

# Rinse-Added Fabric Softener Technology at the Close of the Twentieth Century

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**ABSTRACT:** This paper reviews rinse-added liquid fabric softener products including current and emerging technologies related to composition, raw materials, and manufacturing processes. An introductory overview of the historical drivers, market trends, advantages and disadvantages, and consumer habits and practices that affect these products is also included.

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**KEY WORDS:** Diamidoamine esterquat, environmental, formulation, market, quaternary ammonium cationic surfactants, trends.

## BACKGROUND AND PRODUCT HISTORY

*Value of fabric softeners: feel, freshness, and static reduction.* Fabric softeners provide a means to impart a variety of desirable characteristics to clothing, the most obvious being improved feel or “hand” when the fabric is rubbed across the skin. Through the use of perfume or masking scents, they can also impart a perception of freshness. In addition, they provide a delivery vehicle for attaching other consumer-beneficial additives, such as soil release agents, whitening agents, and antiwrinkling agents. Fabric softener use has been documented to extend garment life (1). They reduce drying time and thus extend garment life by reducing interfiber friction and mechanically induced fiber damage during the tumble-drying process. By virtue of their lubricating properties, they can reduce the formation of wrinkles and make fabrics easier to iron.

Fabric softeners reduce static electricity buildup (2). When applied in the rinse cycle of the washing process, they can provide a moderate reduction in static electrical charge which occurs during the tumble-drying process.

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When applied in the form of a “dryer sheet,” a coated fabric or foam substrate that allows the active ingredient to melt and be transferred by mechanical rubbing in the tumble dryer, almost total static remediation has been claimed in a broad range of patents (3).

*Sources of harshness.* The history of fabric softeners in consumer use is associated with the conversion of laundry detergents from tallow-based soaps to synthetic bases, or syndets. Since ancient times, clothes have been washed with soaps (sodium salts of fatty acids) by hand, and later with a mechanical washing machine. Around 1945, synthetic detergents, primarily based on alkylbenzenesulfonates as well as other secondary surfactants, began to rise in prominence for machine washing in North America. The new generation of laundry detergents was formulated with builders, that is, sequestering agents such as phosphate, carbonate or citrate, to reduce the deposition of insoluble calcium and magnesium salts of soap and alkylbenzenesulfonates. These insoluble calcium and magnesium salts cause redeposition of soil, resulting in a gradual buildup of a dingy, gray film on light-colored fabrics (4).

The presence of sequestering agents resulted in a significant reduction in the amount of lime soaps left behind on clothes. Moreover, mechanical washing machines coupled with improved detergent formulations led to improved removal of oils, clay soils, and other natural fiber lubricants. These residues all contributed to a softer hand and their enhanced removal resulted in a harsher feel of the fabric.

Cotton, still the predominant fiber in today’s textile industry, suffers from unique mechanical wear and tear processes which ultimately create consumer demand for fabric softeners. With repeated laundering, cotton microfibrils break and unravel. Mechanical friction in the washing process induces static charges that cause the mi-

crofibrils to project orthogonally from the fiber bundle upon drying. These microfibrils act as barbs which inhibit fiber-fiber slippage, interfere with fiber flexibility, and are perceived as a source of drag when drawn across the skin (5). All of these phenomena contribute to the total perception of roughness. Softening materials can reduce fiber-fiber interactions by reducing static and allowing microfibrils to lay parallel to the fiber bundle and/or by coating and lubricating the fiber bundle to minimize friction. Further, they can provide a lubricating layer between the fiber surface and human epidermis. The net result is the perception of a less abrasive, more pliable fabric. By this coating/lubricating mechanism, lime soaps, oils, flat clay platelets, and cationic surfactants all function to greater or lesser extent to improve fabric hand.

Marketing of fabric softeners to consumers has exploited fragrance as a key signal for softness and freshness. The perception and appreciation of the fragrance, coupled closely with softening performance, successfully differentiate fabric softeners. Considerable time and effort are involved in the selection of a signature fragrance, since there are at least six unique points of consumer interaction with the product at which the bouquet will change: (i) as the consumer in the store makes his or her initial buy decision, (ii) as the softener is charged to the dispenser or during the rinse step, (iii) as the wet clothes are removed from the washer, (iv) as the dry clothes are removed from the line or drawn out of the tumble dryer, (v) when the clothes are folded and put away, and (vi) when they are donned. Major consumer product companies have incorporated cyclodextrins (6) and chemically modified perfumes (7) that provide for the slow release of fragrance after extended periods of time. Fragrance persistence can affect the number of garment wear cycles between washing.

Rinse-added softeners provide a way to add ingredients during the washing process that may not be compatible with a laundry detergent formulation but that can impart consumer-perceived benefits. Numerous patents have appeared covering the incorporation of polyethylene terephthalate soil release agents, which enhance the release of oily soils from polyester fabric (8,9). More recent patents claim the dual benefit of dye-transfer inhibition and soil release provided by nitrogen-containing polymers (10). Silicones are cost sensitive ingredients which have gone in and out of formulations in North America (11). Biocidal quaternaries suppress mildew and odor formation when wet clothes are left in the washing machine for 6–8 h (12). Cellulase (13) and other enzymes can be delivered here, as well as dye stabilizers (14), ultraviolet (UV) light absorbers (15), chlorine scavengers (16), and optical brighteners (17), all of which are geared to help maintain that “new garment” appearance after multiple wash/dry cycles.

Different classes of chemicals and materials can provide fiber lubrication and some degree of fabric softening. Cationic surfactants are the most ubiquitous ingredients used worldwide as rinse-added fabric softeners. The rea-

sons for this are many. They are cost-effective, being highly efficient at depositing or “exhausting” onto the fabric even at extremely low concentrations. They are effective at reducing microfibril static and interfiber friction. They provide a renewable finish that interferes only minimally with the laundering process. They are based on low-cost raw materials, predominantly tallow, lard, or, alternatively, on seed oils such as palm oil, soybean, or canola (rapeseed) oil (18). They are relatively easy to formulate with conventional mixing equipment and require few supplemental ingredients. They are essentially nontoxic to higher life forms. They are ultimately biodegradable and do not build up in the environment (1).

Cationic surfactants first found use as dye leveling agents in the textile industry in the 1930s. Their softening properties were discovered as a side benefit. Household fabric softener products did not appear until some 20 yr later, when brands such as Sta-Puf and Downy were first introduced in the United States in the mid to late 1950s by A.E. Staley and Procter & Gamble, respectively. Brands such as Comfort, Soupline, Lenor, and Humming were launched in Europe and Japan in the 1960s (19). All of these systems were aqueous dispersions containing 3–8 wt% cationic softener active. The dose size ranged between 4 and 8 ounces (half cup or whole cup per load). For a 6–10 lb wash load, they delivered from 0.1 and 0.25% of softener solids calculated on weight of dry fabric.

An important factor affecting the North American market is that the majority of households have owned mechanical clothes drying machines since the late 1960s. These appliances are now present in almost 80% of homes. Tumble drying imparts significant levels of static electricity to clothes. Especially the combination of a high level of synthetic fabrics and blends with the low-humidity during winter months results in “static cling” and electrical shocks. These problems drove the growth of the “dryer sheet” product segment (20). By 1983, U.S. dryer-sheet sales accounted for 40% of all household fabric softener unit sales. Nevertheless, rinse-added fabric softeners consume around 80% of the 200+ million lb/yr of total softener actives manufactured in the United States. This is explained by the significantly higher level of solids delivered per dose of rinse-added fabric softener, usually 5–7 g of solid per laundry load compared to only 1–3 g delivered per dryer sheet. It is not surprising that rinse-added products deliver considerably better softening than dryer sheets.

Through the 1970s and 1980s, rinse-added softeners had a far greater impact in Europe than in the United States because there were considerably fewer tumble dryers in Western European households and line drying is still standard practice in Europe today. Only 21% of Western European households own tumble dryers, and the number is even lower in most other parts of the world. Thus, outside of the United States and Canada, dryer sheets occupy a minor market niche. In Europe, 100% cotton garments have historically predominated over polyester blends and

the effect of softener is more noticeable to consumers. Pure cotton garments generally require hot ironing to remove wrinkles. Fabric softeners help improve the ease and efficiency of the ironing process. The European market consumes well over 230 million lb/yr of softener actives as a result (20).

## MAJOR MARKETING TRENDS

Proliferation of concentrated products has allowed greater plant throughput with existing equipment as well as a reduction by more than 50% in plastic used for bottles and cardboard for packing boxes (19). The reduced volume also has a direct impact on shipping costs, warehouse storage costs, and store shelf space. However, in many markets low active products predominate and coexist with ultra-concentrates because of consumers' perception of value. Consumers definitely have their own regional sensibilities when it comes to cost per unit volume of fabric softeners.

Globalization of formulations has been another significant trend among major consumer product companies in an effort to reduce costs. Softeners are just one category under pressure in this respect. Some typical components and charge levels for a 5% concentrate dispersion and a 25% ultra dispersion are presented in Table 1. A typical formulation will start with water, then adds the cationic base, preservative, buffering acid, product colorant, optical brightener, fragrance, and optionally a small quantity of electrolyte to adjust viscosity.

Rinse-added softeners target at a delivery of 0.2–0.25% of softener active ingredient based on weight of cloth. For a 6 lb load, this translates to 5.5–6.8 g of softener active. (A dryer sheet, by comparison, delivers 1.5–2.5 g of solid.) As mentioned before, softener products started out with the 8 fl oz whole-cup "economy" and 4 fl oz half-cup "premium" versions. The recent "ultra" category was first introduced to consumers as a dilute-before-use product, to be combined with 3× or 4× the amount of tap water and shaken vigorously to regenerate the 5–8% concentrate dispersion (Table 2). However, consumers in many developed markets have learned to accept the use of 1 to 1.5 fl oz (30–45 cc) of the ultra products. In fact, new washing machines in North America are being built with fabric softener dispensers

which hold 4 fl oz (115 cc) or less, forcing consumers away from the whole-cup "economy" products.

Along this direction, Procter & Gamble developed an after-market dispenser for ultra fabric softeners designed to deliver 1 fl oz. The "Downy Ball" has a cap that opens during the spin cycle of a vertical-axis washing machine as a result of centrifugal force (21). The softener is dispersed from the ball during the deep rinse cycle. The ball is effectively free of softener residue, which often accumulates in machine dispensers, and is thus easily refilled for use in the next load.

## FABRIC SOFTENER MOLECULES

*Dialkyldimethylammonium compounds.* Most cationic softener actives are molecules which contain two fatty chains of 16–18 carbons each, with the cationic charge located roughly in the middle. The commonly used fabric softeners bear structural similarities to lecithin and other phosphatidylcholines which constitute cell membranes (Scheme 1). Their propensity to form colloidal particle dispersions comprising multilayered vesicles similar to cell membranes has a definite impact on formulation characteristics, mechanism of deposition, and softening performance (22).

As a product class fabric softeners are not pure compounds. Like other surfactant raw materials, they are derived from feedstocks which themselves comprise mixtures or distributions of various fatty carbon chains. Commercial production processes almost always generate a mixture of single, double, and sometimes triple, chain derivatives, but these mixtures are easier to disperse and formulate than the pure di-chain compounds. The complexity of the mixtures, containing mono-, di-, and trialkyl components of varying chain lengths, has led to difficulties in relating nominal chemical structure to phase behavior, deposition mechanism, and softening performance (5).

Over the last 40 yr, numerous investigations have been carried out on the kinetics and mechanism of cationic fabric softener deposition (2,23–25). Kunieda and Shinoda (26) and Laughlin *et al.* (27–29) investigated the basic phase behavior of dioctadecyldimethyl ammonium chloride as a model for the commercially ubiquitous product derived

**TABLE 1**  
Typical Formulation Ingredients Used in U.S. Household Fabric Softeners

Ingredient	Example	Concentrate <sup>a</sup> (5%)	Ultra concentrate (25%)
Cationic base	Diamidoamine or diester quaternary	5%	24%
Preservative	Glutaraldehyde, formaldehyde	0.1%	0.2%
Buffering acid	Phosphoric, sulfuric, hydrochloric	to pH 3.5	to pH 3.5
Dye, optical brightener	"Product colorant," cationic compatible	100 ppm each	100 ppm each
Fragrance	Water-dispersible or water-soluble	0.2–0.8%	0.8–4%
Electrolyte	CaCl <sub>2</sub> or NaCl	None	0.3–1%
Water	Filtered, softened, potable	Q.S. 100%	Q.S. 100%

<sup>a</sup>Q.S., quantity sufficient (to yield 100%).

**TABLE 2**  
**Typical Compositions and Product Characteristics of U.S. Household Fabric Softeners**

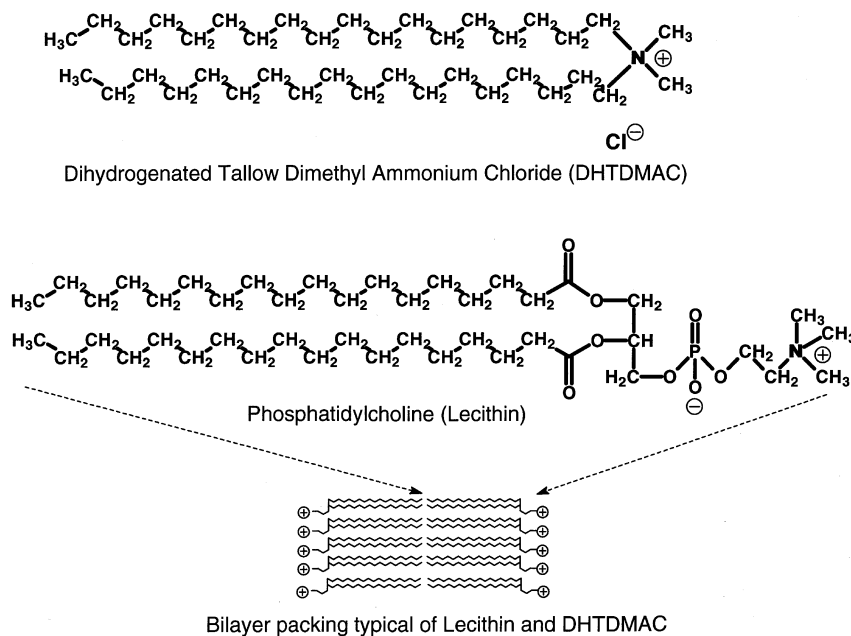
Typical 6 lb wash load	Regular (economy)	Concentrate (premium)	Ultra	Dryer cycle
Solids	1–3%	5–8%	15–26%	1.5–2.5 grams
Amount used	224 cc (whole cup)	112 cc (half cup)	30–45 cc (1–1.5 fl oz)	1 sheet
Delivered solids (g)	2–6	7	5–7	1.5–2.5
Delivered solids on dry fabric weight (%)	0.1–0.22	0.25	0.2–0.25	0.05–0.1
Viscosity (centipoise)	20–100	40–200	50–200	Solid
Softening	Good	Excellent	Very good	Detectable
Static control	Good	Good	Good	Excellent

from tallow and have come to sometimes contradictory conclusions.

The triglycerides or fatty acids from which most softeners are manufactured comprise a distribution of fatty chains, which is dependent on the feedstock. The optimal combination of low cost, good performance, and formulatability is achieved using  $C_{18}$  and  $C_{16}$  chain lengths, which are commonly found in tallow, lard, and some seed oils. Experience has demonstrated that shorter chain lengths such as lauryl ( $C_{12}$ ) and myristyl ( $C_{14}$ ) contribute little softening performance. Another general trend is the effect of unsaturation (Table 3). Softeners based on hydrogenated tallow triglycerides or stearic acid have higher melting and storage temperatures (and usually require more solvent for ease of handling) but offer premium softening and lubricating qualities. The fabric is left with a very slick hand. Dispersed in water, these saturated softeners form colloidal dispersions usually composed of lamellar or gel phases with highly organized bilayers. The inherently high viscosity and high volume fraction of the colloidal particles make it difficult to formulate concentrated products. With unsaturated fatty chains, the softening effect is somewhat reduced and the fabric is left with a

dry but very supple, flexible hand. Highly unsaturated softeners when dispersed in water produce colloidal dispersions in water composed of particles in cubic or isotropic phases as a result of more disordered fatty chain packing. The lower phase viscosity allows for easier formulation of concentrated products by addition of electrolytes which reduce the colloidal particle volume, ostensibly by an osmotic process.

One of the earliest commercial softeners, developed in the 1950s, was di(hydrogenated tallow) dimethyl ammonium chloride (DHTDMAC). This product is prepared by the fairly complicated process, detailed in Scheme 2: Tallow is steam-split by the Colgate-Emery process and the fatty acids are distilled from the glycerin "sweetwater." The fatty acids are then subjected to ammonolysis over a bauxite catalyst to generate fatty nitriles which are subsequently reduced by hydrogenation over a nickel catalyst to yield a mixture of fatty amines. Two equivalents of fatty amine are disproportionated with elimination of ammonia to give the di(hydrogenated)tallowamines. These are subjected to reductive alkylation with formaldehyde in the presence of a nickel catalyst to yield the dialkylmethylamine which is quaternized with methyl chloride (30). Alternatively, the di-



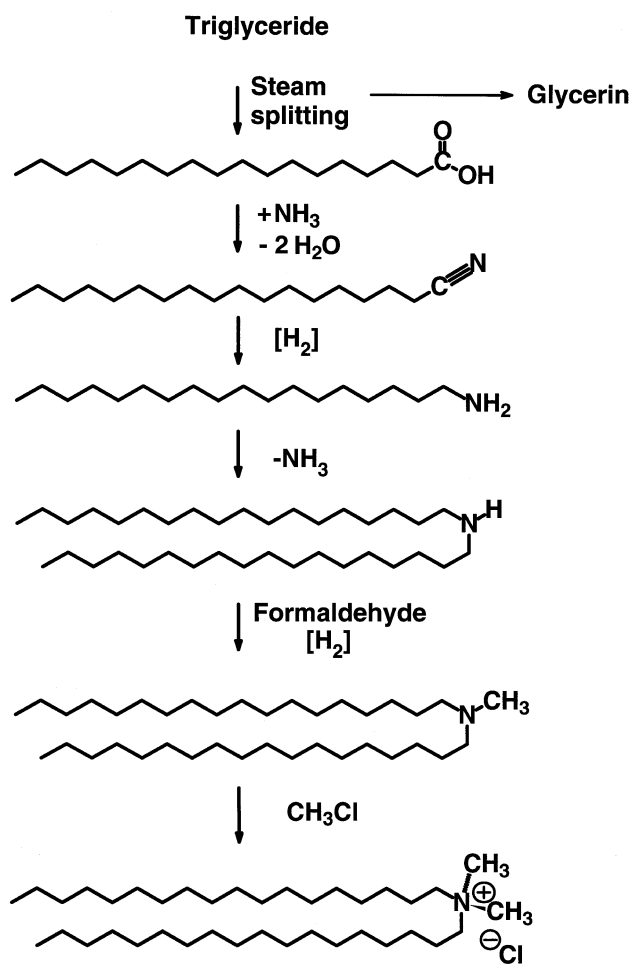
**SCHEME 1**

**TABLE 3**  
Effects of Chain Saturation on Characteristics of Fabric Softeners

Unsaturation (iodine value, IV)	Low (IV = 1–10)	High (IV = 45–60)
Handling temperature (°C)	>65	40–55
Softening	Premium	Good–excellent
Hand	Slick	Dry
Stable concentrate, aqueous	~15% maximum	~25% maximum

alkylamine may be sequentially alkylated and quaternized with two equivalents of methyl chloride and sodium hydroxide, resulting in very pure sodium chloride and DHTDMAC. The sequence of multiple processing steps under operating conditions of high-temperature and -pressure results in higher manufacturing overhead costs.

The hydrogenated chains, coupled with the absence of other chemical functional groups along the chain (such as amides or esters), furnish a product which is inherently more crystalline in the solid phase. Hence, it is generally handled as a 75% active material and diluted with a mixture of alcohol and water to reduce the pumping temperature.



Dispersed in water, the hydrogenated product has liquid crystalline properties. Examination of a 5% DHTDMAC dispersion in water with polarized-light optical microscopy reveals the dispersion droplets to be strongly birefringent, indicating that they are in a liquid crystalline phase (26,27). This phase makes for excellent softening performance, when the product deposits on the fabric, after line-drying or mechanical tumble-drying. The liquid crystalline phase also makes DHTMAC extremely viscous when concentrated above 5%. In practice, DHTDMAC is not used at concentrations above 15%.

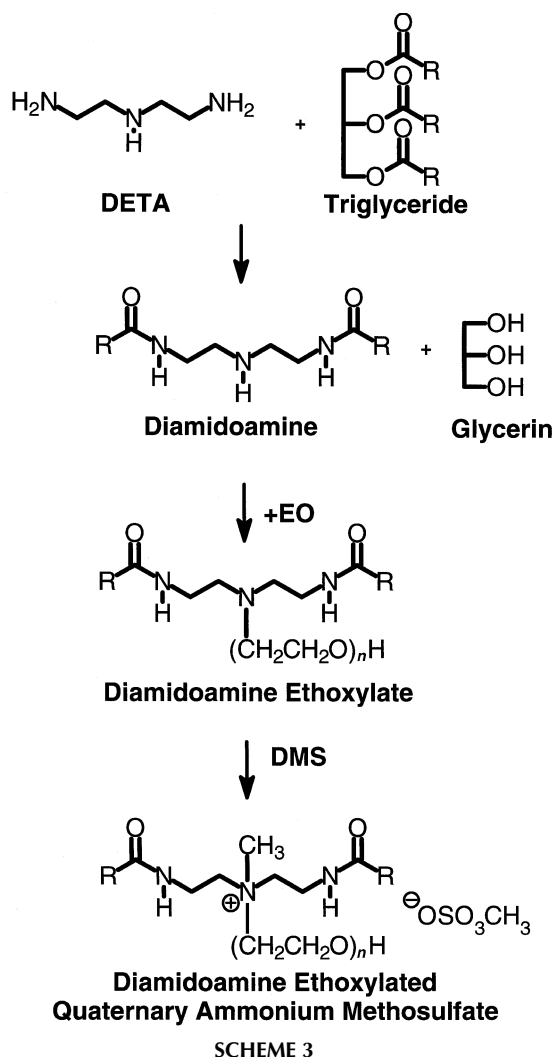
The use of DHTDMAC has dramatically decreased over the last 5 yr since its elimination as an active ingredient in northern Europe. The usage rate has now stabilized and will probably increase in the short term as a result of consumption by emerging markets.

*Diethylenetriamine derivatives.* Around the same time that DHTDMAC was developed, several actives were developed based on diethylenetriamine (DETA) which offered the advantage of lower-cost manufacturing processes and hence in lower-cost products. This category includes diamidoamine ethoxylate quaternaries and imidazolium quaternaries.

Diamidoamine ethoxylates are produced through the reaction of a triglyceride or fatty acid with diethylenetriamine to give a diamidoamine, followed by conversion of the secondary amine at the center into a tertiary amine by ethoxylation or propoxylation. The tertiary amine is generally quaternized with dimethyl sulfate to yield diamidoamine ethoxylated quaternary ammonium methyl sulfates (DAAEQAMS, Scheme 3, where EO is ethylene oxide and DMS is dimethylsulfate) (31).

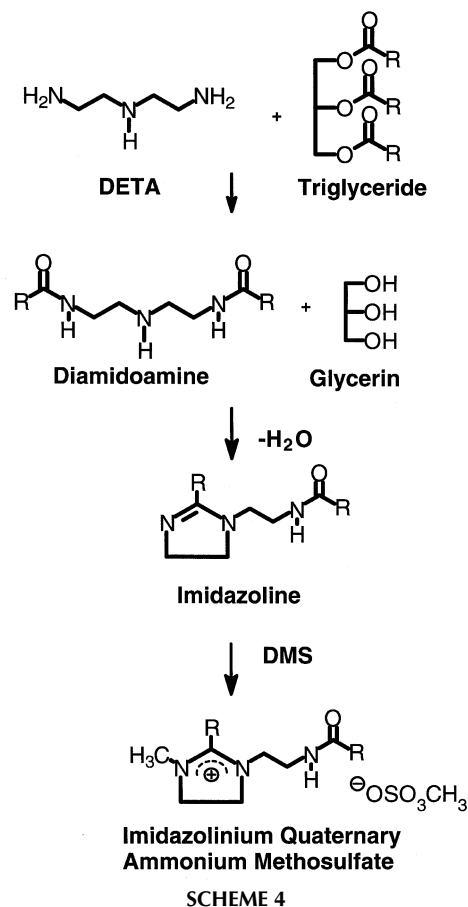
Methyl chloride quaternization is typically not practiced with this class because of the lower reactivity of this tertiary amine due to steric hindrance. A major advantage for this class of products is that they can be derived from whole triglycerides, which are cheap and available from a number of different sources including animal fats and seed oils. Product cost and performance can be adjusted by mixing different triglycerides to modify the chain distribution or iodine value. Also, DAAEQAMS based on unsaturated fatty chains can be readily formulated to 25% to meet market requirements for concentrated, high-active products. The ability to produce concentrated dispersions can be attributed to the lack of crystallinity and to the formation of particles that are in the cubic or isotropic and not in the liquid crystalline lamellar phase. Microscopic examination reveals an absence of birefringent particles (absence of Maltese crosses). The tradeoff for this product class is ease of concentration in exchange for premium softening. Completely saturated fats lead to DAAEQAMS that have softening performance much closer to DHTDMAC but that suffer from unstable viscosities at concentrations above 15% as a result of the presence of particles in the lamellar liquid crystalline phase.

A related product class is that of the imidazolium soft-



eners, which are also based on the reaction of triglycerides or fatty acids with DETA. However, rather than forming the tertiary amine *via* ethoxylation, the diamidoamine formed in the first reaction step is subjected to elevated temperature and vacuum, resulting in loss of one molecule of water and ring closure to the imidazoline. The imidazoline is then alkylated with dimethylsulfate which can react at either of the ring nitrogens (and does so in a specific ratio) to give the imidazolium quaternary ammonium methyl sulfates (IQAMS) shown in Scheme 4 (32). Prior to quaternization, the imidazoline ring is susceptible to hydrolysis at elevated temperature, which reverses the reaction and regenerates the diamidoamine. Once quaternized, the ring is stable in the presence of water and not susceptible to hydrolytic ring opening. As a class, IQAMS have a higher manufacturing cost because of the extreme reaction conditions and extended reaction times necessary to convert the diamidoamine to the imidazoline.

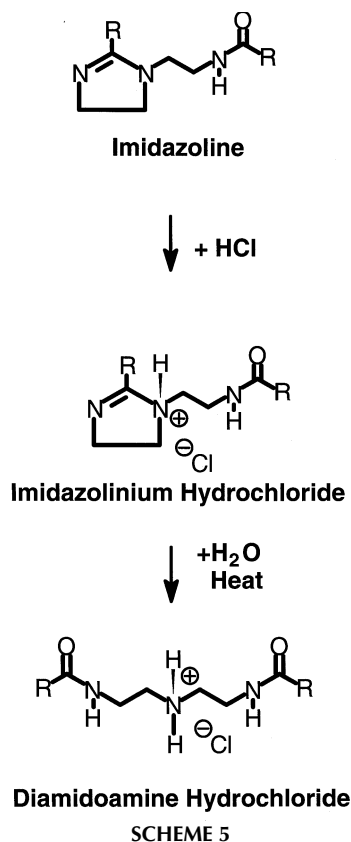
For the soft (unsaturated) tallow-based products, softening is quite comparable to that of the diamidoamines, without the requirement for ethylene oxide (EO) reaction capability. Softening performance is quite comparable to that of



diamidoamines. However, IQAMS are more difficult to formulate at concentrations above 20% solids without the use of special additives. IQAMS based on saturated chains provide softening almost comparable to DHTDMAC, but suffer from the same drawbacks in formulating concentrates. They find use as dryer sheet actives, which require elevated melting temperatures, and industrial (textile) applications. Mixtures of soft tallow imidazolium and alkyldimethylbenzyl-ammonium chloride (ADBAC) quaternaries are registered with the U.S. Environmental Protection Agency as softener/sanitizers for the industrial and institutional markets (12).

When the unquaternized imidazoline amine is dispersed into acidified water containing sufficient acid to neutralize the amine content, a stable dispersion of the imidazolium salt can be formed. This protonated, unquaternized version is stable at room temperature for several months and has the added advantage that, when deposited on fabric and subjected to the heat of tumble drying, hydrolytic ring opening regenerates the diamidoamine in the salt form and results in an improved softening effect (Scheme 5). This technology is still covered by several patents (33,34) but is no longer commercially practiced, having been replaced by esterquats.

*Esterquats.* In the 1970s three families of molecules were patented that were based on alkanolamines esterified with fatty acids or esters. The motivation at the time was proba-



bly the search for better cost performance. It is not at all clear whether their development was influenced by the emerging environmental movement. However, changing environmental standards in Europe resulted in the commercialization of these three chemistries.

Around 1970, BASF and Hoechst published patent applications disclosing diester-based quaternaries of methyl diethanolamine (MDEA). MDEA can be effectively esterified with two equivalents of fatty acid and then quaternized with methyl chloride, dimethyl sulfate, or benzyl chloride (Scheme 6). A number of examples are covered in German, French, and British patents (35,36). Diethyloxy-ester dimethylammonium methyl sulfate (DEEDMAS) product was commercialized in 1974 by Stepan Company under the tradename Bio-Soft Q164\*, but it failed to gain market share and remained insignificant for virtually 20 yr. Tougher biodegradability requirements in Europe

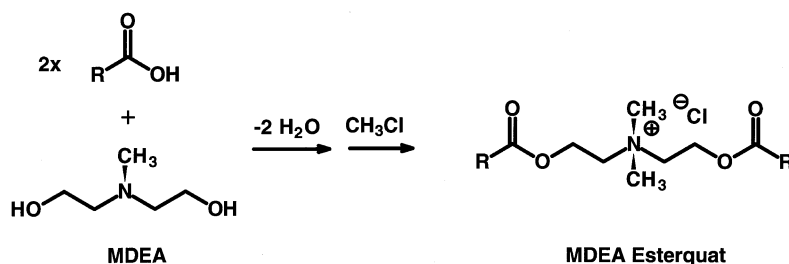
prompted its commercial relaunch, and its use has continued to grow around the world. The softening performance of this product is excellent, but it is somewhat difficult to formulate into stable dispersions. However, numerous recent patents have claimed new formulation processes and stabilization technology (37–39).

In 1977 esterquats based on triethanolamine (TEA) were patented for use as fabric softeners (40). Esterification of TEA with two equivalents of fatty acid gives a thermodynamically controlled statistical distribution of mono-, di-, and triesters, which commercially are quaternized with DMS (Scheme 7). At the time, triethanolamine-based esterquats did not compete well in the North American market against DHTDMAC- and DETA-based softeners on a cost/performance basis. However, in Europe, their cost and performance compared more favorably with these same softeners (41). Better performance could be attributed mainly to the effect in line drying, which seems to narrow the performance gap compared with tumble drying.

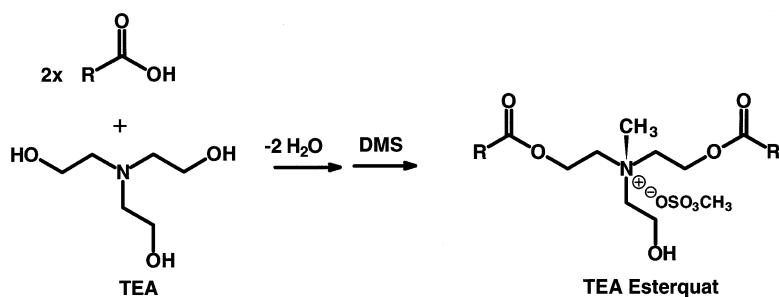
The TEA esterquat technology made it possible to formulate ultra concentrated products containing 20–24% solids. As a result, Europe saw some of the first ultra concentrated dilute-before-use softeners packaged in plastic pouches, or sachets. Many companies in Europe have used this technology since the early 1980s and it has since proliferated around the world (42). Recent advances in this technology have resulted in significantly improved cost/performance, offering products capable of exceeding DETA-based softeners in both tumble-dry and line-dry applications (43).

Also in 1977, an esterquat was patented which had a strong structural resemblance to lecithin but which is built on *N,N*-dimethyl-3-aminopropane-1,2-diol (DMAPD) (Scheme 8) (44). The amine hub, which can be prepared from dimethylamine and 1-chloro-2,3-propanediol, is reacted with two equivalents of fatty acid and then quaternized with methyl chloride. This product was commercialized in Europe because of its excellent biodegradability, low aquatic toxicity and softening performance, and is covered by numerous process and composition patents around the world (45,46).

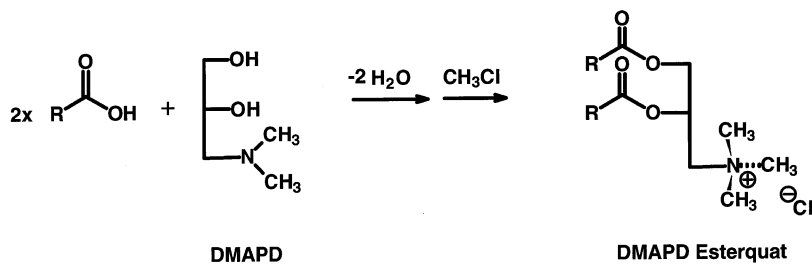
Japan has seen the commercialization of an amide-esterquat hybrid between diamidoamine chemistry and esterquat chemistry. This chemistry is based on aminopropylmethylmonoethanolamine (APMMEA), which is prepared



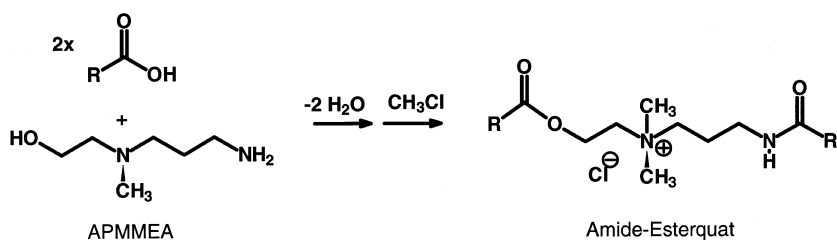
SCHEME 6



SCHEME 7



SCHEME 8



SCHEME 9

by cyanoethylation of methyl monoethanolamine, followed by reduction of the nitrile to the primary amine. This alkanolamine is condensed with two equivalents of fatty acid to produce an amide ester, which is further alkylated on the tertiary nitrogen to yield the quaternary ammonium salt (Scheme 9) (47). Claims covering this general chemical structure appeared in patents dating back before 1954 (48).

Literally hundreds of molecules have been patented for their fabric softening properties. Several thousand patents have been filed in the last 30 yr covering variations and combinations of new and old molecules as well as formulations of softener actives with different cosurfactants and functional additives (3,5,18,22,49). In practice, the mole-

cules described earlier represent probably 99% of the cationic actives being used in rinse-added fabric softeners around the world today as a result of their ability to meet regional performance requirements (Table 4).

#### FABRIC SOFTENER FORMULATION DEVELOPMENT AND EVALUATION

The fabric softener formulation process has been referred to as a black art because of the myriad factors which appear to affect its outcome. A number of variables must be understood and controlled in the fabric softener formulation process.

**TABLE 4**  
Performance Properties of Commercial Fabric Softening Active Ingredients<sup>a</sup>

	DHTDMAC	Diamido amine	Imidazoline	Diester
Tumble-dry softening	+++++	+++	+++	++++
Line-dry softening	+++++	++++	++++	++++
Rewetting	+	+++	++	++++
Static control	+++	+++	++++	++++
Nonyellowing	-	+++	++	+++

<sup>a</sup>DHTDMAC, dihydrogenated tallow dimethylammonium chloride; -, strong negative performance.



The cationic softener bases discussed previously are generally self-emulsifying, and form colloidal dispersions of micron-sized particles, when dispersed with adequate mixing into properly tempered water at temperatures above their clear point. As higher solids levels are approached, the amount and type of solvent in the softener base, the amount and timing of salt additions, and the quantity and the solubility of the perfume affect the final dispersion viscosity and product stability. These additional product requirements sometimes necessitate the inclusion of secondary surfactants as emulsifiers or stabilizers.

Formulations must be tested under accelerated conditions to simulate extended product shelf life and the effects of unplanned freezing. Storage stability evaluations are commonly performed on prototype products. Typical stability test protocols are as follows: (i) storage at 25°C for 4 wk; (ii) storage at 40°C for 4 wk; (iii) storage at 5°C for 4 wk; (iv) storage at -20°C, with three cycles of freeze/thaw (24 h frozen, 24 h thawing). It is widely recognized that stability for 1 mon at elevated temperature is far from a perfect simulation of 6 mon at room temperature but the demands for shorter time-to-market require such compromises. Physical properties which are monitored at regular intervals include appearance, viscosity, pH, odor, and dispensability.

Several performance properties that can be affected by treatment of fabrics with cationic softeners are detailed in Table 5. Specific test methods can be found in ASTM D5237-92 (50), as well as in AATCC (51) standard test manuals. The most important measurement is softening performance, which is best evaluated by a human panel. It can be conducted by paired comparisons or by ranking groups of four to six towels.

The protocol of paired comparisons is fully described in Meilgaard, Cville, and Carr's treatise on sensory evaluation (52). It allows for the direct A to B comparison of samples treated with experimental products and yields an overall

ranking of the samples against a high standard and an unsoftened control. With replicate pairs and a group of 20 panelists, this system can be used to verify differences at the 95% confidence interval. When 15 or more panelists choose softener A, and 4 or fewer choose B, then A is judged superior at the 95% confidence interval. However, repeated testing is often required to eliminate lurking variables.

## ENVIRONMENTAL CONSIDERATIONS

Increasing concern about the safety of household products over the last 15 yr have prompted the investigation of softener actives to determine their toxicity to terrestrial and aquatic life, their biodegradability, and their predicted and actual concentration in the environment (21,53,54). The cationic fabric softeners described earlier are practically nontoxic by oral or dermal administration. Monitoring and testing by the major consumer product companies has led to the conclusion that no adverse impact on human safety is associated with the use of DHTDMAC, amidoamine, imidazoline, or ester-based quaternary softener actives (55).

The aquatic toxicity to selected species of fish and benthic organisms is dependent on the class of fabric softener, with some species more sensitive to certain classes than others. However, results from biodegradability screening tests tend to be conservative and underestimate the rate and extent of quaternary ammonium compound degradation in the environment. As a result, German and Dutch authorities brought about the voluntary removal of DHTDMAC from the Northern European market. This resulted in its replacement with esterquaternaries which met test guidelines of the Organization for Economic Cooperation and Development (OECD) (20).

The amount of residual cationic active found in the environment is dependent on a complex set of factors which includes use level, population density, and waste treat-

**TABLE 5**  
**Performance Evaluation Protocol for Fabric Softener Formulations**

Attribute	Test method	ASTM /AATCC method <sup>a</sup>
Fabric softness	Test panel—paired comparison	ASTM D5237-92 (15.04) AATCC Procedure 5: Fabric Hand
	Test panel—ranking	
Static control	Static bundle, panel test	ASTM D5237-92 (15.04) AATCC Test Method 115-1995
	Static meter	
	Faraday cage Fabric to metal cling test	
Rewet	Sink disk test	ASTM D5237-92 (15.04) AATCC Test Method 70-1997 AATCC Test Method 22-1996
	Dye migration	
	Water absorption test	
Whiteness retention	Panel test reflectometer	ASTM D5237-92 (15.04)

<sup>a</sup>The American Society for Testing and Materials (ASTM) methods (50) and American Association of Textile Chemists and Colorists (AATCC) methods (51) are both ranking methods. There is no ASTM or AATCC paired comparison test.

**TABLE 6**  
**Fate of Fabric Softeners in the Environment**

Route	Chemical/physical interaction	Ecological fate of softener
Washing machine	Adsorption on fabric	—
Garment laundering	Extraction and complexation with anionic detergents	Waste treatment plant <ul style="list-style-type: none"> <li>• Elimination from water &gt;95%</li> <li>• Primary biodegradation 80–94%</li> <li>• Total biodegradation               <ul style="list-style-type: none"> <li>Laboratory 80–100%</li> <li>Treatment plant 30–60%</li> </ul> </li> </ul>
Waste treatment sludge	Soil amendment	Farm land <ul style="list-style-type: none"> <li>Level: 10–20 ppm</li> <li>No accumulation</li> <li>No phytotoxicity</li> <li>No effect on soil aeration</li> </ul>

ment processes. Even at the height of DHTDMAC use in Europe, levels in the environment were at least 10× below the No Observable Effect Concentration for even the most sensitive organisms (20,56).

Extensive testing of cationic fabric softeners has shown that softener actives are reduced by more than 90% through a two-step waste treatment process. During primary and secondary sewage treatment softeners are microbially degraded to carbon dioxide and also they adsorb to the particulate sludge. The adsorbed portion can subsequently be biodegraded further if the sludge is incorporated into agricultural land. Through these mechanisms, all cationic fabric softeners discussed here undergo ultimate biodegradation (Table 6) (1).

Studies demonstrating safe environmental levels of fabric softeners have been generated by chemical manufacturers (42), consumer product companies (57,58), and industry associations (56). As a result, all of the cationic softener actives described previously remain in production and use around the world today. However, DHTDMAC consumption has decreased worldwide and is not expected to recover in Europe.

## REGIONAL MARKET TRENDS

*North America.* The expected growth rate is around 3% per year. The primary market driver is better cost performance. A market equilibrium seems to have been reached between ultra concentrates containing 20–25% solids and regular concentrates in the 4–8% range, although the Downy Ball is having a noticeable impact on consumption of ultra concentrated products. Formulators have sought to rationalize and reduce their inventory of raw materials which has spurred the development of bases suitable for use across the full spectrum of concentrations. Biodegradability is not expected to become a barrier in North America. The softener actives described earlier are ultimately biodegradable, are removed by standard waste treatment processes, and do not persist in the environment. As a result, all of the

softener actives previously mentioned will continue to be available to formulators in North America for the foreseeable future.

Also, the slow introduction of new water- and energy-efficient washing machines is not expected to have a significant impact on the type or amount of fabric softener consumed in the foreseeable future.

*Central and South America.* Expected growth rate is >5% per annum. Primary market drivers will continue to be premium performance and strong fragrance. Since softeners are luxury products, consumers are looking for perceivable softening and strong fragrance as a co-signal. Thick low-solids formulations will continue to predominate because they imply higher active content and better value. Consumers in this region are not yet comfortable paying a lot of money for a small bottle containing a thin liquid. However, the message to reduce packaging waste is being introduced to consumers through refillable-product advertising. Line drying will continue to predominate. All of the classes of fabric softener actives are viewed as safe and sufficiently biodegradable. As a result, all of the softener actives discussed earlier will continue to be available to formulators in Central and South America for the foreseeable future.

*Europe.* The prognosis for Europe is for slow, but continued growth of fabric softeners, at a rate slightly above 2% per year including increased sales into Eastern Europe. Environmental concerns about fabric softeners have cooled now that ester-based quaternaries have replaced DHTDMAC throughout Northern Europe and most of Southern Europe as well. Even the drive toward ultra-concentrated systems (4×) has declined, as consumer product companies have diluted their most concentrated products back to 12–18% solids. This is undoubtedly in response to consumer discomfort with paying a high price for a very small bottle containing a low-viscosity product.

*Asia.* In Asia today Japan accounts for fully half of all fabric softener consumption in the region. Biodegradability is an issue in Japan as a result of high per capita use and

**TABLE 7**  
Global Fabric Softener Consumption<sup>a</sup> Estimates

Region\Year	1995	2000	2005	Growth <sup>b</sup> (%)
Europe	115	130	140	2.5
United States	72	86	104	3.5
Mexico	7	10	15	7
South America	17	23	31	6
Asia	50	50-75	50-100	0-8
Total	261	300+	350+	4

<sup>a</sup>Fabric softener consumption is in 000's of metric tons per annum.

<sup>b</sup>Growth based on a 10-yr projection from 1995-2005.

population density. The markets in Japan, South Korea, Australia, and New Zealand are well developed, and are only expected to increase at 1-2% per year. These markets have reached an equilibrium between concentrated fabric softeners and regular dispersions.

The anticipated significant growth in the Association of Southeast Asian Nations (ASEAN) has clearly been impacted by the regional economic decline. Throughout the ASEAN nations, biodegradability is not a significant issue because of the low per-capita use levels. Cost and performance are still the key criteria for selection of softener actives. All the families of softener actives described above are available in this region. Where pricing of DHTDMAC is low, it is widely used, especially since most products contain only 2-6% softener actives and a higher viscosity is desirable.

Fabric softener use is increasing. Softeners are luxury products which obviously are not critical to the basic laundry cleaning process. Their use depends on consumer perception that they deliver added comfort and are a gesture of caring and affection. Their market viability depends upon the availability of discretionary income and convenience in use. As the Gross National Product of developing nations throughout the world expands and as older washing equipment gives way to newer equipment with

dispensers, so will the use of fabric softeners expand. This is mirrored in the projections of a 3-8% annual growth rate in various markets around the world (Table 7).

Global consumer product companies have sought to reduce research and development costs by centralizing the development process. Their goal has been to produce master formulas and reduce the cycle time to market for line extensions. However, differing habits, preferences, and equipment employed in various markets around the world can significantly affect the formulation development process (Table 8). As a result, products need to be tailored to local consumer standards and preferences in order to maintain market share and profits.

A number of attributes must be optimized to meet the requirements of various markets. The disparate and sometimes conflicting priorities in these markets make it difficult to develop one new molecule which can be effective around the world. Further, a great deal of effort has been devoted to identifying synergistic blends of existing actives. These factors, in conjunction with increasing regulatory hurdles, make it difficult to develop and launch new softener bases which can offer significant performance improvements in any one of these attributes.

A major advantage of the cationic actives described previously is their listing on the chemical inventories of many countries around the world. A body of data already exists to support their excellent human and environmental safety records. The cost of safety and biodegradability testing for new molecules is significant, and can easily exceed \$200,000. Safety testing and country-by-country registration stand as significant barriers to the introduction of new softener chemistry.

## OUTLOOK

The fabric softener market is growing: It will follow GNP and the rise in living standards around the world.

**TABLE 8**  
New Softener Base Requirements

Customer requirements	North America	South America	Europe	Asia
Safety-lack of toxicity-low irritancy	+++	+++	+++	+++
OECD biodegradability <sup>a</sup>	+	+	+++++	++
Softening performance	+++	++/+++	++/+++	+++
Competitive pricing (cost)	+++	+++	+++	++++
Ability to produce 25% ultra products	+++	+/++	+++	+/++
Finished product stability	+++	+++	+++	+++
Base color and odor	++/+	++/+	+	+
Ease of formulation	++	++	++	+++
Technology ownership	+++/++	+++	+++	0
Yellowing/rewet	+	++	++	++
Static remediation	++	++	0	0
Scorch-resistant	0	++	+++	++
Solvent volatile organic carbon compounds and hazards	+++	+	++	+
Solvent flammability/combustibility	++	+	+	+
Combi-multifunctional product	+	?	0	+

<sup>a</sup>Organization for Economic Cooperation and Development.

Waste reduction and product concentration have made a significant impact: Concentrates, refillables, and reductions in packaging waste are good for everybody and will ultimately affect all regions to some extent.

An evolution of raw material choices has taken place. More active ingredients are available than ever before and no significant limitations driven by environmental concerns appear to be on the horizon.

New product attributes are difficult to create for this product category: It will be hard to coax new, better, different performance out of fabric softeners but research will continue to find new active ingredients and consumer benefits because fabric softeners are profitable product lines and meet real consumer needs.

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