had the following particle size distribution:

Mesh Size	% Soda Ash
On 20	0.3
On 40	2.0
On 60	69.6
On 80	22.5
On 100	3.1
On Pan	2.5

EXAMPLE III

Utilizing a detergent intermediate prepared from a coarse light soda ash as described in Example I and the surfactant identified as (2) in Example II a detergent composition was prepared. The composition consisted essentially of by weight:

Ingredient	Amount
Detergent Intermediate	32
Sodium Metasilicate	15
Sodium Sulfate	45
Sodium Gluconate	8

This composition was tested and compared with a standard domestic detergent for foaming characteristics using a milk soil test and an egg soil test.

Predicated on the theorem that the number of revolutions of a dishwasher rotor arm is inversely proportional to the foam generated therein. The test employed was substantially as follows:

Into a domestic dishwasher, equipped with an electric counter which counts the revolutions of the rotor, maintained at the desired temperature is placed a watch glass containing 20 parts of detergent and either 12 parts of dry powdered milk or 15 ml. of stirred raw whole egg, depending on the desired soil. As the door in the dishwasher is closed, the counter is turned on and a stop watch is commenced. The number of revolutions of the rotor arm is then recorded after the first minute of operation and after the second minute. The difference therebetween is the number of revolutions for the second minute of operation. The same determination is made for the third and fourth minutes to determine the number of revolutions for the fourth minute of operation. The average of the two readings is utilized as the recorded value of RPMs.

The results of this test for the built detergent prepared from the present invention and the comparative detergent were as follows:

	Average Rotor Arm Speed, RPM		
	No Soil	Milk Soil	Egg Soil
1. At 100° F.			
Present Invention	107	95	77
Domestic Product	86	70	70
2. At 120° F.			
Present Invention	109	93	81
Domestic Product	90	71	72
3. At 140° F.			
Present Invention	113	95	84
Domestic Product	88	77	79

It can be seen from the above that in all instances the detergent prepared from the detergent intermediate of the present invention outperformed the domestic product.

It should be further noted that soiled drinking glasses placed contemporaneously in the dishwasher with the soiled watch glass having the detergent composition of the present invention placed thereon emerged from the dishwasher with no water streaks or spots.

Having thus described the invention, what it is desired to claim and secure by Letters Patent is:

1. A process for the preparation of a detergent intermediate comprising absorbing onto a coarse light soda ash at a temperature of from 15°–95°C, in a pan pelletizer a nonionic surfactant at a temperature of from 15–65° C, the soda ash having absorbed thereon from about 15 to 30 percent by weight of surfactant based on the weight of the intermediate.

2. The process of claim **1** wherein the soda ash has a mesh size of −20 +100 U.S. Standard Sieve number.

3. The process of claim **2** wherein the soda ash has a U.S. Standard Sieve number mesh size of −10 +80.

4. The process of claim **1** wherein the surfactant is sprayed onto the soda ash.

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