Soap-Based Detergent Formulations I. Comparison of Soap–Lime Soap Dispersing Agent Formulations With Phosphate Built Detergents[1]

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ABSTRACT

Blends of soap with small amounts of lime soap dispersing agents are efficient detergents in hard water and require little or no tripolyphosphate builder. Lime soap dispersing agents examined include sulfated ethoxylated fatty alcohols, sulfated N-(2-hydroxyethyl) fatty amides, methyl esters of α-sulfo fatty acids, 2-sulfoethyl fatty acid esters and N-methyl-N-(2-sulfoethyl) fatty amides as well as nonionics derived from tallow alcohols. Detergency evaluations were carried out with three commercial soiled cotton cloths as well as by a laboratory multi-wash technique. Formulations containing 80% soap, 10% lime soap dispersing agent and 10% builder gave optimum detergency values. Builder effectiveness was rated tripolyphosphate>silicate (1:1.6)> metasilicate = citrate = oxydiacetate = nitrilotriacetate>carbonate≫sulfate. The detergency of soap-lime soap dispersed combinations compared favorably with a standard brand household heavy duty granular detergent in 50, 150 and 300 ppm hardness water on three soiled cloths.

INTRODUCTION

The development of molecularly dehydrated phosphates contributed heavily to the successful growth of the synthetic detergent industry and therefore was instrumental in the replacement of soaps (1). The major functions of phosphates are: (a) sequestration of calcium and other hard water ions, (b) maintenance of alkalinity through buffering action, (c) contribution to colloidal stability, and (d) maintenance of a free flowing powder which does not cake.

Prior to the development of synthetic detergent formulations, soap had a long history of satisfactory usage except in hard water. It was customary to use additional soap in hard water areas, so that the excess sodium soap would keep the calcium soap in suspension. While this procedure worked well for the washing operation, rinsing produced dilute hard water solutions from which lime soap curd readily precipitated. This shortcoming of soap can now be alleviated with the aid of so-called lime soap dispersing agents (LSDA) which keep the lime soaps finely divided and suspended in hard water.

To be an efficient LSDA the surfactant must possess a long alkyl chain and a bulky hydrophilic group (2). Several soap-LSDA combinations have been proposed for use in soap-syndet toilet bars (3,4), but none have been recommended for powdered detergent formulations. The effects of various builders on soap-LSDA formulations have also not been reported. This paper deals with the effect of compositional variations on solution appearance and on detergency in single and multiple washings.

EXPERIMENTAL PROCEDURE

Materials

Tallow fatty acids, a commercial product (Wilson-Martin Co., Philadelphia, Pa.) having the composition 24.7% stearic acid, 42.4% oleic acid, 27.2% palmitic acid, 5.1% myristic acid and 0.5% lauric acid, was neutralized with sodium hydroxide in ethanol solution to form sodium tallowate soap. U.S.P. oleic acid was similarly neutralized to prepare sodium oleate. Methyl esters of α-sulfo tallow fatty acids (5) ether alcohol sulfates (6), sodium 2-sulfoethyl tallowate and sodium *N*-methyl-*N*-(2-sulfoethyl) tallow amides (7) and tallow alcohol derived nonionics (8) were synthesized as described previously. Sodium silicates with SiO_2/Na_2O ratios from 1.0-2.0 were supplied by Philadelphia Quartz Co., Philadelphia, Pa. Sodium oxydiacetate (diglycolate), sodium citrate, sodium tripolyphosphate, sodium carbonate and sodium sulfate were standard laboratory reagents. Nitrilotriacetate, trisodium salt (NTA) was supplied by the Hampshire Chemical Division of W.R. Grace & Co., Nashua, N.H. A salt-free linear alkyl benzene sulfonate (LAS) was prepared from Ultrawet K (Arco Chemical Co., Philadelphia, Pa.).

Test Methods

The Borghetty and Bergman test method (9) was used to measure lime soap dispersing power. Turbidity was measured in a 1 cm cell in an Electrophotometer II (Fisher

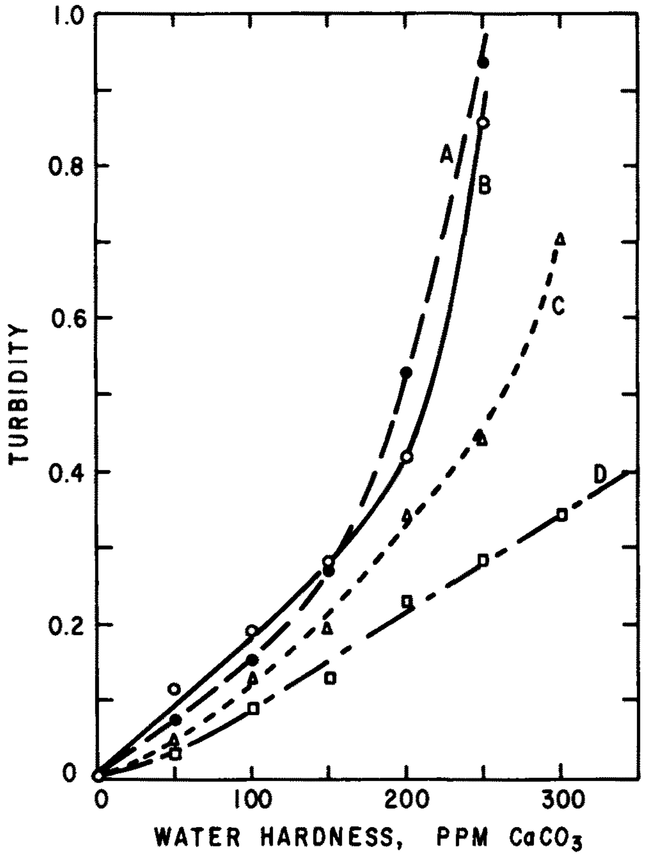

FIG. 1. Effect of water hardness on turbidity of soap-NaMeα-ST mixtures: A, 0.2% sodium oleate; B, 0.19% sodium oleate + 0.01% NaMeα-ST; C, 0.18% sodium oleate + 0.02% NaMeα-ST; D, 0.16% sodium oleate + 0.04% NaMeα-ST.

[1]Presented at the AOCS Meeting, Atlantic City, October 1971.
[2]East. Market. Nutr. Res. Div., ARS, USDA.

TABLE I

Effectiveness of Selected Lime Soap Dispersing Agents

Lime soap dispersing agent, sodium salt	Lime soap dispersing power, % (9)	ΔR, Soap 80% + LSDA 20%	ΔR[a] Soap 80% + LSDA 10% + builder 10%					
			$Na_5P_3O_{10}$	Sodium citrate	Sodium oxydiacetate	Na_2SiO_3	Na_2CO_3	Na_2SO_4
Sodium 2-sulfoethyl tallowate	10	32.2	35.7	31.2	30.9	37.1	31.8	26.2
Sodium methyl α-sulfotallowate	8	34.2	39.4	35.3	38.0	34.3	31.6	29.0
Sodium sucrose α-sulfotallowate	10	34.9	38.0	34.3	35.3	33.6	31.7	28.3
Disodium 2-sulfoethyl α-sulfostearate	5	30.7	33.8	33.5	32.3	31.9	29.7	25.1
Sodium N-methyl-N-(2-sulfoethyl) tallow amide	6	32.8	34.7	34.4	33.2	31.6	30.5	28.2
Sodium sulfated ethoxylated tallow alcohol, (3.3 moles of EO)	4	30.3	42.7	31.8	29.0	35.4	32.8	27.5
Sodium sulfated *N*-(2-hydroxyethyl) tallow amide	4	31.4	35.9	34.8	30.6	35.2	30.5	30.3
Sodium linear alkylbenzene sulfonate (LAS)	40	23.6	33.9	29.9	27.2	29.6	25.8	23.7
Soap 90% + builder 10%	---	---	35.9	27.0	29.3	31.5	28.2	23.9

[a]ΔR values obtained in single wash tests at 0.2% total concentration at 60 C in 300 ppm hardness water. ΔR for soap alone = 28.8.

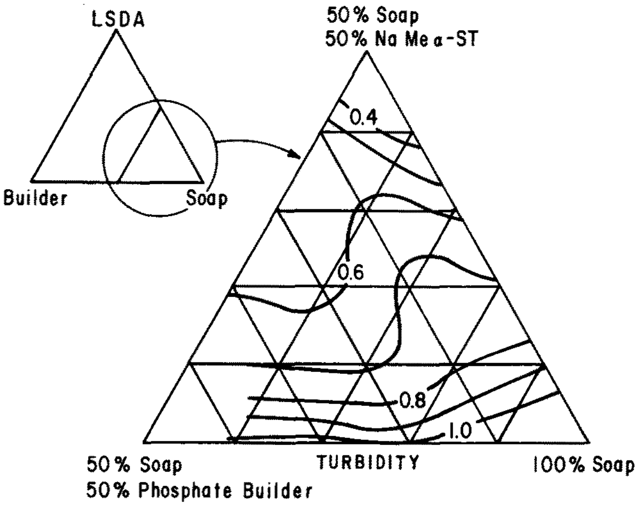

FIG. 2. Turbidity of ternary mixtures of tallow soap, NaMeα-ST and phosphate builder (55% $Na_5P_3O_{10}$, 24% Na_2SO_4, 10% $Na_4P_2O_7$, 10% Na_2SiO_3, 1% CMC).

Scientific Co., Pittsburgh, Pa.) using the tristimulus green filter (λ = 525 mμ).

The Terg-O-Tometer (U.S. Testing Co., Inc., Hoboken, N.J.) was used for all detergency evaluations. For single wash tests, Testfabrics and EMPA 101 soiled cotton cloths (Testfabrics, Inc., New York, N.Y.) were used as well as U.S. Testing Co. cloth. Ten 4 in. circular swatches were washed in a liter of solution at 60 C for 20 min, 110 cycles per min. Detergency was measured by the increase in reflectance after washing (ΔR). Analysis of variance indicated that the following differences in ΔR values had a 95% probability of being significant: Testfabrics 1.6-1.8, U.S. Testing 0.4-0.5, EMPA 0.6-0.7.

A multiwash test such as that described by Schwartz and Berch (10) tends to show the beneficial effects of phosphate builder over other inorganic builders of similar pH for cotton detergency. Vacuum cleaner sweepings, collected from a Philadelphia office building, were sifted to pass a 40 mesh screen and applied to cotton swatches from an aqueous suspension. Thus 5 g of soil was applied to ten 4 in. circular swatches of cloth (No. 400W, 80 x 80 bleached cotton print cloth from Testfabrics having an initial reflectance of 90.5). Ten soiled swatches were washed in each Terg-O-Tometer beaker. The soil and wash cycle was repeated five more times. Redeposition was determined by washing two unsoiled swatches per beaker along with the 10 soiled swatches in each washing step. Analysis of variance showed that differences in reflectances greater than 1.0 had a 99% probability and differences greater than 0.8 had a 95% probability of being significantly different.

RESULTS AND DISCUSSION

The effect of variation in the degree of water hardness upon the turbidity of 0.2% sodium oleate and mixtures of sodium oleate and sodium methyl α-sulfotallowate (NaMeα-ST) is illustrated in Figure 1. Soap alone and soap containing 5% NaMeα-ST (based on total solute concentration) showed a sharp rise in turbidity above 200 ppm $CaCO_3$ water, and precipitated at 300 ppm. The equivalence point for the complete reaction of 2 moles of sodium oleate with 1 mole of calcium ion occurs at 300 ppm water hardness for 0.2% sodium oleate solution. The addition of 10 and 20% LSDA resulted in proportionate decreases in turbidity with no evidence of precipitation.

The turbidity behavior of the ternary system soap-LSDA-sodium tripolyphosphate ($Na_5P_3O_{10}$) is represented graphically in Figure 2 where optical density is plotted as a function of composition of the ternary mixture. Turbidity

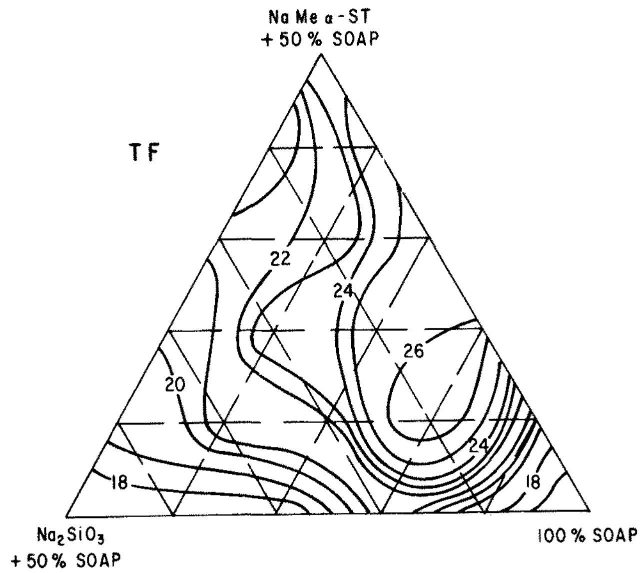

FIG. 3. Detergency of ternary system soap-NaMeα-ST-Na_2SiO_3 on Testfabrics soiled cotton fabric.

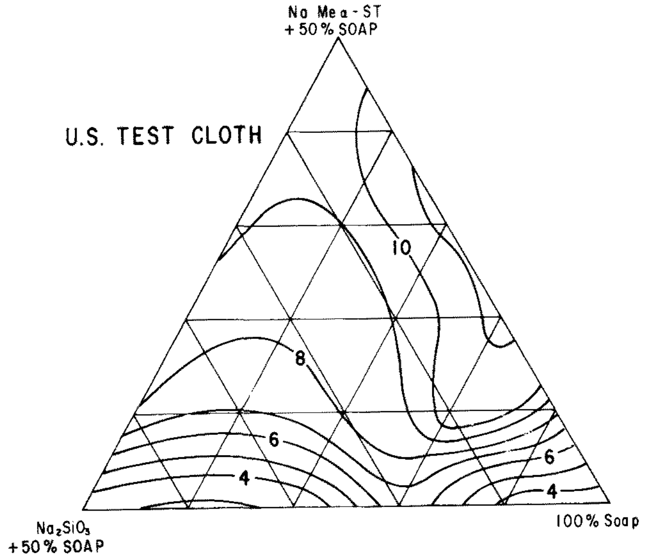

FIG. 4. Detergency of ternary system soap-NaMeα-ST-Na_2SiO_3 on U.S. Testing Co. soiled cotton fabric.

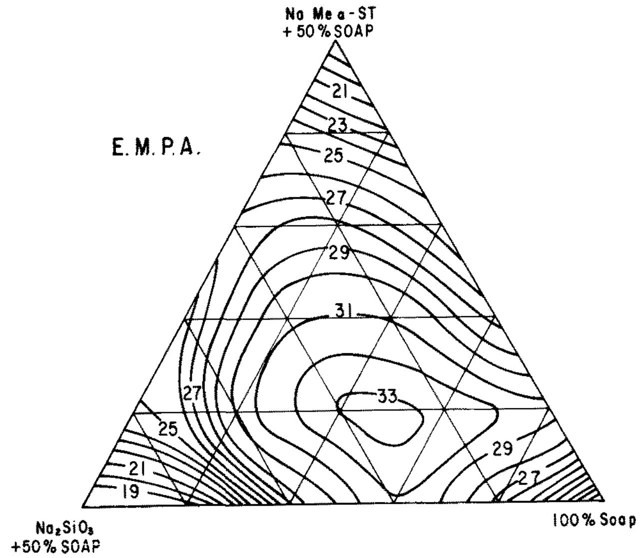

FIG. 5. Detergency of ternary system soap-NaMeα-ST-Na_2SiO_3 on EMPA 101 soiled cotton fabric.

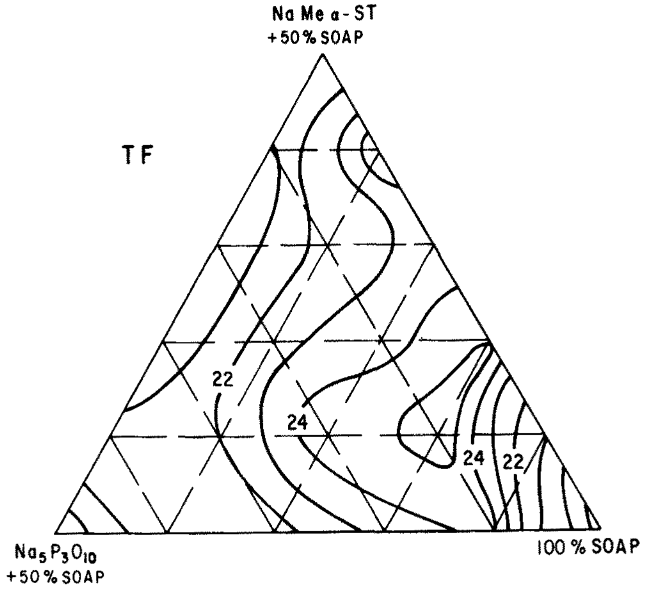

FIG. 6. Detergency of ternary system soap-NaMeα-ST-$Na_5P_3O_{10}$ on Testfabrics soiled cotton fabric.

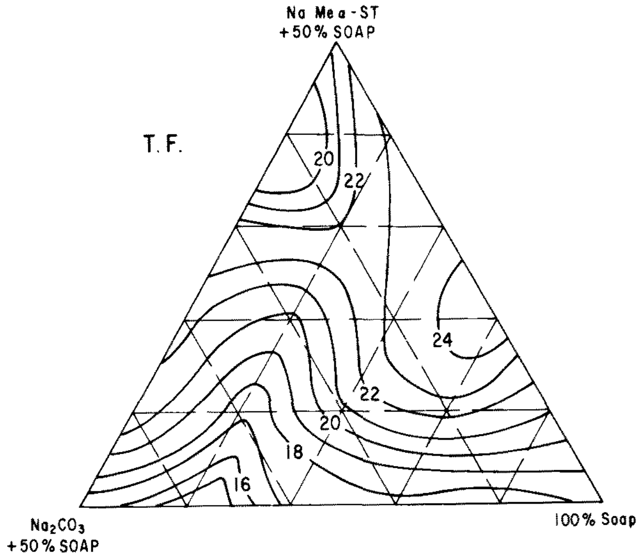

FIG. 7. Detergency of ternary system soap-NaMeα-ST-Na_2CO_3 on Testfabrics soiled cotton fabric.

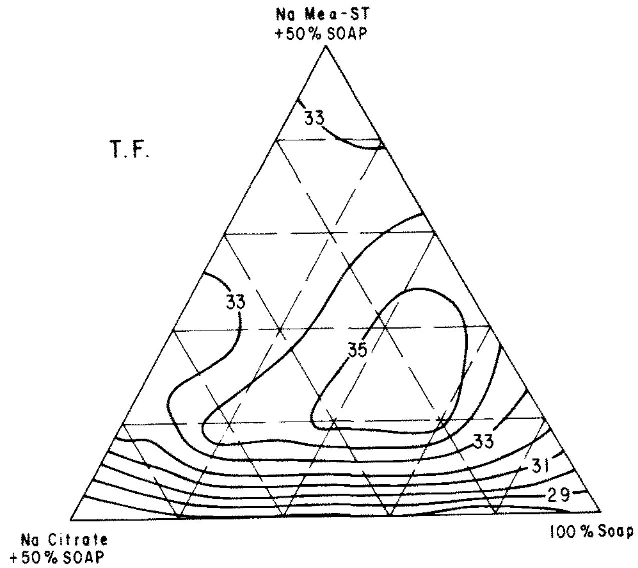

FIG. 8. Detergency of ternary system soap-NaMeα-ST-sodium citrate on Testfabrics soiled cotton fabric.

TABLE II

Composition of Soap–Lime Soap Dispersing Agent–Builder Systems at Detergency Maxima

Experiment	Additives to soap: Lime soap dispersing agent	Additives to soap: Builder	Composition of ternary system at detergency maximum: Soap, %	LSDA, %	Builder, %
1	R CH (SO_3Na) $COOCH_3$(NaMeα-ST)[a]	$Na_5P_3O_{10}$	80	10	10
2	→	Na_2SiO_3	80	10	10
3	→	$Na_2O/1.8\ SiO_2$	80	10	10
4	→	Na_2CO_3	80	20	0
5	→	$Na_3C_6H_5O_7$(citrate)	80	10	10
6	→	N $(CH_2COONa)_3$(NTA)	80	10	10
7	→	O $(CH_2COONa)_2$(SODA)	80	10	10
8	R'$C_6H_4SO_3Na$ (LAS)[b]	Na_2SiO_3	60	40	0
9	→	Na_2CO_3	60	40	0
10	RO $(CH_2CH_2O)_3SO_3Na$[a]	Na_2SiO_3	80	10	10
11	n-$C_{16}H_{33}O$ $(CH_2CH_2O)_{10}H$	Na_2SiO_3	80	10	10
12	→	Na_2CO_3	70	20	10

[a]R is tallow-derived alkyl.
[b]R' is petroleum derived alkyl ($C_{12}H_{25}$ average).

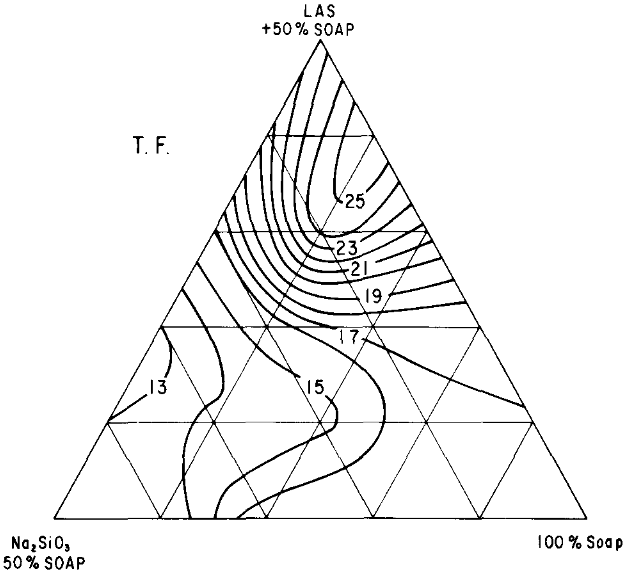

FIG. 9. Detergency of ternary system soap-LAS-Na_2SiO_3 on Testfabrics soiled cotton fabric.

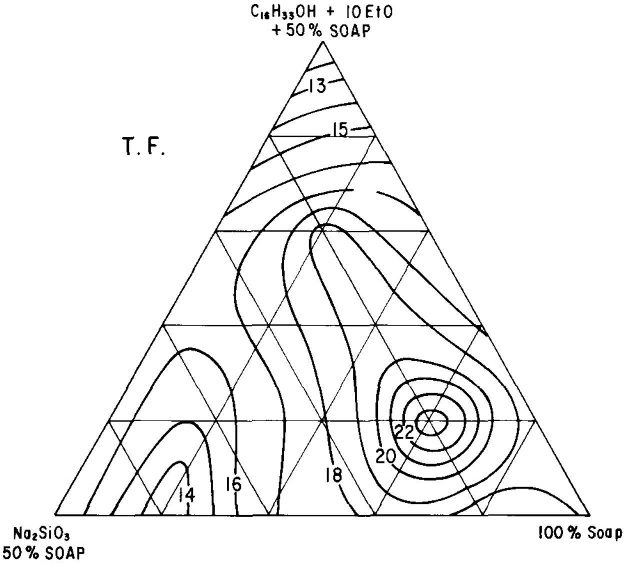

FIG. 10. Detergency of ternary system soap-$C_{16}H_{33}$-$(OCH_2CH_2)_{10}OH$-Na_2SiO_3 on Testfabrics soiled cotton fabric.

data were obtained for the ternary compositions corresponding to each of the intercepts, and each curve represents compositions of approximately equal turbidity.

Figure 2 shows that turbidity decreased steadily as the amount of LSDA was increased; however a change in the amount of $Na_5P_3O_{10}$ builder had little or no effect on turbidity in this region.

A typical wash load of clothes holds about an equal weight (100% pickup) of the wash water which in the rinse cycle is diluted about 1:10 with water of equal hardness. The stability of such rinse water solutions was tested by diluting 1:10 with 300 ppm hard water. While soap alone always formed curds in this test, the use of 10% or more LSDA resulted in stable suspensions.

Borghetty and Bergman (9) have developed a convenient test for lime soap dispersing power which measures the amount of dispersing agent required to maintain an oleate soap dispersion in 300 ppm hard water. A number of surfactants were screened for lime soap dispersing power and the more effective LSDA, then were compared with each other in single detergency tests as shown in Table I. Both binary soap-LSDA blends and ternary systems of soap-LSDA-builder were studied. As expected high detergency, as expressed in reflectance increase (ΔR) of the test fabrics, resulted from compounds of high lime soap dispersing power (hence low Borghetty-Bergman values); however good detergency was not necessarily a linear function of the Borghetty-Bergman test values. There appears to be no direct relationship between detergency and lime soap dispersing power as shown by a single wash test technique.

Detergency of binary mixtures could be further enhanced by the replacement of one half of the LSDA by one of several detergent builders. The order of effectiveness of builders evaluated, as measured in the single wash tests, was tripolyphosphate>citrate = metasilicate = NTA = oxydiacetate>carbonate≫sulfate. Tripolyphosphate enhanced the detergency for all lime soap dispersing agents studied. Sulfate was detrimental to detergency in most of the systems. Citrate, metasilicate, NTA and oxydiacetate enhanced detergency only in some ternary systems.

In view of the fact that the detergency of soap is enhanced by the presence of LSDA and builders, a more detailed detergency study was undertaken wherein the relative proportions of soap, LSDA and builder were varied. Selected results of this study are represented in the form of eight triangular diagrams of Figures 3-10. These diagrams show detergency (in terms of ΔR values) for varying proportions of three component systems at a total concentration of 0.2% of the formulated detergent and at 300 ppm water hardness. In these graphs T.F. stands for Testfabrics, Inc. soiled cotton cloth, EMPA for EMPA 101 cotton soiled cloth, and U.S.T. for U.S. Testing, Inc. soiled cotton cloth. Since the most significant changes in detergency were observed in the 50-100% concentration range of soap and 0-50% of the LSDA and builder respectively, the triangular graphs of Figures 3-10 are restricted to these concentration ranges. The curves of the diagrams connect compositions of approximately equal detergency. In many of the systems studied we observed a rather striking phenomenon of a detergency maximum occurring at an approximate composition of 80% soap, 10% LSDA and 10% builder. Figures 3, 5, 6, 8 and 10 illustrate this phenomenon. Most surfactants used as lime soap dispersing agents in this study enhanced the detergency of soap in binary systems. The potentiation of sodium methyl α-sulfotallowate was further enhanced by the addition of builders such as sodium metasilicate (Fig. 3 and 5) sodium tripolyphosphate (Fig. 6), sodium citrate (Fig. 8), as well as by NTA and by sodium oxydiacetate for which diagrams are not shown. On the other hand addition of sodium carbonate did not enhance the detergency further (Fig. 7).

When the LSDA was a typical nonionic surfactant such as the ten mole ethylene oxide adduct to *n*-hexadecyl alcohol (Fig. 10), a maximum was obtained at 80% soap-10% LSDA-10% sodium metasilicate, but the maximum had a lower detergency value than those of the anionic LSDA formulations.

The bulk of the detergency evaluations were carried out using Testfabrics cotton test cloth only. Because of the bias inherent in laboratory detergency tests with standard soiled test cloths we expanded our detergency evaluation of the Soap-NaMeα-ST-Na_2SiO_3 system to include U.S. Testing and EMPA test cloths as well. The enhanced potentiation due to the presence of a builder was barely apparent with the U.S. Testing cloth, (Fig. 4) whereas it was quite apparent with EMPA. However in this instance maximum detergency was observed at a ratio of 70% soap, 10% LSDA and 20% builder (Fig. 5) as opposed to 80:10:10 observed with Testfabrics cloth.

Several ternary soap-LSDA-builder systems were evaluated in analogous fashion. Table II gives a summary of the findings with Testfabrics cloth. Wherever a detergency maximum was found, the corresponding component ratio is

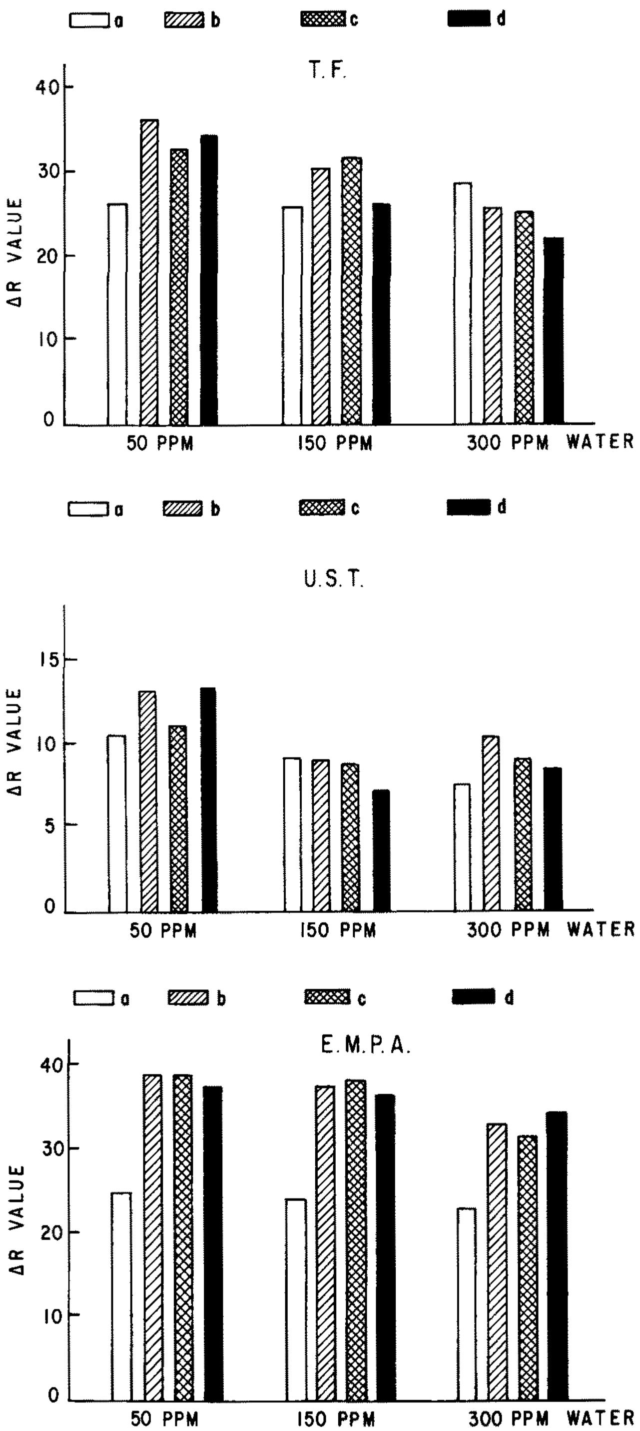

FIG. 11. *Comparative detergencies* (ΔR) of standard detergent and ternary system: A. T.F. = Test fabrics soiled cotton fabric; B. U.S.T. = U.S. Testing Co. soiled cotton fabric; C. E.M.P.A. = EMPA 101 soiled cotton fabric. Soap-NaMeα-ST-Na_2SiO_3 at 0.2% total concentration and 60 C: a = standard detergent; b = 80% soap, 10% NaMeα-ST, 10% Na_2SiO_3; c = 70% soap, 20% NaMeα-ST, 10% Na_2SiO_3; d = 70% soap, 10% NaMeα-ST, 20% Na_2SiO_3.

indicated. When the builder does not enhance the detergency of a given soap-LSDA system, the component ratio entry shows the builder concentration as zero, i.e., a detergency maximum is attained with a mixture of soap and LSDA alone. As Table II shows, the detergency of the soap-NaMeα-ST system can be enhanced by various builders such as sodium tripolyphosphate, silicate, citrate, NTA and disodium oxydiacetate. Sodium carbonate proved to be an ineffective builder for this detergent system. On the other hand a soap-LAS system cannot be potentiated by either sodium silicate or sodium tripolyphosphate. Since LAS is a

TABLE III

Effect of Silicate Additives on Detergency of Soap—LSDA Systems[a]

Lime soap dispersing agent sodium salt	Soap 80% + LSDA 10% + silicate 10%			
	SiO_2/Na_2O Ratio			
	1:1[b]	1.6:1[c]	1.8:1[d]	2.0:1[c]
2-Sulfoethyl tallowate	37.1	41.6	37.0	34.5
Methyl α-sulfotallowate	34.3	39.7	36.5	35.9
Sucrose α-sulfotallowate	33.6	38.7	35.9	35.2
N-Methyl N-(2-sulfoethyl) tallowamide	31.6	36.2	35.1	35.1
Sulfated ethoxylated tallow alcohol (3.3 moles of EO)	35.4	36.9	34.4	33.7
Sulfated N-(2-hydroxyethyl) tallowamide	35.2	41.5	35.0	35.0
Soap 90% + silicate 10%	31.5	36.8	35.1	27.8

[a]0.2% concentration in 300 ppm water at 60 C; ΔR; Test fabrics cloth, lot 2.
[b]Sodium meta silicate = metso.
[c]B-W.
[d]Starso.
[e]GD.

relatively inefficient lime soap dispersant, a 60% soap-40% LAS ratio is required for maximum detergency.

A sulfated oxyethylated tallow alcohol proved to be as effective an LSDA as NaMeα-ST and responded to building with sodium metasilicate. A typical nonionic surfactant, a 10 mole EO adduct to *n*-hexadecanol, exhibited an LSDA behavior in a ternary soap-LSDA-builder system similar to that of NaMeα-ST.

Sodium silicates are known to be efficient and inexpensive detergent builders whose effectiveness depends upon the Na_2O/SiO_2 ratio, and it seemed appropriate to investigate this aspect of the ternary soap systems in some detail. Thus silicates of SiO_2/Na_2O ratios varying from 1:1-2:1 were examined in soap-LSDA mixtures for optimum detergency, and results are listed in Table III. The most effective SiO_2/Na_2O ratio found was 1.6:1. Because of the high alkalinity of soap, ratios above 2:1 were not studied.

The soap-LSDA-builder system, soap-NaMeα-ST-Na_2SiO_3, was compared (see Fig. 11) in washing ability to a well known commercial heavy duty granular detergent at 0.2% of concentration in 50, 150 and 300 ppm hard water on three soiled cloths, Testfabrics, U.S. Testing Co. and EMPA. Although a complete ternary study was made as reported above, only the formulations of maximum deter-

TABLE IV

Multi-Wash Tests: Soap-Lime Soap Dispersant Systems[a]

Experiment	Formulations	-ΔR Soiling	-ΔR Redeposition
	Control values		
	Cloth original value 90.5		
1	Soap 100%	29.7	12.1
2	LAS 20% + builder[b] 80%	22.4	17.4
	Binary systems soap 80% + LSDA 20%		
3	Sodium methyl α-sulfotallowate	14.0	7.9
4	Sodium sulfated N-(2-hydroxyethyl) tallowamide	14.5	7.5
5	Sodium sulfated ethoxylated tallow alcohol[c]	15.3	8.0
6	Sodium 2-sulfoethyl tallowate	17.5	8.1
	Ternary systems soap 80% + LSDA 10% + builder 10%		
7	Sodium sulfated ethoxylated tallow alcohol[c] 10% + Na_2SiO_3 10%	12.0	6.9
8	NaMeα-ST 10% + Na_2SiO_3 10%	13.2	7.9
9	NaMeα-ST 10% + sodium citrate 10%	14.0	8.6
10	NaMeα-ST 10% + sodium oxydiacetate 10%	13.1	8.7
11	NaMeα-ST 10% + Na_2CO_3 10%	13.6	8.2
	Ternary systems of varying ratios of soap and LSDA + Na_2SiO_3		
12[d]	Soap 80% + NaMeα-ST 10% + Na_2SiO_3 10%	13.2	7.1
13	Soap 70% + NaMeα-ST 10% + Na_2SiO_3 20%	13.0	6.7
14	Soap 60% + NaMeα-ST 10% + Na_2SiO_3 30%	14.2	7.1
15	Soap 80% + sodium sulfated ethoxylated tallow alcohol[c] 10% + Na_2SiO_3 10%	12.4	7.0
16	Soap 70% + sodium sulfated ethoxylated tallow alcohol[c] 10% + Na_2SiO_3 20%	12.3	7.1
17	Soap 60% + sodium sulfated ethoxylated tallow alcohol[c] 10% + Na_2SiO_3 30%	12.1	6.9

[a]300 ppm hard water; 60 C; concentration 0.2% + 0.002% of CMC.
[b]Builder contains 10% $Na_4P_2O_7$, 55% $Na_5P_3O_{10}$, 1% CMC, 10% Na_2SiO_3 and 24% Na_2SO_4.
[c]3.3 Moles EO.
[d] Replicate run of experiment 8.

gency of 80:10:10, 70:20:10 and 70:10:20 of soap-LSDA-builder are shown in the figure. The observed ΔR values were equal or greater than those for the commercial detergent for all three soiled cloths except in the case of formulation d, which showed some slight deficiencies, and in the case of all formulations at 300 ppm hardness using Testfabrics cloth where the commercial detergent was superior to the soap formulations.

The Schwartz and Berch multiwash test (10) was used to confirm the good detergency of the most promising formulations. Table IV lists the results with several of the binary and ternary systems, as well as with a phosphate built LAS, showing both detergency and redeposition data. Since in this procedure clean fabric is used as the start, the detergency values represent the loss in whiteness due to dirt buildup (soiling) which the detergent was not able to remove. The net result is a decrease in reflectance (-ΔR) over the original reflectance (90.5%) of the unsoiled cotton fabric. Redeposition is likewise expressed in reflectance loss (-ΔR) due to dirt pickup of the unsoiled swatches from the wash water. Table IV shows that binary systems containing soap and LSDA, such as sodium methyl α-sulfotallowate, the sodium salt of N-(2-hydroxyethyl) sulfated tallow amide, the sodium salt of sulfated oxyethyl (3EO) tallow alcohol and sodium 2-sulfoethyl tallowate, were superior to soap alone as well as to built LAS with respect to both soiling and redeposition values. Detergency was further improved when small amounts of builders were added to the soap-LSDA systems. This was observed for sodium metasilicate, sodium citrate, sodium oxydiacetate and sodium carbonate. Carbonate did not show much of a contribution to detergency in the single wash tests (Table II). This points out that contradictory detergency results can be obtained through the use of different laboratory techniques. On the other hand we were able to obtain superior detergency and redeposition data by the multiwash technique of several ternary systems (Experiments 12-17) which also gave superior detergency by the single wash method.

Although it is difficult to extrapolate from laboratory detergency data to actual home washing conditions, the results of the above experiments indicate that some soap-LSDA or soap-LSDA-builder systems can be effective detergents in hard or soft water. Lime soap dispersing agents not only prevent the precipitation of lime soap curds in the washing and rinsing cycles but also improve the detergency of soap. Detergency can be further enhanced by the addition of small quantities of certain builders, such as sodium tripolyphosphate, sodium silicate, sodium diglycolate and sodium citrate, at levels well below those required to sequester all of the calcium and magnesium in hard water.

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