RESULTS AND DISCUSSION

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Our recent studies were focused on the preparation of biocial polymeric quaternary ammonium halide salts which were synthesized based on variations in quaternizing halide (chloride and bromide) agents. Also the polyquats families were designed and synthesized with the intention of creating polymers with increasing concentrations of biocidal centers by using halide agents have more than tow halide atoms.

In a continuation to our effort in this field, we converted the polymeric quaternary ammonium chloride salts to acetate salts. The goals of this conversion are, firstly preparation of new polyquats and observed their antibacterial activities. Secondly, make a confirmation for synthesized polyquat chloride salts by two ways

- 1-Practically, by observed the lead chloride precipitated from reaction of lead acetate with those polyquats
- 2-Spectroscopically, from IR spectra which indicate carbonyl group of acetate and from ¹H-NMR

Preparation of polymeric ammonium salts of HMTA with CH₂Cl₂ (1 and 8)

In the present investigation, the polymeric quaternary ammonium salt (1) is prepared from reaction of hexamethylene-tetramine(HMTA) and methylene chloride (CH₂Cl₂) in a distilled water then

the chloride anions were substituted by acetate anions to prepare polymeric quaternary ammonium acetate salt (8) from reaction of (1) with lead acetate(Ac₂Pb).(cf. Scheme 29).

Scheme 29. Preparation of polymeric quaternary ammonium salts of HMTA-CH₂Cl₂: (1) chloride salt and (8) acetate salt

The structure of compounds ${\bf 1}$ and ${\bf 8}$ were proved from

- (i) Elemental analyses (cf. Table 1).
- (ii) IR spectra of (1 and 8) illustrated in Figs. (14 and 15) respectively which showed bands at 2950 and 2948 cm⁻¹ due to C-H aliphatic of methylene groups, also Fig.(15) showed characteristic broad bands of v C=O acetate group at 1562 and 1403 cm⁻¹ antisymmetrical and

symmetrical stretching respectively which absent completely from polyquat (1) Fig.(14).

(iii) 1 H-NMR of polyquat (1) Fig. (16) showed signals at (δ ppm), 4.60-4.81 (14H, NCH₂N + NCH₂N⁺ + $^{+}$ NCH₂N⁺) and polyuat (8) Fig. (17) showed signals at 1.71 (3H, CH₃COO) , 4.50-4.66 (14H, NCH₂N + NCH₂N⁺ + $^{+}$ NCH₂N⁺).

Table(1)

Polymer	Yield %		Analyses Calc./Found			
	Colour	repeating unit M.Wt	C	н	N	Cl
. 1	87 White	C ₇ H ₁₄ Cl ₂ N ₄ 225.1	37.7 35.1	6.3 5.7	24.9 25.1	31.5 29.5
8	95 White	C ₁₁ H ₂₀ N ₄ O ₄ 272.3	48.5 49.3	7.4 7.8	20.6	_

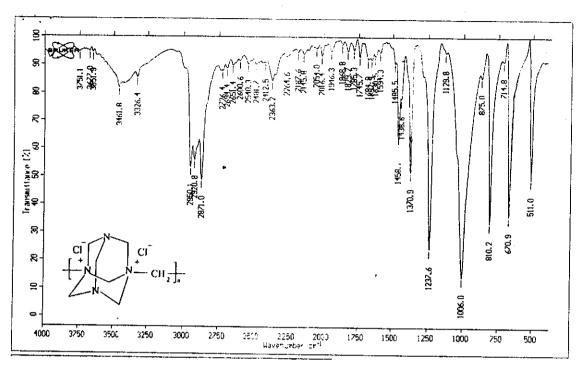


Fig. (14): Infrared spectrum of polymeric quaternary ammonium chloride salt of HMTA with CH₂Cl₂.

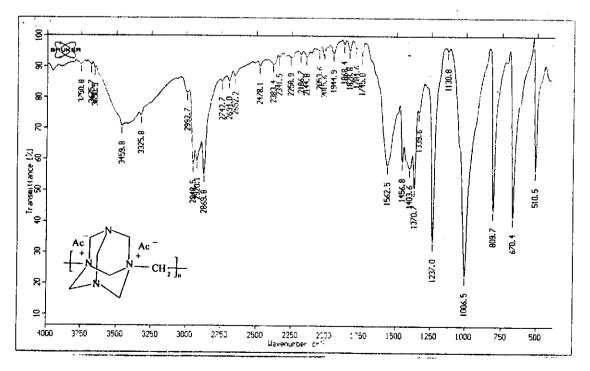


Fig. (15): Infrared spectrum of polymeric quaternary ammonium acetate salt of HMTA with CH₂Cl₂.

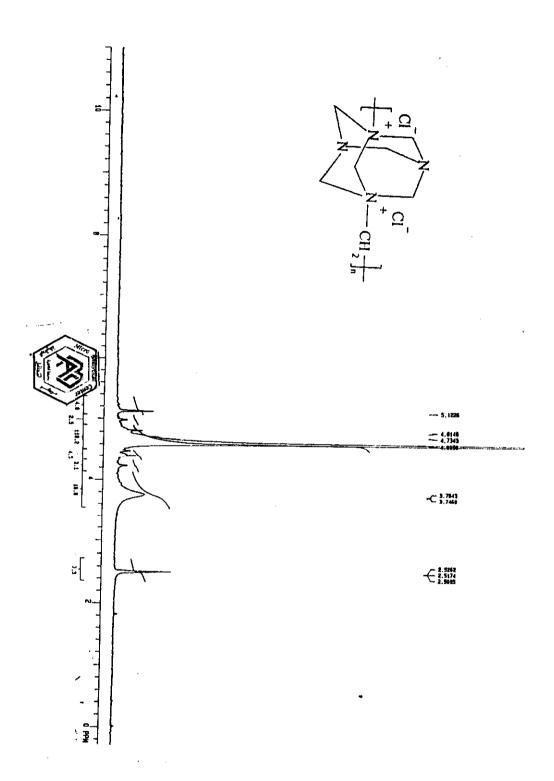


Fig. (16): ¹H NMR of polymeric quaternary ammonium chloride salt of HMTA with CH₂Cl₂.