obtained in Run 2. Analyses of the laboratory stripped oil from Run 2 are: refined color, 35 Y, 5.32 R; bleached color, 20 Y, 1.19 R; lowest refining loss 6.49%. Refinings were done by A.O.C.S. Official Method CA 9a-52 and bleaching tests were made in accordance with A.O.C.S. Official Method Cc 8a-52.

In Runs 4 and 5 free gossypol was reduced in the marc by extraction with butanone to 0.01%, and total gossypol was reduced to 0.14% and 0.19%, respectively. It was shown in Runs 4 and 5, using cottonseed from the same lot, preparing the flakes the same, using the same solvent and the same solvent rate, that the percentage free and total gossypol in the extracted meal from Run 5 was as high as that from Run 4 although twice the quantity of butanone was used and the time of extraction (including soaking time) was more than tripled.

Cost

Material and equipment for the plant cost $15,511 and the installation was $6,512.

Summary and Conclusions

A new improved batch solvent extraction plant and its versatility of design have been described. Data have been given on the cost and initial test operations. The performance of the plant has been tested, and its value as a research tool has been demonstrated by the production of free gossypol and meals low in gossypol for use in research. Simplicity of operation is borne out by the fact that only one operator is needed. A limited number of detail drawings of the plant are available on written request from the Southern Regional Research Laboratory.

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The Effect of Electrolytes on Soil Redeposition in Laboratory and Laundry Practice

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J. POWNEY and R. W. Noad (1) examined the deposition of ilmenite black on cotton fabric in the presence of soap, synthetic detergents, and certain sodium salts, such as chloride, carbonate, hydroxide, silicate, and phosphate. They found that soap gave good protective action which decreased with rise in pH and with the addition of sodium chloride or carbonate. Synthetic detergents of the long chain alkyl sulfate type were found to possess low protective action. Similar conclusions were reached by Weatherburn *et al.* (2, 3), using a similar procedure.

To ascertain the contribution of redeposition of soil in total detergent action, deposition of carbon from Aquadag suspensions on cotton cloth was examined in the presence of mono- and divalent cations as well as some protective colloids. Using solutions of the same compositions, these results were correlated with multiple-cycle soiling and washing on hand towels under ordinary home-laundry conditions. The results of Powney and Noad were confirmed and extended.

In the present work synthetic anionic detergents exhibited an inferiority in the laundering of cottons as compared to ordinary soap. It was demonstrated that synthetic detergents wash more poorly in hard than in soft water. Improvement in hard water detergency was noted when synthetics were mixed with phosphate and when certain protective colloid agents were added. The latter also improved soft water detergency. Soap did not show a washing deficiency in hard water, a fact which was apparently due to the removal of hard water ions of calcium and magnesium as insoluble fatty acid salts while an excess of sodium soap remained. Builder salts used either with soap or detergent enhanced soil removal but at the same time tended to increase soil deposition from the wash liquor. The present work also showed that a most important function of builders in the washing process is to suppress the effect of calcium or magnesium ion.

Experimental

I. Soil Deposition Test. A suspension was made containing 0.5 g. of Aquadag[1] in 1 liter of solution containing the salts or detergent systems to be examined. The Aquadag used in these experiments was a 22% solids paste of colloidal graphite in water containing a small amount of suspending agent. The density of the paste was 9.35 lbs./gal. The fineness of the Aquadag particles was arbitrarily designated by the letter "A," which means a maximum particle size of 4 microns with the majority of particles either 1 micron or less and the average size of particles over 1 micron being 2 microns. To the salt- or detergent-Aquadag suspension was added a clean cotton swatch (6 in. x 6 in.), which was stirred in a Tergotometer

[1]Acheson Colloids Corporation, Port Huron, Mich.

type apparatus at 110°F. for 20 minutes. Swatches were removed, rinsed, and dried, and reflectance was read on a Hunter Multipurpose Reflectometer. This reading was compared with the original reading of the unsoiled swatch. The difference gave a measure of units of soil deposited.

II. Practical Towel Test. Ordinary commercial hand towels were supplied to a number of housewives or placed in plant wash rooms and soiled during normal use. These towels were collected at regular intervals and laundered in the laboratory under controlled conditions in a regular washing machine. After washing, the towels were returned to the housewife or washroom for re-soiling, and the cycle was repeated for 10 successive soilings and launderings. Towels used in the test were measured for brightness at the beginning of the test (that is, before setting them out for the first soiling) and again at the completion of the test (after the final and 10th laundering) with a Hunter Reflectometer in the manner described by W. E. Thompson (4). Two towels (called "control" towels for convenience), retained in the laboratory but added to a given load of soiled towels at each laundering, were used as a measure of the pick-up of redeposited soil from the wash liquor. These were also measured for reflectance at the beginning and end of the test. The control towels received as many launderings as the soiled test towels but were never set out for soiling. Comparison of the initial or original reading and the final reading after the 10th laundering of the control towels gave a measure of redeposited soil. Comparison between the original and final reading of the regular test towels gave a measure of the total or net washing of the system in question.

TABLE I

Reproducibility of Soil Deposition Results

	Units of Soil Deposited	
	0.3% Soap	0.25% Detergent
Test No. 1	6.3	27.3
Test No. 2	6.7	25.9
Test No. 3	6.5	25.4
Test No. 4	6.9	25.4
Test No. 5	8.3	25.9

Results and Discussion

I. Aquadag Deposition Tests. Table I illustrates the order of reproducibility of results of the Aquadag deposition test. It may be seen that this method gave fairly dependable results.

Deposition values from Aquadag suspension were obtained for some pure and commercial soaps, some commercial synthetic detergents, and certain inorganic sodium salts. These data are given in Tables II and III. The higher the numerical value given in the tables, the blacker the swatch, and hence the greater the deposition. It may be seen that both built and unbuilt soaps suspend Aquadag soil effectively. Among the saturated soaps better soil suspension was obtained with the higher molecular weight members. It was noteworthy that built soaps and commercial synthetic detergents deposited more soil with increasing concentration. Extremely poor soil suspension was obtained with sodium chloride, carbonate, tetraborate, pyrophosphate, and metasilicate. Ordinary commercial synthetic detergents containing sodium sulfate were only moderately effective in suspending soil. The average soil deposition values for the groups of materials given in Tables II and III were plotted in Figure 1 and show the general soil deposition

TABLE II

Effect of Soaps and Salts on Soil Deposition in Distilled Water[a]

	Units of Soil Deposited		
	"As Is" Concentration		
	0.05%	0.30%	0.55%
Sodium laurate	13.5	11.1	12.8
Sodium myristate	7.9	9.1	9.2
Sodium palmitate	4.9	2.5	0.3
Sodium stearate	3.5	2.7	2.1
Sodium oleate	3.0	5.3	6.6
Unbuilt commercial soap "A"	3.8	6.5	8.2
Unbuilt commercial soap "B"	3.9	6.7	5.6
Built commercial soap "A" (70% soap)	8.8	11.6	24.8
Built commercial soap "B" (70% soap)	3.9	11.9	14.5
Built commercial soap "C" (70% soap)	4.6	11.6	12.4
Built commercial soap "D" (85% soap)	4.9	7.9	9.8
Sodium carbonate	48.8	55.8	55.5
Sodium metasilicate	40.7	47.8	46.0
Tetra-sodium pyrophosphate	46.9	55.6	56.5
Sodium tetraborate	46.8	54.5	56.0
Sodium chloride	57.9	57.2	58.8

[a]Aquadag suspension alone deposits 47.3 units of soil in distilled water.

TABLE III

Effects of Commercial Synthetic Detergents[a] on Soil Deposition in Distilled Water[b]

	Units of soil Deposited		
	"Active Ingredient" Concentration		
	0.0125%	0.100%	0.200%
Nacconol NR (approx. 40% active, 60% salt)	......	31.5	......
Na salts of sulfated coconut monoglycerides (approx. 35% active, 65% salt)	12.4	28.9	31.8
Na salts of sulfated coconut-tallow monoglycerides (approx. 35% active, 65% salt)	10.1	30.1	34.4
Igepon-T (approx. 35% active, 65% salt)	......	29.2	......

[a]Detergents employed were commercial products and contained neutral salts in indicated amounts.
[b]Aquadag suspension alone deposited 47.3 units of soil.

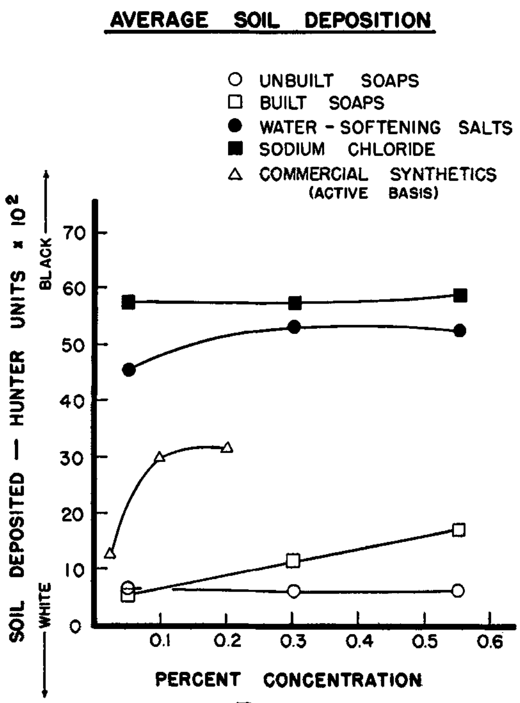

FIG. 1

TABLE IV

Effect of Electrolytes and Non-Electrolytes on Soil Deposition in Distilled Water Detergent Systems

	Units of Soil Deposited				
	Concentration of Organic Detergent				
	0.0125%	0.05%	0.10%	0.20%	0.50%
Anionic detergent, salt-free	7.3	5.2	6.6	8.0	13.6
35% detergent + 65% Na_2SO_4	12.2	21.1	25.6	29.7	32.3
Sodium oleate	5.1	4.1	4.4	5.1	7.8
35% sodium oleate + 65% Na_2SO_4	9.3	12.1	14.5	19.0	23.5
35% detergent + 65% sodium carbonate	......	......	19.5	......	......
35% detergent + 65% tetrasodium pyrophosphate	......	......	17.7	......	......
35% detergent + 65% sodium metasilicate	......	......	13.8	......	......
35% detergent + 65% carboxymethyl cellulose	......	......	2.3	......	......
35% detergent + 65% corn starch	......	......	7.7	......	......

characteristics of various types of soaps, commercial detergents, and inorganic salts.

Examination of salt-free synthetic detergent solutions in soil deposition tests was then made. For this work a quantity of salt-free detergent (sodium salts of sulfated coconut monoglycerides) was prepared by extraction of a commercial material with water-isopropanol as solvent. The resulting salt-free detergent material and also a sample of sodium oleate (99% pure soap) were then examined in soil suspension tests. Tests were run on these materials in distilled water solution over a wide range of concentration, at first alone, then mixed with increasing amounts of a typical monovalent electrolyte, such as sodium sulfate. Tests were also run on mixtures of detergent with added water-softening salts as well as with certain materials, such as starch and carboxymethyl cellulose. The results are given in Tables IV and V.

TABLE V

Effect of Increasing Salt Concentration on Soil Deposition

0.1% Distilled Water Solutions Na Salts of Sulfated Coconut Monoglycerides	
% Na_2SO_4 Added	Units of Soil Deposited
0.000	6.6
0.030	14.5
0.112	21.1
0.186	25.6

These data showed that sodium oleate and the sulfated anionic detergent behaved similarly with respect to deposition of soil. When each was examined alone, that is with no added electrolyte, low soil deposition values were obtained. When the same amounts of sodium sulfate were added to both soap and detergent, much higher soil deposition values were obtained. Carboxymethyl cellulose mixed with detergent reduced soil deposition values while corn starch did not affect the soil deposition values of the original detergent solution.

The effect of the common divalent ions commonly present in natural hard waters on the soil deposition values of some of the previous detergent systems was next examined. Figures 2 and 3 show that soil deposition values increased by addition of either sodium sulfate or salts of calcium and magnesium to 0.1% solutions of sodium salts of sulfated coconut monoglycerides. As much soil was deposited by small concentrations of hard water salts of the order of 0.1% as was obtained by a 0.2% addition of sodium sulfate. These results showed that a synthetic detergent which forms relatively soluble calcium and magnesium salts exhibited a pronounced weakness for suspending soil in hard water. Further it was apparent that synthetic detergents required protection against divalent hard water ions to make possible satisfactory washing in hard water. Soap is self-protecting in respect to hard water ions. With soap, the foam point in hard water also approximately corresponds with the point where practically all the calcium and magnesium ions have been effectively removed by

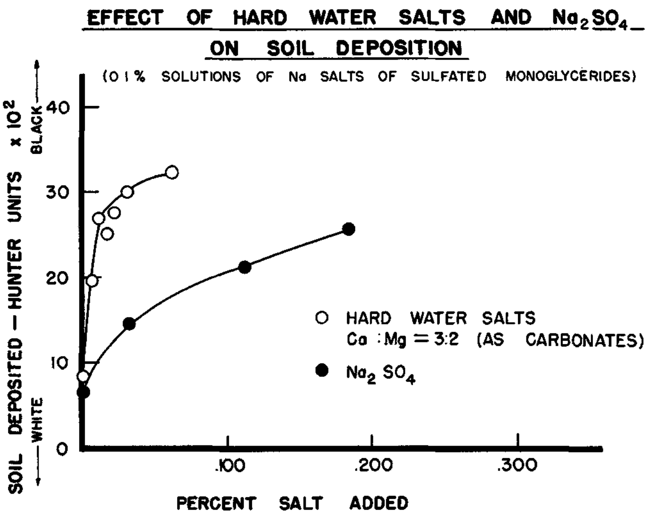

FIG. 2

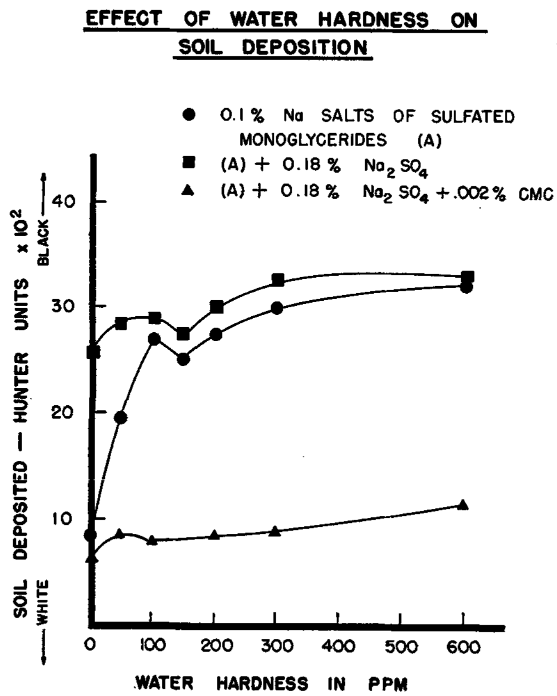

FIG. 3

precipitation. Thus in actual practice the housewife using soap probably never launders in solutions containing any appreciable amount of free Ca^{++} or Mg^{++} even in extremely hard water since she uses the presence of foam as a guide to the proper amount of soap to employ for washing.

Some water-softening builder salts were examined in 0.1% solutions of the sodium salts of sulfated coconut monoglycerides in hard water (Table VI).

TABLE VI

Effect of Water-Softening Salts on Soil Deposition in Hard Water

0.1% Solutions of Sodium Salts of Sulfated Coconut Monoglycerides Plus 0.18% of the Following Materials	Units of Soil Deposited
Detergent, no additions	24.6
Sodium metasilicate	19.4
Tetrasodium pyrophosphate	14.9
Calgon	18.8
Soda ash	14.4

Also the effect of sodium tripolyphosphate on soil deposition of a synthetic detergent in hard water was examined and compared to sodium sulfate. Figure 4 is a plot of the data obtained and shows that tripolyphosphate reduces soil deposition in hard water in the presence of a detergent.

II. Practical Towel Tests. It was found that the trend of redeposition results obtained in these practical towel tests is in the same direction as the Aquadag soil deposition tests. The results showed that soap is an excellent laundering agent compared to the synthetic detergents examined with respect to net washing and especially in regard to deposited soil (Table VII).

The improvement in soil deposition is quite apparent when a water softening agent such as sodium tripolyphosphate, for example, is used in conjunction with a detergent. Improvement in soil deposition is also noted when CMC is used in a detergent mix (Table VIII).

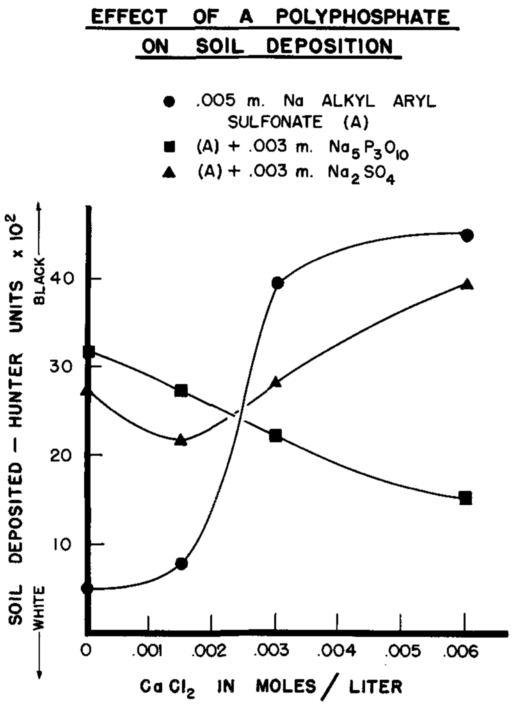

FIG. 4

TABLE VII

Practical Laundering Tests

Reflectance of Towels After 10 Cycles of Soiling and Laundering[a]

	"Test" Towels		"Control" Towels (Redeposited Soil)	
	Soft Water 50 p.p.m.	Hard Water 300 p.p.m.	Soft Water 50 p.p.m.	Hard Water 300 p.p.m.
A) 0.0875% Na salts of sulfated monoglycerides	71.0	67.2	77.7	71.7
B) 0.0875% — Na salts of sulfated monoglycerides + 0.1625% Na_2SO_4	70.3	67.5	74.4	71.3
C) B + 0.0175% CMC	......	71.0	......	78.3
D) 0.55% commercial built soap[b]	......	75.0	......	78.2

[a] Reflectance of original towels = 80.5.
[b] 70% Sodium soap.
14% Tetrasodium pyrophosphate.
7% Sodium silicate.
9% Soda ash.

TABLE VIII

Practical Laundering Tests

Reflectance of Towels After 6 Cycles of Soiling and Laundering[a] 0.4% Total Concentration — 300 p.p.m.

	"Test" Towels	"Control" Towels (Redeposited Soil)
Series I		
A) 25% Na alkyl sulfate + 75% Na_2SO_4	61.9	67.6
B) 25% Na salts of sulfated monoglycerides + 75% Na_2SO_4	61.2	65.3
Series II		
AA) 25% Na alkyl sulfate + 75% $Na_5P_3O_{10}$	66.3	71.1
BB) 25% Na salts of sulfated monoglycerides + 75% $Na_5P_3O_{10}$	67.1	71.6
Series III		
Unbuilt soap	70.0	73.7

[a] Reflectance of original towels = 76.5.

Conclusions

1. Fatty acid soaps and pure synthetic detergents have a far greater suspending power for Aquadag soil than either of these types when used in the presence of inorganic salts.
2. Any inorganic salts of the type of sodium chloride, sodium sulfate, calcium, or magnesium salts, the common alkali or phosphate builders, all tend to cause deposition when used alone or with surface-active materials.
3. Fatty acid soaps are self-protecting against hard water salt effects.
4. Synthetic detergents require protection against calcium and magnesium salts, which can be achieved by adding calcium ion suppressants. The effect of neutral salts, such as sodium chloride and sodium sulfate, can be eliminated by omitting them from the composition. When these salts are present in minor amounts, the use of protective colloids, such as carboxymethyl cellulose, affords some protection.

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