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REVIEW

Ester Quats: The Novel Class of Cationic Fabric Softeners

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Abstract: Esterquats, which are quaternary ammonium compounds having two long (C_{16}-C_{18}) fatty acid chains with 2 weak ester linkages, represent a new generation of fabric softening agents, having replaced the dialkyldimethylammonium salts (e.g. DTDMAC and DSDMAC).The inclusion of ester linkages into the aliphatic chains has significantly improved the kinetics of biodegradation of the cationic surfactants, lowering the environmental exposure levels. This new generation of fabric softening agents combines a good environmental profile with the structural features required for an effective fabric conditioner. The present paper reviews the synthesis, types, actives combines a good environmental profile with the structural features required for a properties and applications of esterquats.

Key words: esterquat, quaternary ammonium surfactant, fabric softener, biodegradability

1 INTRODUCTION

Esterquats are known and the earliest mentions in the open literature date back to the 1930s[1]. The term "esterquat is commonly used, somewhat vaguely, to describe a family of cationic surfactant compounds that is heterogeneous in the chemical sense[2]. Cationic surfactants are the ubiquitous ingredients used worldwide as rinse-added fabric softeners. Cationic surfactants contain quaternary ammonium ions as their hydrophilic parts (Table 1). This class of surfactants has gained importance because of its bacteriostatic properties. They are effective at reducing microfibril static and interfiber friction[3]. These cationic surfactants are used less for their wetting abilities than for their pronounced germicidal properties and their ability to adsorb onto the surface of hair, skin and fabrics. Cationic surfactants represent a broad family of commercial compounds. Two common types of cationic surfactants are long chain amines and quaternary amine salts.

Quaternary ammonium compounds are cationic surfactants. Although cationic surfactants account for only 5-6% of the total surfactant production, they are exceptionally useful for some specific uses; because of their peculiar properties. Quaternary amine type cationic surfactants are very important as fabric softeners. They absorb on the surface of fibers with their hydrophobic groups oriented away from the fibers. This reduces the friction between fibers and imparts a soft, fluffy feel to the fabric.

This same mechanism accounts for the behavior and use of cationic surfactants as hair conditioners. Quaternary ammonium salts are effective in neutral and alkaline as well as acidic medium. They are used in the formulation of disinfectant cleaners, swimming pool biocides and fabric softeners[4, 5]. During the nineties quaternary ammonium salts containing ester groups have replaced traditional cationic surfactants in fabric softeners. While these quats do soften fabrics very effectively, they also can make them less absorbent. This new generation of fabric softening agent combines a good environmental profile with the structural features required for an effective fabric conditioner (Fig. 1)[6].

A fabric softener is a liquid composition added to washing machines during the rinse cycle to make clothes feel better to the touch. Fabric softener use has been documented to extend garment-life[7]. It reduces static electricity buildup[8]. These products work by depositing lubricating chemicals on the fabric that make it feel softer, reduce

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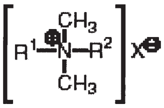
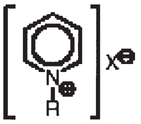
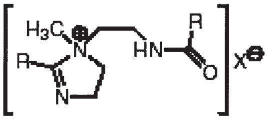
Table 1 Classification of Cationic Surfactants.

Type		**Formula**
Tetraalkylammonium salts		$R^1, R^2 = C_1, C_{16}-C_{18}$ $R^1, R^2 = C_{16}-C_{18}$ $R^1 = C_8-C_{18}, R^2 = CH_2C_6H_5$
Alkylpyridinium salts		$R = C_16-C_{18}$
Imidazoliumquaternary-ammonium salts		$R = C_{16}-C_{18}$

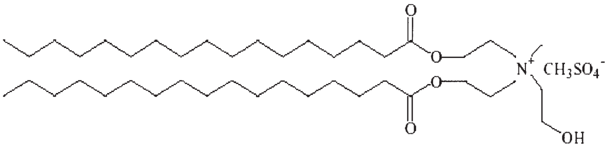


Fig. 1 Example of an 'Esterquat'.

static cling, and impart a fresh fragrance. The first fabric softeners were developed by the textile industry during the early twentieth century. These formulations soon found their way into the commercial market[9]. The popularity of these products dramatically increased over the next decade as manufacturers developed new formulations that provided improved softness and more fragrances that are appealing.

2 HISTORY OF ESTERQUATS

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 with different cosurfactants and functional additives.

In 1977, esterquats based on triethanolamine (TEA) were patented for use as fabric softener[10]. 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 (dimethyl sulphate). At the time, triethanolamine-based esterquats did not compete well in the North American market against DHTDMAC (dehydrogenated tallowdimethyl ammonium chloride) and DETA (diethylene triamine) based softeners on a cost/performance basis. However, in Europe, their cost and performance compared more favorably with these same softeners[11]. Better performance could be attributed mainly to the effect in line drying, which seems to narrow the performance gap compared with tumble-drying.

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 probably the search for better cost performance. Around 1970s, Basf and Hoechest published patent applications disclosing diester-based quaternaries of methyldiethanolamine (MDEA). MDEA can be effectively esterified with two equivalents of fatty acid and then quaternized with methyl chloride, dimethyl sulfate or benzyl chloride. A number of examples are covered in German, French, and British patents[12,13]. Tougher biodegradability requirements in Europe prompted the commercial relaunch, of fabric softener. 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[14-16].

The TEA esterquat technology made it possible to formulate ultra concentrated products containing 20-24% solids. Many companies in Europe have used this technology since the early 1980s and it has since proliferated around the world[17]. 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[18,19].

3 RAW MATERIALS

Esters are generally synthesized by reaction of an acid with an alcohol. Due to the thermal instability of quaternary ammonium compounds, they are not recommended for use with alcohol or acid functions in the esterification. In case of esteramines, esterification is usually carried out with tertiary alkanolamines and fatty acids. The most frequently used alkanolamines are listed in the Table 2 with their physical properties.

Table 2 Alkanolamines Used in the Preparation of Esteramines.

Alkanolamines	MW	Freezing polint (℃)	Boiling point (℃)	Specific gravity (20℃, g/cm^3)	Viscosity (20℃, mPa.s)
Dimethyl ethanolamine	89	−59	135	0.888	3.8
Methyl ethanolamine	119	−21	247	1.042	101
Tri ethanolamine	149	18	360	1.126	1013
3-(Dimethyl amino)-1,2-propenediol	119	—	216-217	1.004	—
Aminoethylethanolamine	104	−38	244	1.0304	141
1-(2 hydroxyethyl piperazine)	130.18	−10	246.3	1.0614	—

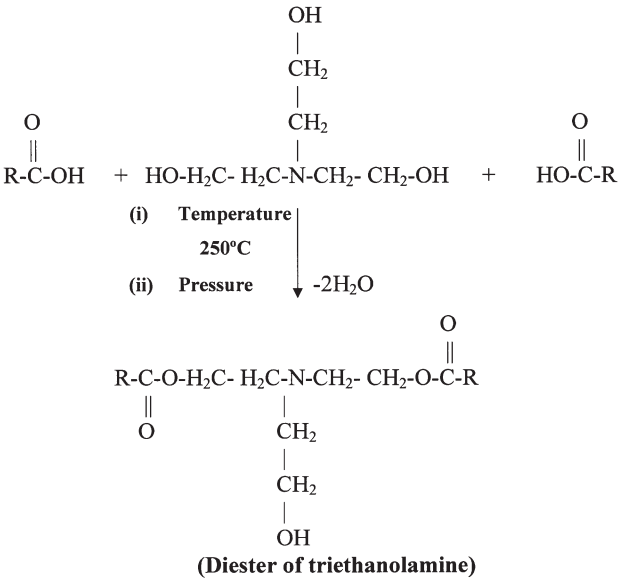


Fig. 2 Synthesis of Esterquats.

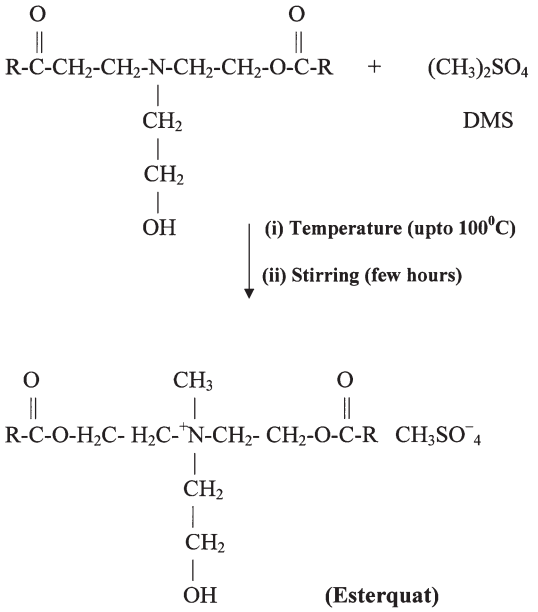


Fig. 3 Synthesis of Esterquats with Dimethyl Sulfate.

4 SYNTHESIS OF ESTERQUATS

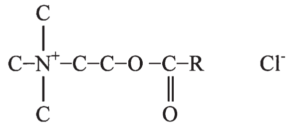
Esters are generally prepared by reaction of an acid with an alcohol (Fig. 2). Most often, the esterquats are prepared by reacting a tertiary alkanolamine with fatty acids, followed by reaction with an alkaylating agent to the corresponding quaternary. In the presence of a catalyst, the raw materials are heated upto temperatures 250℃ to obtain high conversion and water removal is enhanced by stripping with an inert gas or under vacuum[20]. The reaction times vary from a few hours to >10 h, depending on the reaction conditions and the reactivity of the components. The corresponding esteramine is reacted with an alkylating agent like dimethyl sulfate or methyl chloride to obtain the corresponding quaternary ammonium compound[21,22] (Fig. 3). The quaternizations are carried out at temperature up to 100℃, and the reaction can be completed in a few hours but may take longer with less reactive esteramines.

4.1 Classification of Esterquats

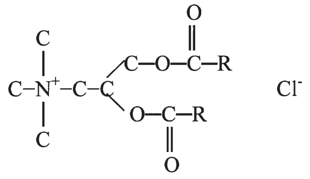
Esterquats: those derived from alkanolamines and fatty acid. Examples are shown in the Fig. 4. This type of esterquat can be divided in mono esterquats, di esterquats and triesterquats, depending on the structures of the alkanolamine and stoichimetry of the reaction[23-26].

4.2 High diester content' compounds

Triethanolamine can react with up to 3 moles of fatty acid to give the triesteramine. If less than 3 moles of fatty acid is used, the equilibrium reaction mixture contains mono-, di- and triesteramines. Figure 5 shows that the reaction product of 2 moles of fatty acid with triethanolamine contains the diesterquat as the major component, but mono- and triesterquats are present as well. More recently, it has been discovered that esterquats with an optimized 'high diester content' afford improved fabric



Monoesteramine of Dimethylethanolamine



Diesterquat of 2, 3-dihydroxypropylammonium chloride

Fig. 4 Examples of Esterquats from alkanolamines.

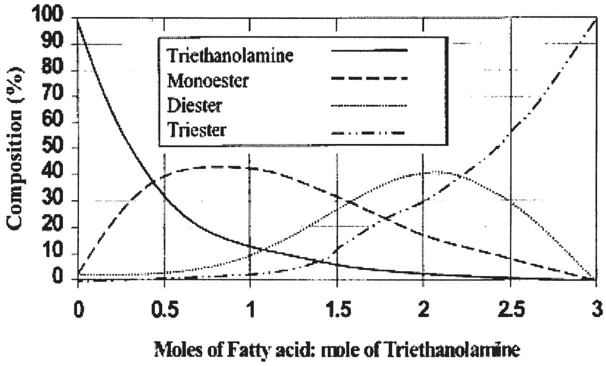


Fig. 5 Composition of the Esterquat Reaction Products from Various Ratios of Triethanolamine and Fatty Acid.

softening properties to those with lower diesterquat content. Specifically, a process has been developed to produce esterquats with greater than 55% by weight diester quaternary and less than 25% of a triester quaternary based on the total quaternary ammonium salt[27].

Other esterquats are those derived from sugar derivatives. Examples are shown in the Fig. 6, on these esterquats glucose, sorbitole or gluconic acid is incorporated in the molecule via esterification of the carboxylic acid or hydroxyl group[28-30].

Third class of esterquat is the betaine esters (Fig. 7), derived from aminocarboxylic acids and thus have a reverse ester group compared to regular esterquat based on alkanolamine[31-37]. The patent literature also contains examples of betaine esters containing two long chain alkyl groups[38-40].

Esterquats with two different ester bonds, RC(O)O- and ROC(O)- in the same molecule, are reported by Niewenhuis (Fig. 8)[41]. They are prepared by reacting dimethylethanolamine with fatty acid and subsequent quaternization with alkylchloroacetate.

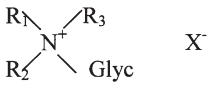
Esteramides are the class of esterquat, prepared as alternative to diesterquats. In this class, the hydroxyl group will form an ester function and the primary or secondary amine will react to an amide (Fig. 9)[33,42].

Esters of choline have attracted special attention because the primary degradation products, choline and fatty acids, are both natural metabolites in the body. A series of choline esters has been synthesized and evaluated as disinfectants with controlled half-lives[43,44]. Following are the few examples of esterquats (Fig. 10).

5 PROPERTIES OF ESTERQUATS

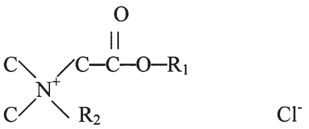
5.1 Physical properties

Most esterquats are solid materials that do not have a defined melting point but decompose upon heating. Most monoesterquats have good water solubility. Di and tri esterquats are usually less solubile in water. The physical properties of the solids change with increasing moisture contents. The di- and higher esterquats are less hygro-



R_1, R_2=H, Akyl or Hydroxyalkyl
R_3= Alkyl (1-4)
X=Halide, Phosphate, etc.
G=Glycosyl group

Fig. 6 Esterquats from Sugar Derivatives.



R_1, R_2=H, Akyl or Hydroxyalkyl

Betaine-esterquat

Fig. 7 Betaine Esterquat.

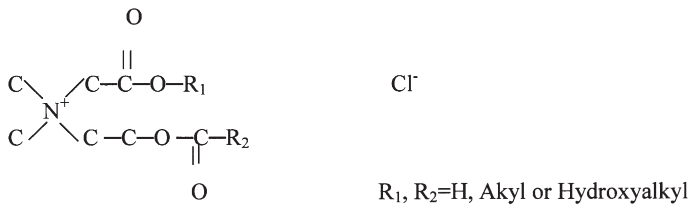


Fig. 8 Esterquats with Two Ester Bonds.

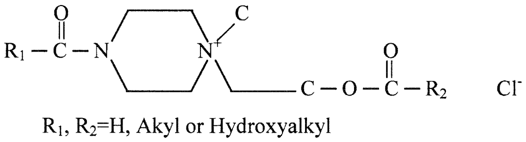


Fig. 9 Amide Esterquat.

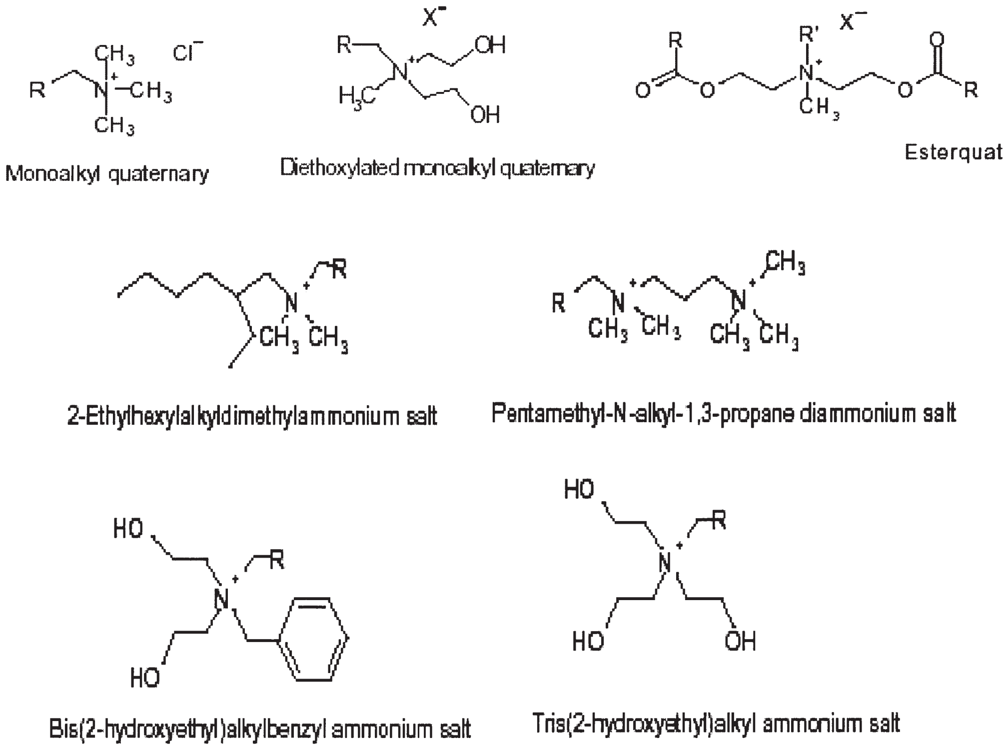


Fig. 10 Esterquats of Choline.

scopic.

5.1.1 Biodegradability

The environmental properties of esterquats have been studied extensively during the last few years. The diesterquats that are used in large volumes today as actives in fabric softeners have been subject to very comprehensive studies. This molecule and its primary metabolites have been shown to be readily and ultimately biodegradable in Organization for Economic Cooperation and Development (OECD) test procedures. A no. of new diesterquats and amidoester quats were synthesized and tested for biodegradation and softening Methyldiethanolamine, 3-methoxylpropylamine, diethanolaminopropylamine, aminoethylethanolamine, dimethylethanolamine, and diethanolamine were reacted, either "as is" or after alkoxylation, with tallow fatty acid and further converted into dialkyl quats[45].

These new generations of fabric softening agents combine a good environmental profile with the structural features required for an effective fabric conditioner. Ester linkages have been shown to be rapidly and completely hydrolyzing with half-lives for disappearance in the sewer and activated sludge in the order of hours and minutes respectively[46].

5.1.2 Hydrolytic stability

Since the early 1990s, rinse cycle fabric softening in Europe relies on esterquats; they spread around the world afterwards. However, the presence of the ester function in the molecule structure, which facilitates its rapid biodegradation in the sewage, also threatens its chemical stability on storage. The esterquat hydrolysis in softeners has been studied as a function of the molecule structure, of the finished product composition and of some of its physical characteristics. As a result, two possible mechanisms of esterquat degradation have been proposed and the likeliest identified. It is based on the replacement of the counterions in the Stern layer of the esterquat particles by fatty acid molecules present in solution. Because of the positive charge disappearance resulting from the neutralization of the ammonium ions, protons are not, repelled any more and acid catalysis of the ester bond hydrolysis may occur[42].

6 APPLICATIONS OF ESTERQUATS

6.1 Esterquats as a fabric softener

Approximately one-third of all cationic surfactants are used to produce fabric softeners and for many years, they were almost exclusively based on a di-tallow dimethyl quaternary ammonium compound[47]. The basic structural requirement for a surfactant used for fabric softening is the presence of two C_{12} to C_{18} alkyl groups and at least one positively charged hydrophile (Fig. 11)[48,49].

$$R_2N^+Me_2X^-$$

where $R=C_{12}$ to C_{18} alkyl/alkenyl

Fig. 11 The Basic Structural Requirement for a Surfactant Used for Fabric Softening.

6.2 Antistatic property

Cationic surfactants possess excellent antistatic properties, and are commercially employed in the textile and plastics industries. It is theorized that they function by making the textile surface more conductive so that static charges do no accumulate. The water of hydration aids in dissipating electrostatic charges. A glycerol derivative has good conditioning and antistatic action and is recommended for use in shampoos and hair rinses[50].

6.3 Dyeing

The receptivity of a fiber for a given dye stuff may not be uniform; resulting in uneven dyeing. This effect can be reduced or eliminated by use of a leveling agent. The leveling agent usually contains some functional group similar to that of the dye. The leveling agent competes with the dye for sites on the fiber or else the dye forms a complex with the leveling agent, which allows slow release of the dye to the fabric. When the temperature of the bath is raised, the leveling agent is gradually displaced by the dye. Esterquats are good candidates to promote level dyeing, the slowing or retardation of the rate of exhaustion of certain cationic dyes on the fabrics. A quaternary ammonium salt of an alkoxylated ester amine is used as a deinking agent for recycled wastepaper[51].

6.4 Hair care

Quaternary ammonium compounds are the key raw materials for conditioners. Quaternaries are highly substantive to the hair via attraction to the anionic charges on the hair shafts providing reduction of combing forces, increased luster, and improved antistatic properties[52,53]. Recently developed esterquats are widely used as a hair conditioning agents (Table 3). The diesterquat derived of dimethylamino-1,2-propanediol is claimed suitable for use in hair conditioning compositions[54]. Esterquat compounds demonstrate excellent conditioning effects on all types of hair[55-59].

Other complex reaction mixture of esterquats based triethanolamine with fatty acids has been suggested for use in hair and body care[60-62].

6.5 Other applications

Gemini quats suggests many applications of potential interest as follows. Gemini quaternaries are exhibiting excellent emulsification properties for this application, particularly coco-based and tallow-based Gemini ester-

Table 3 Esterquat for Hair Conditioning.

Components/INCI declaration[a]	Wt	Function
Dipalmitoylethyldimonium chloride	2%	Conditioning agent
Propyl glycol	7.5%	Solubilizer
Hydroxyethylcellulose	0.6%	Thickening
Cetyl alcohol	2.5%	Consistency regulator
Methoxy PEG-17/dodecyl glycol copoloymer	3%	Emulsifier
Water	Add up to 100%	
pH	3.5	

[a] International Nomenclature Cosmetic Ingredient According to CTFA.

quats with a spacer group of 4 methylenes. Di-coco and di-cocoester gemini quats are effective flotation agents (for example in mining calcite) over a range of spacer groups, either alone or in combination with other flotation agents to optimize selectivity.

This is just a small selection of potential end-uses for Gemini quaternaries, but it illustrates an aspect of cationic surfactants where there is still scope for innovation in product and application development.

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