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Ali et al.

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[54] **DETERGENT COMPOSITION**

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[51] **Int. Cl.** **C11d 1/14**

[58] **Field of Search** ..... 252/554, 535, DIG. 14

[56] **References Cited**

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[57] **ABSTRACT**

Primary and secondary alkylsulfonates with carbon numbers lower than those which afford optimum detergency are used as hydrotropes in detergent formulations. These alkylsulfonates are linear, and have a carbon content ranging from C_7 to C_8 inclusive.

5 Claims, No Drawings

DETERGENT COMPOSITION

This invention relates to novel hydrotropes suitable for incorporation in detergent compositions.

Hydrotropes are salts which effect substantially greater solubility of slightly soluble substances than does water at the same temperature. This behavior is the opposite of the common salting - out effect following the addition of many electrolytes to aqueous solutions of numerous solutes.

Typical of hydrotropes salts are the alkali or alkaline earth salts of the sulfonates of toluene, xylene. The most generally used hydrotrope in detergents is sodium xylenesulfonate because it is inexpensive and effective as a solvating agent. In the manufacture of heavy duty detergent powders they reduce the viscosity of the slurry before spray - drying. In heavy duty liquid detergents they may act as solubilizers and coupling agents or even as cloud point depressants.

In accordance with the present invention it has been discovered that heptyl and octyl sulfonates can act as hydrotropes in detergent compositions when from 3 to 9 weight percent thereof are incorporated in such compositions.

The subject secondary alkyl sulfonates can be prepared by a sulfoxidation reaction disclosed and claimed in commonly assigned copending patent application Serial No. 40,740 filed on May 26, 1970.

In this reaction the C_7–C_8 n-paraffins alone or admixed are reacted with oxygen and sulfur dioxide under substantially anhydrous conditions in the presence of acetic anhydride or of a low molecular weight acyl oxide at temperature of about 25 oc to about 55 oc under pressures of 0 to 100 psig.

The primary alkylsulfonates can be prepared by a bisulfite reaction involving reacting the 1-olefin or 1-olefins with aqueous bisulfite in a mutual solvent under basic conditions at relatively low temperatures in the presence of an oxygen-containing initiator. This reaction is disclosed and claimed in commonly assigned copending application Serial No. 47,485 filed June 18, 1970.

For optimum results, reaction conditions, falling within the scope of the above mentioned applications, have been ascertained.

Surprisingly for the same set of conditions, the short chain olefins require longer reaction periods (15–20 hours) than the longer chain olefins (n8 hours). The analysis of the paraffin feedstocks used to prepare the secondary sulphonates is shown in Table II. The sulphoxidation reaction was carried out in a stirred tank reactor at atmospheric pressure. Reaction Conditions are summarised in Table III, and it should be noted that considerable difference exists between the reaction rates of the C_7 and C_8 paraffins. This is probably due to the large amount of impurities (mainly naphthenes) in the former sample (see Table II). Both the primary and secondary sulfonates have been analysed; the results are given in Table IV. Finally various surface active and detergent properties are summarised in Table V together with those of a linear alkylate sulfonate (Nacconol NF), included for comparison.

Viscosity measurements given below show that by incorporating 5 percent C_7 and C_8 alkylsulfonates, the viscosity of a 25 percent dodecylbenzene sulfonate solution (Nacconol NRSF) is reduced.

Sample	SSU at 100°F.
Nacconol NRSF (25%)	40
" +O-Xylene sulfonate (5%)	33
" +C_7 Primary " "	34
" +C_8 Primary " "	33
" +C_7 Secondary " "	34
" +C_8 Secondary " "	33

The solubilizing properties were determined by (Table A) measuring the solubility of a given C_{11}–C_{19} primary alkylsulfonate sample, as well as on blends of this sample and the hydrotrope under investigation. In determining the solubility, sufficient water was added so that only little solids remained un dissolved. The carbon number distribution of the C_{11}–C_{19} primary alkylsulfonate is shown in Table B. These compounds have optimum detergency.

TABLE A

Sulfonate Sample	Hydrotrope (Sulfonate)	%Hydrotrope in blend	Solubility of blend at 30°
Mixture of C_{11}–C_{19} and C_{15}–C_{18} Primary Sulfonate	Nil	Nil	4.3 g./100 ml
"	O-Xylene	19	4.7 g./100 ml
"	C_7-Primary	22	12.8 g./100 ml
"	C_8-Primary	27	13.2 g./100 ml
"	C_7-Secondary	26	17.2 g./100 ml
"	C_8-Secondary	23	16.8 g./100 ml

TABLE B

Carbon Number Distribution of C_{11}–C_{19} Primary Alkylsulfonate

Gas Liquid Chromatography Identification	%
C_{11}	3.5
C_{12}	5.0
C_{13}	5.5
C_{14}	5.6
C_{15}	13.7
C_{16}	22.4
C_{17}	21.6
C_{18}	13.4
C_{19}	6.3
C_{20}	2.5
C_{21}	0.5

TABLE I 1-OLEFIN BISULPHITE REACTION CONDITIONS

Reaction Temperature:				60°C
pH of Ammonium Sulphite Solution:				8.3
Initiator:				t-butyl perbenzoate
Run No.	520[a]	517[a]	522[b]	523
Charge				
Olefins, C No.	C_7	C_8	C_{12}	C_7
moles	1.0	1.0	0.5	0.5
Sulphite, moles	1.0	1.0	0.5	0.5
volume, ml	620	620	310	310
iso-Propanol, ml	620	620	310	310
Reaction Time, Hr.	20	20	8	15

(a) — In order to prepare large samples, it was necessary to do several runs and combine the products. The runs shown here are typical ones.
(b) — This run using C_{12} olefins is included for comparison to illustrate the difference in reactivity due to carbon chain lengths.

TABLE II — ANALYSIS OF C_7 AND C_8 PARAFFINS USED IN SULPHOXIDATION

Gas Liquid Chromatography Identification	C_7 Sample	C_8 Sample
C_3	0.01	
i-C_4	0.01	
n-C_4	0.06	
i-C_5	0.06	
n-C_5	0.08	
2-Methylpentane	0.09	
3-Methylpentane	0.04	
n-C_6	0.22	
Methylcyclopentane	0.09	
Cyclohexane	0.07	

2-Methylhexane	1.67	
Unid	0.35	
3-Methylhexane	2.24	
Dimethylcyclopentanes	1.00	
i-C_7	0	0.15
n-C_7	91.98	0.42
Methylcyclohexane	0.55	—
Unid	0.19	—
i-C_8	—	1.16
n-C_8	0.30	93.46
i-C_9	—	3.02
n-C_9	—	1.79
Optical Density at 270 mu	0.06	0.1

TABLE III — N-PARAFFIN/SULPHOXIDATION REACTION CONDITIONS

Sulphoxidation Run No.	STR-77	STR-78
Paraffin Charge, C No.	n-C_8	n-C_7
Volume, ml	1000	1000
Reaction Temperature	35	35
SO_2/O_2, l/hr.	20/10	20/10
Recycle Rate, l/hr.	2	2
Product Formation, g./hr.	100–110	45–50

TABLE IV — ANALYSIS OF C_7 AND C_8 ALKYLSULPHONATES

Sample Description:

Source	n-Paraffin/Sulphoxidation		1-Olefin/Bisulphite	
Carbon No.	C_7	C_8	C_7	C_8
RS No.	1122/70	1123/70	917/70	918/70
Sulphonate Content, %-wt.	81	79	79	85
Sulphate Content, %-wt.	19	21	21	15
Monosulphonate Content, %-mole (Sulphate-free basis)	76	63	90	93

A suitable range of detergent constituents employing the hydrotropes of this invention comprises.

Hydrotropes — 20 to 30 weight percent, based on the total active matter content

Detergent — 15 to 30 g. per 100 wt. solution (Na alkyl sulfonate)

Foam stabilizer (fatty acid alkylolamide) — 0 to 5 g. per 100 wt. solution used (as required)

Water — balance.

More specific detergent formulations are the following (Na salts): (in parts by weight)

	A	B	C	D
Active Matter (eg LAS)	25	25	25	25
C_7 Primary alkylsulphonate	5			
C_8 Primary "		5		
C_7 Secondary "			5	
C_8 Secondary "				5

A suitable method for determining whether the described hydrotropes are biodegradable is not available. The Soap and Detergent Association method of analysis is insensitive to C_7–C_8 and other short chain alkylsulfonate. However, as the alkyl chains are linear, the compounds are probably biodegradable.

What is claimed is:

1. A detergent composition consisting of about 3 to 9 weight percent of a C_7 or C_8 linear alkylsulfonate as a hydrotrope, from 15 to 30 weight percent of linear alkylsulfonate detergent having 11 to 19 carbon atoms in the alkyl moiety, up to 5 wt. percent of a fatty acid alkylolamide foam stabilizer and the balance water.

2. A composition according to claim **1** containing about 5 parts by weight of a C_7 primary alkylsulfonate and about 25 parts by weight of a linear alkylsulfonate having a carbon content in the C_{11}–C_{19} range.

3. A composition according to claim **1**, containing about 5 parts by weight of a C_7 secondary alkylsulfonate and about 25 parts by weight of a linear alkylsulfonate having a carbon content in the C_{11}–C_{19} range.

4. A composition according to claim **1** containing about 5 parts by weight of a C_8 secondary alkylsulfonate and about 25 parts by weight of a linear alkylsulfonate having a carbon content in the C_{11}–C_{19} range.

5. A composition according to claim **1** containing about 5 parts by weight of a C_8 primary alkylsulfonate and about 25 parts by weight of a linear alkylsulfonate having a carbon content in the C_{11}–C_{19} range.

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