Alkyldimethylamine Oxides as Synergistic Fabric Softeners

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The interaction of solid alkyldimethylamine oxide and ditallowdimethylammonium chloride (DTMAC) and ditallowdimethylammonium sulfate (DTMAS) quats in representative types of fabric softener systems was studied with particular focus on synergistic behavior. Softening, whiteness retention, wetting, static build-up and detersiveness were evaluated for laundry rinses, laundry detergents and dryer sheets. In laundry rinses, blends of amine oxide and DTMAC proved to be synergistic for improving the wetting of cotton towels. Although no synergism was observed in laundry detergents, formulations containing amine oxide gave better detersiveness than systems with DTMAC without the splotching associated with the quaternary salt. In dryer sheets, it was discovered that blends of amine oxide and DTMAS gave synergistic softening of cotton towels and were unexpectedly effective in preventing static charge build-up on polyester fabric.

KEY WORDS: Amine oxide, detergent, dryer sheet, fabric softener, high active solid, quat, rinse, synergy.

Fabric softeners are used in a variety of forms in consumer applications. They may be incorporated into dryer sheets, used as a post-wash laundry rinse, or may actually be incorporated into the detergent itself. In these applications, softeners actually do more than just soften fabric (1). They also give the fabric greater bulk, improved ease of ironing, reduced charge build-up and decreased fabric drying time.

Ditallowdimethylammonium chloride (DTMAC) is the major class of fabric softener in use today (1). While an effective and inexpensive softener, DTMAC possesses several performance deficiencies, including yellowing of fabric, poor rewetting of treated fabric, inefficient antistatic activity on polyester, reduced washability of softened fabric and incompatibility with the anionic surfactants commonly used in laundry detergents (2). Additionally, the adsorption of this fabric softener to activated sludge is of growing concern to environmental groups in Europe (3). The ability of quaternary ammonium salts to survive anaerobic digestion systems also presents the potential for their build-up under the anaerobic conditions likely in landfills containing solid waste from sewage treatment plants (3).

Amine oxides are reported to be effective softeners in their own right and are also known to be adjuvants that improve rewettability when used in conjunction with DTMAC (4–6). However, a systematic study of amine oxide and DTMAC blends for performance characteristics has not been reported. Previously, the dilute nature of amine oxides tended to limit their use to liquid formulations. Now, a newly developed, highly active form of amine oxide makes it practical to incorporate amine oxide into laundry detergent powders and dryer sheets.

The research described herein addresses the interaction of solid alkyldimethylamine oxide with DTMAC and ditallowdimethylammonium sulfate (DTMAS) in representative softening systems with particular focus on synergistic behavior. The work covers softening, whiteness retention, wettability, static control and detersiveness in laundry rinses, laundry detergents and dryer sheets.

MATERIALS AND METHODS

Reagents. The alkyldimethylamine oxide was supplied by Ethyl Corporation (Baton Rouge, LA), the ditallowdimethylammonium chloride was obtained from Akzo Chemie (Chicago, IL), and the ditallowdimethylammonium methyl sulfate was obtained from Sherex Chemical Company (Dublin, OH).

Fabric. Medium-grade cotton terrycloth towels were obtained from Best Values Textiles, Denver, CO. Polyester and 35% cotton/65% polyester sheeting for static control testing was obtained from Testfabrics, Middlesex, NJ. Untreated polyester dryer sheets were provided by Reemay, Inc., Old Hickory, TN. The fabric was treated in an automatic washer (model A940) and automatic dryer (model DE9800) from Maytag (Newton, IA).

Evaluation methodology. CSMA test protocols were followed for the preparation and evaluation of treated fabric (7): fabric stripping (Method DCC-13E), fabric treatment (Method D-13A), softness evaluation (Method D-13B), whiteness evaluation (Method D-13C), rewettability (Method D-13D) and static control (Method D-13F). Panel tests for softness and whiteness were conducted as a double-blind study for 90 data points/evaluation.

Treatment parameters were selected to represent practical conditions. Washes were conducted at a medium loading at 38°C and 150 ppm as calcium carbonate water hardness (3Ca/2Mg).

Fabric treatment. For the laundry rinse study, ten hand-towels were washed on medium loading for 30 min in the presence of 37.5 g of an anionic laundry detergent. During the rinse cycle, sufficient softener to correspond to 0.1% towel weight was added. A drying time of 60 min on "normal" at about 65°C was used.

For the detergent study, ten hand-towels were washed on medium loading for 30 min in the presence of 37.5 g of a test detergent formulation containing 5% fabric softener. A drying time of 60 min on "normal" at about 65°C was used.

For the dryer sheets, ten hand-towels were washed on medium loading for 30 min in the presence of 37.5 g of an anionic laundry detergent. After adding the wet towels to the dryer, a polyester dryer sheet with a 1 g softener loading was added on top of the towels. A drying time of 60 min on "normal" at about 65°C was used.

Panel test for softness and whiteness. Thirty panelists wearing dark sunglasses were asked to evaluate five towels treated with different softening formulations, ranking them alternately "harshest" then "softest". After removing the glasses, panelists then ranked towels alternately "whitest" and "least white". The softness and whiteness testing was conducted as a triplicate evaluation.

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Rewettability. After four wash/rinse/dryer cycles, towels were cut into five-by-six-inch strips and marked one centimeter from the narrow edge. Each strip was then lowered to the mark into a 0.01% Rhodamine B dye bath for six minutes, after which time the distance of dye movement was recorded in centimeters.

Static control. After a single washer/dryer cycle, the separate pieces of the laundry bundle (23% polyester, 46% cotton/polyester, 31% cotton) were removed from the dryer and dropped into a Faraday cage. The initial voltage was recorded. For the rinse cycle testing, the relative humidity was 35% at 22°C. For the dryer sheet testing (on a different day), the relative humidity was 50% at 22°C.

RESULTS AND DISCUSSION

Consumers have come to rely upon softening products to provide not only softness but also static control and "freshness" to their laundry. Since the consumer is the ultimate judge of the effectiveness of a fabric softener, any evaluation of fabric softeners must use consumer perception as the standard by which efficacy is measured. Accordingly, panel tests were a key evaluation in our study of amine oxides in laundry rinses, laundry detergents and dryer sheets.

Laundry rinses. Although the goal of this part of our study was the interaction of DTMAC with amine oxide as a rinse softener, first it was necessary to select the optimum amine oxide homolog. Accordingly, octyldimethylamine oxide (C8 AX), tetradecyldimethylamine oxide (C14 AX), and octadecyldimethylamine oxide (C18 AX) were selected and compared as softeners for cotton terrycloth towels. As controls, unsoftened towels and towels treated with a standard softener, DTMAC, were included in the study.

The relative softening ability of these compounds was determined in extensive panel tests and ranked from softest, 5, to least soft, 1. In these tests, a difference of ±0.3 in the panel rankings fell within the 95% confidence level range. After a single wash/rinse cycle, the best softeners, DTMAC and C18 AX, were equivalent to each other. The C14 AX was ranked by panelists as the next best softener followed by C8 AX, which was indistinguishable from the untreated system (Fig. 1). After four wash/rinse cycles, DTMAC was ranked the best softener, closely followed by C18 AX and C14 AX, which were now equivalent to each other. At this stage, panelists were finally able to differentiate the C8 AX from untreated towels. Gradual build-up of the softening agents is a possible explanation for the changes in relative ranking between a single and repeated wash/rinse cycle.

During the course of the softness evaluations, panelists observed and scored differences in whiteness of the test materials from whitest, 5, to least white, 1, with a variation of ±0.5 for a 95% confidence level. The results after one cycle (Fig. 2) showed the untreated towels to be whiter than softened towels. All treated towels gave comparable whiteness retention. However, the results were quite different after four cycles. The amine oxide homologs were commensurate with the untreated sample. DTMAC-treated towels were the least white and, in fact, were actually described by some panelists as "tan".

The wettability of towels from the four wash/rinse cycles was then measured. The results (Fig. 3) indicated that the C18 AX was equal to the untreated towel. Unexpectedly, the C8 and the C14 amine oxides exhibited improved wetting, with C8 AX being clearly the best by far. Towels treated with DTMAC showed poorer wettability than even the untreated towels.

Based on the above results, the C18 AX was selected for further investigation in conjunction with DTMAC. As a first step, various blends of C18 AX and DTMAC were evaluated as softeners for cotton terrycloth towels. In terms of softening, after one and four (Fig. 4) wash/rinse cycles, certain blends of C18 amine oxide with "quat" were found to be mutually antagonistic: 75% C18 AX/25% DTMAC and 50% C18 AX/50% DTMAC. In the first cycle, the 25% C18 AX/75% DTMAC blend was indistinguishable from the pure C18 AX and DTMAC. This changed after four cycles to the point that DTMAC was ranked higher than either the pure C18 AX or the 25% C18 AX/75% DTMAC blend.

After one cycle (Fig. 5), all treated towels gave comparable whiteness retention except DTMAC, which once again produced towels that were visibly "dingy". Whiteness retention after four cycles remained greatly improved by blending amine oxide with DTMAC.

Unexpectedly, all blends of the C18 AX proved to be synergistic with DTMAC for wetting of the treated test materials after four cycles (Fig. 6), with all blends showing superior wettability relative to either C18 AX or DTMAC by themselves. Since the C8 AX was a better wetting agent than the C18 AX, we also examined C8 AX's interaction with DTMAC. Contrary to our expectations of synergism, though, the C8 AX interacted with DTMAC in an additive fashion for improved wetting.

Continuing with the study of C18 AX/DTMAC interactions, the ability to control static charge build-up on selected fabrics was measured (Fig. 7). The efficiency of static reduction varied greatly with both the type of fabric and formulation. On polyester, the most effective materials were C18 AX and DTMAC, which were equivalent. All blends showed some measure of static reduction, which varied with blend composition. On cotton, DTMAC was the most efficient static control agent. C18 AX by itself appeared to be inactive at controlling static on cotton. Blends of C18 AX and DTMAC were more effective than expected if the components were acting in a strictly additive fashion. No static control was evident for any agents tested on 35% cotton/65% polyester material. We are unable to explain this result. For a laundry bundle consisting of 23% polyester, 46% cotton/polyester, and 31% cotton, DTMAC and two blends (75% C18 AX/25% DTMAC; 50% C18 AX/50% DTMAC) were the most effective and indistinguishable from each other.

Laundry detergents. Although rinse softeners were the most widely used type of formulation by our panel, several of them noted the inconvenience of adding softener to the rinse cycle. They felt that a combined detergent/softener package would be more desirable. Many had tested the combination packages currently on the market but were dissatisfied with the performance afforded by these products. Presumably, this deficiency was a consequence of the mutual incompatibility of many cationic and anionic detergent components. Therefore, incorporation of a nonionic softener such as an amine oxide into a detergent seemed to be a logical next step in this program.

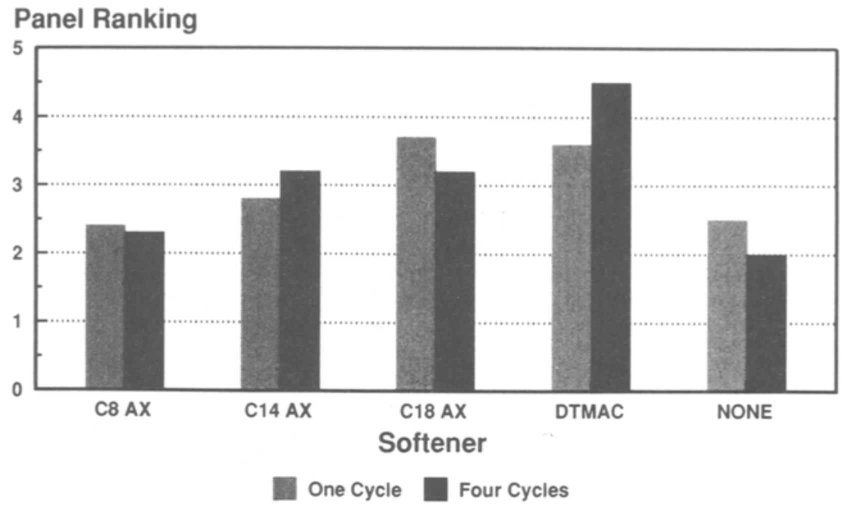

FIG. 1. Laundry rinse: softness rating. Variability = ±0.3 for 95% confidence level.

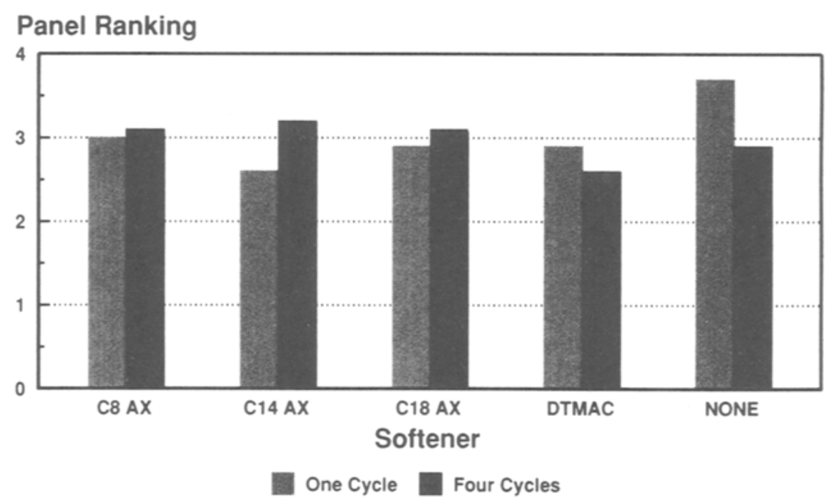

FIG. 2. Laundry rinse: whiteness rating. Variability = ±0.5 for 95% confidence level.

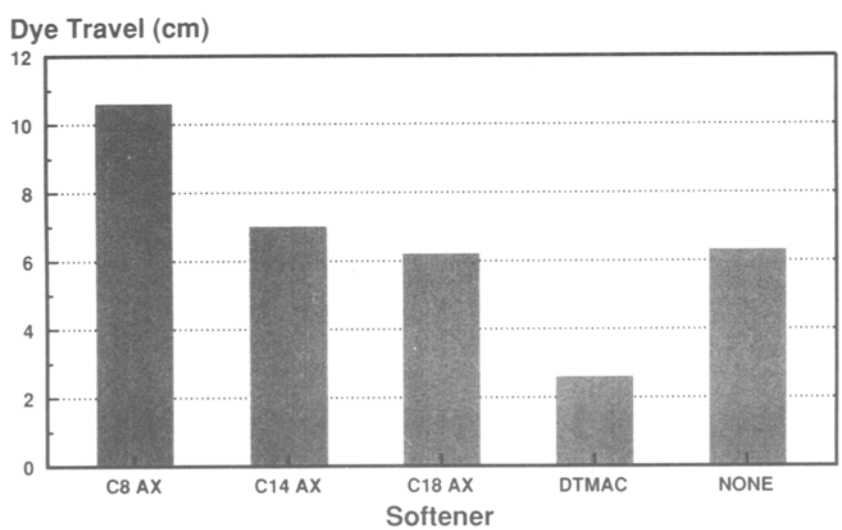

FIG. 3. Laundry rinse: wetting rating after four cycles.

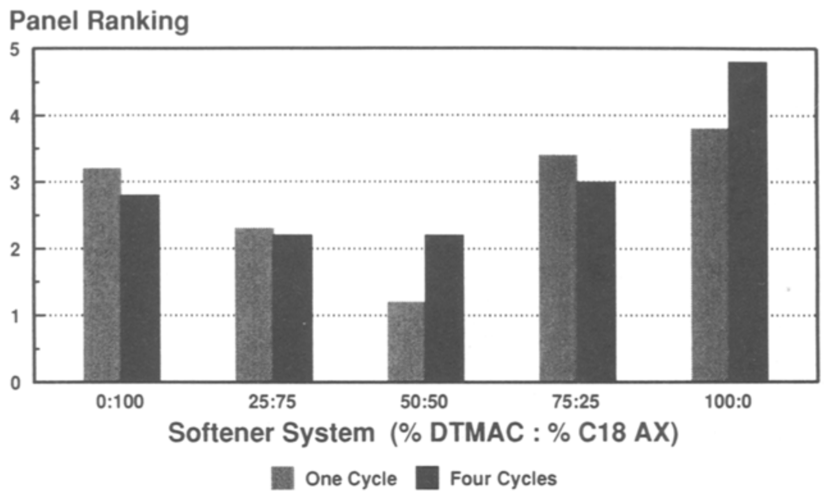

FIG. 4. Laundry rinse synergy: softness rating. Variability = ±0.3 for 95% confidence level.

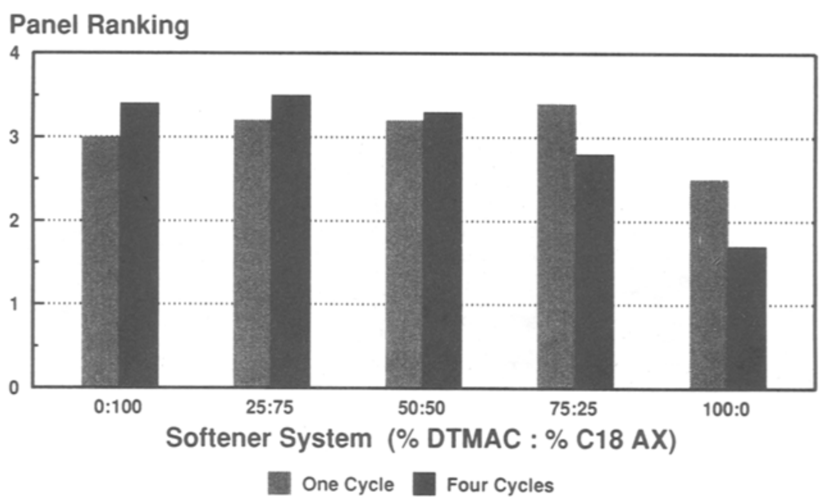

FIG. 5. Laundry rinse synergy: whiteness rating. Variability = ±0.5 for 95% confidence level.

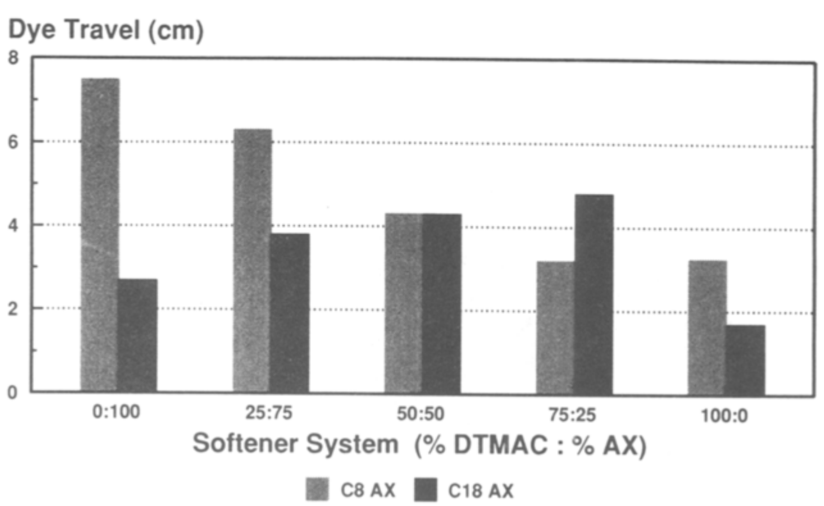

FIG. 6. Laundry rinse synergy: wetting rating after four cycles.

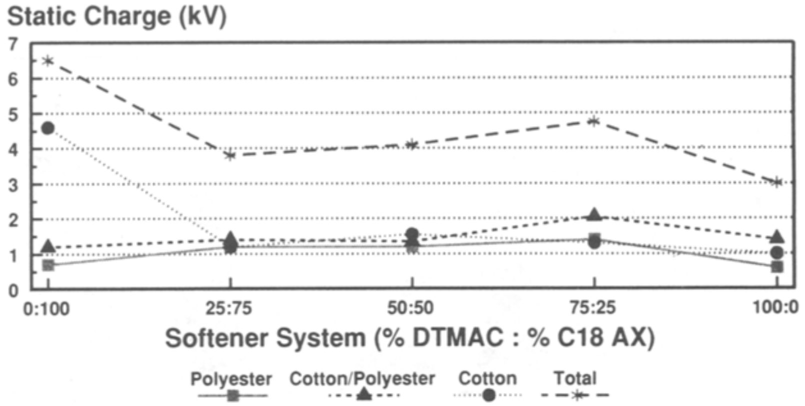

FIG. 7. Laundry rinse synergy: antistatic activity after one cycle. Values for untreated fabrics are: polyester, 2.63 kV; cotton/polyester, 1.01 kV; cotton, 4.09 kV.

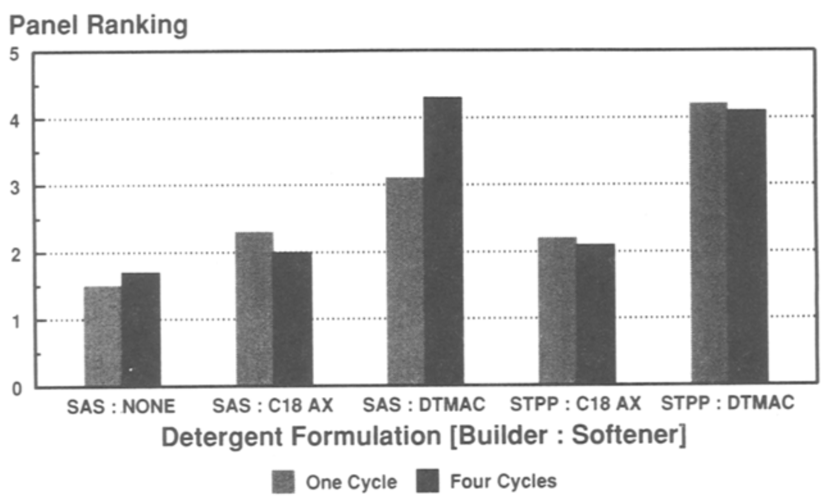

FIG. 8. Laundry detergent: softness rating. Variability = ±0.3 for 95% confidence level.

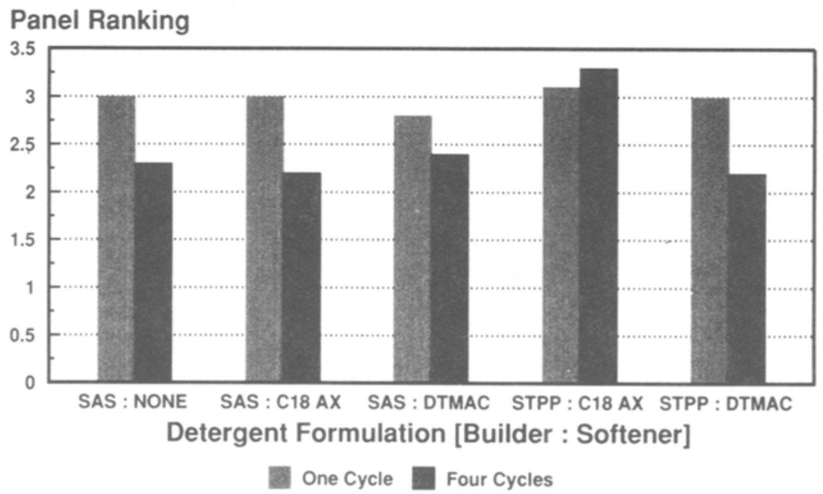

FIG. 9. Laundry detergent: whiteness rating. Variability = ±0.5 for 95% confidence level.

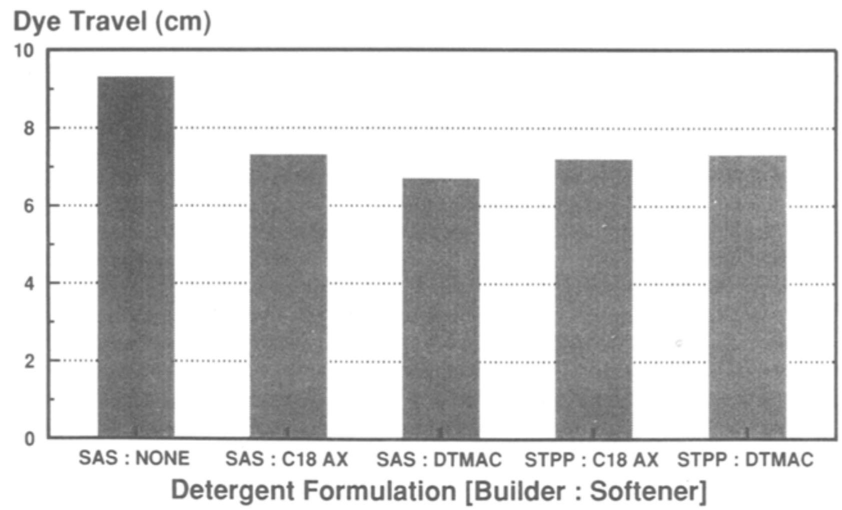

FIG. 10. Laundry detergent: wetting rating after four cycles.

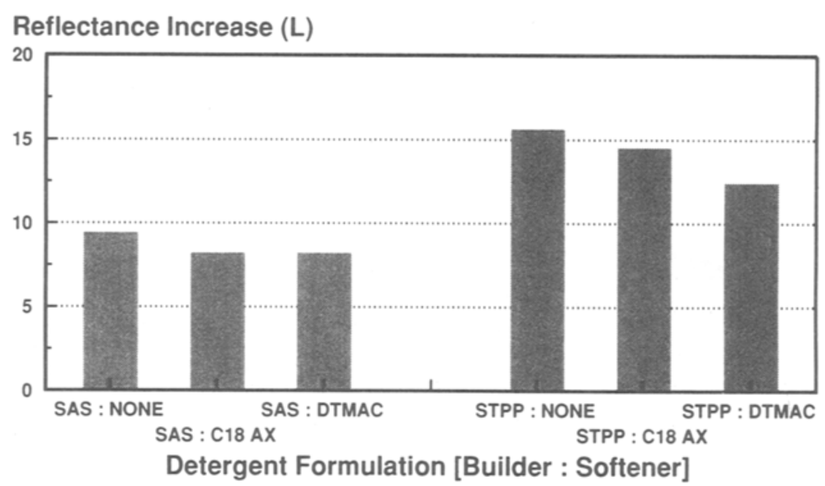

FIG. 11. Laundry detergent: detersiveness rating from tergotometer evaluations of dust-sebum cotton/polyester test fabric.

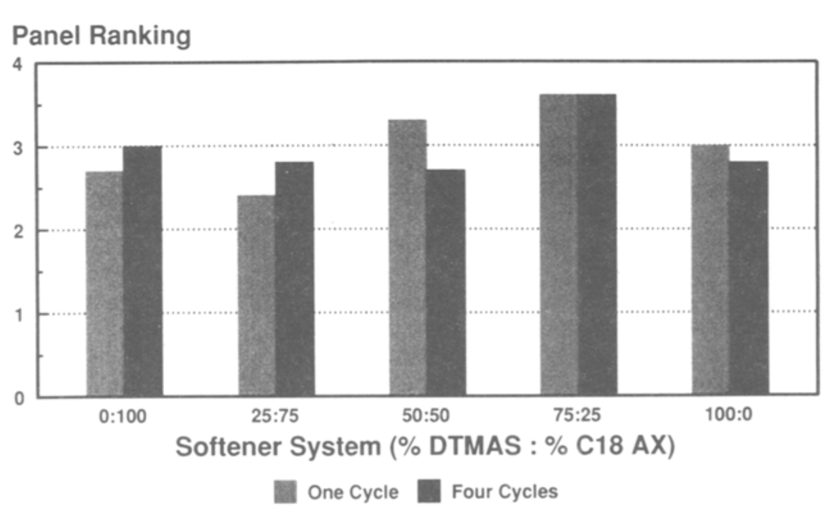

FIG. 12. Dryer sheet synergy: softness rating. Variability = ±0.3 for 95% confidence level.

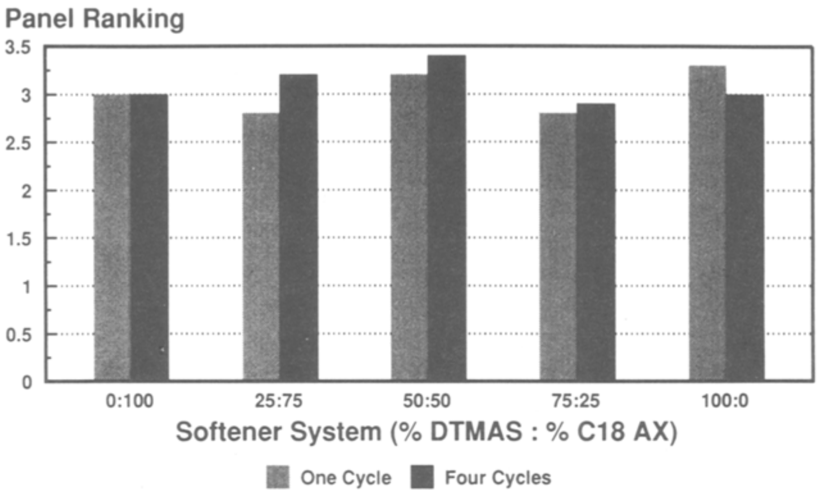

FIG. 13. Dryer sheet synergy: whiteness rating. Variability = ±0.5 for 95% confidence level.

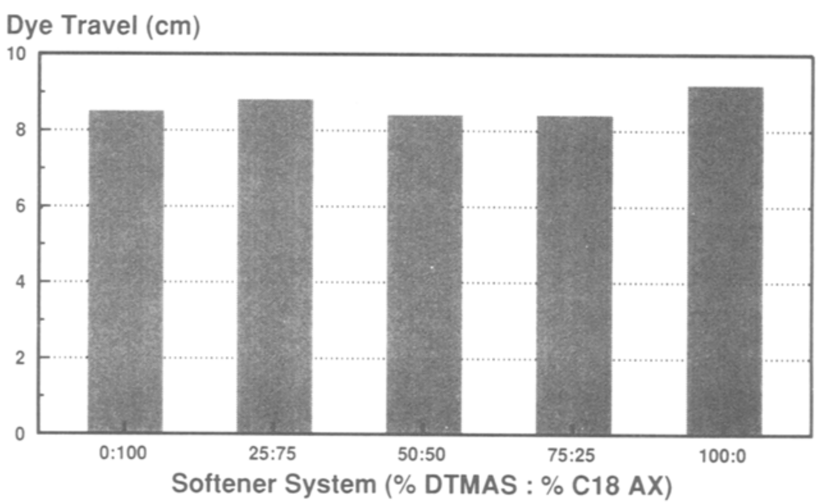

FIG. 14. Dryer sheet synergy: wetting rating after four cycles.

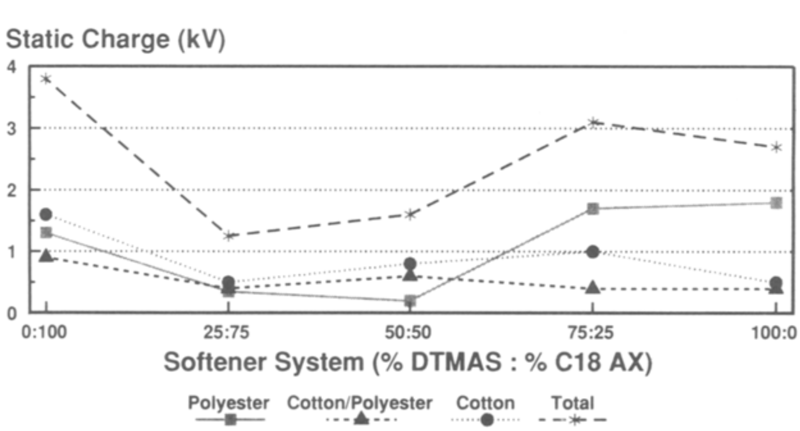

FIG. 15. Dryer sheet synergy: antistatic activity after one cycle. Values for untreated fabrics are: polyester, 10.9 kV; cotton/polyester 2.2 kV; cotton, 2.9 kV.

Due to the potential for a builder to affect softener performance, both phosphate and nonphosphate formulations containing 5% softener were tested as softeners on unsoiled, cotton terrycloth towels. Regardless of the nature of the builder, detergents containing DTMAC were ranked the softest for both 1 and 4 cycles (Fig. 8). Formulations containing C18 AX were indistinguishable from untreated towels.

In terms of whiteness retention, all formulations were equivalent after a single cycle (Fig. 9). However, after 4 cycles, towels treated with the phosphate-built C18 AX were noticeably whiter than the other formulations.

The wettability of towels from four wash/rinse cycles was then determined (Fig. 10). In the phosphate-built formulations, both DTMAC and C18 AX impeded wetting to the same extent. In the nonphosphate systems, though, the C18 AX gave better wetting than DTMAC. All systems wetted to a lesser degree than the control with no softener.

Of course, the major function of a detergent/softener combination remains cleaning rather than softening. The detersiveness of the test formulations was evaluated in a Tergotometer study on dust-sebum soiled cotton/polyester swatches (Fig. 11). The conditions used were consistent with those for the softening evaluations. Overall, both the C18 AX and DTMAC reduced detersiveness. For the phosphate-built detergents, the C18 AX system gave better cleaning than the one with DTMAC. For the nonphosphate detergents, the C18 AX and DTMAC were comparable in cleaning efficacy. However, regardless of the builder, the DTMAC detergents yielded significant splotching of the washed fabric.

Dryer sheets. In the third major area, dryer sheets, softening was evaluated once again in a panel test of cotton terrycloth towels. As in the laundry rinse study, blends of C18 AX and DTMAS were tested for potential synergy. After one cycle (Fig. 12), two blends (25% C18 AX/75% DTMAS and 50% C18 AX/50% DTMAS) did indeed prove to be synergistic, giving greater softness than the oxide or quat alone. After four cycles, only the 25% C18 AX/75% DTMAS system remained synergistic. All other formulations were nearly equivalent.

In both one and four cycles, all the dryer sheet formulations produced the same degree of whiteness retention (Fig. 13).

The DTMAS and 75% C18 AX/25% DTMAS blend gave the best wetting after four cycles (Fig. 14) and were indistinguishable from each other. The pure C18 AX was less effective than DTMAS for wetting. This was the reverse of the relative wetting of C18 AX and DTMAC in the rinse study. A possible explanation for this reverse trend may be linked to the amount of softening agent transferred onto the test fabric. Measurements revealed that 60% of the pure oxide and of the blends were transferred to the fabric. Only 30% of the pure quat was transferred.

The dryer sheet antistatic evaluation produced surprising results (Fig. 15). On polyester, two blends (75% C18 AX/25% DTMAS and 50% C18 AX/50% DTMAS) were more effective than their pure components at reducing static electricity. On 35% cotton/65% polyester, all blends were equivalent to DTMAS and superior to C18 AX. On cotton, a 75% C18 AX/25% DTMAS system was equivalent to DTMAS, which gave the best static reduction. All blends were superior to C18 AX. For a laundry bundle consisting of 23% polyester, 46% cotton/polyester, and 31% cotton, the two synergistic blends (75% C18 AX/25% DTMAS and 50% C18 AX/50% DTMAS) were the most effective antistatic agents.

In laundry rinses, blends of C18 amine oxide and DTMAC proved to be synergistic for improving the wetting of cotton towels. Although no synergism was observed in laundry detergents, formulations containing amine oxide gave better detersiveness than systems with DTMAC, without the splotching associated with the quaternary salt. In dryer sheets, it was discovered that blends of amine oxide and DTMAS gave synergistic softening of cotton towels and were unexpectedly effective in preventing static charge build-up on polyester fabric.

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