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Oxidative Chemical Polymerization of Some 3-alkyloxylaniline Surfactants and Characterization of the Obtained Polymers

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Meta-hydroxyaniline was reacted with decyl bromide, dodecyl bromide, and cetyl bromide to produce new aniline derivative surfactants. The monomers (0.4 mol/L) were polymerized in aqueous solution of hydrochloric acid (1.0 mol/L) using sodium dichromate (0.1 mol/L) as oxidant at room temperature. The crystallinity of the prepared polymer samples was investigated by X-ray diffraction and electron microscopic analysis. The thermal behavior of these polymers was studied by thermogravimetric analysis (TGA), also the electrical properties of the obtained polymer samples were measured and discussed. The a.c.-electrical conductivity was found to be increased by the increase of number of carbon atoms in the substituted alkyl group in the following order (cetyl > dodecyl > decyl).

Keywords: poly 3-alkyloxylaniline surfactants, a.c. conductivity, TGA, X-ray diffraction, electron microscopic studies

INTRODUCTION

Polyaniline as a electronically conducting polymer has attracted considerable attention. Because of its excellent environmental stability in the electroconducting form, it has many potential applications, and unique electrical and optical properties [1–12].

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The applications of polyaniline have been limited due to its poor processability [13], which is true for most conducting polymers. Several studies have been done in order to improve the solubility of polyaniline, among them, using functionalized protonic acids as dopant, like *p*-toluene-sulphonic acid, octyl-benzene-sulphonic acid, dodecyl benzene-sulphonic acid [14–16], poly(styrene) sulphonic acid [17–18], and phosphoric acid esters [19]. An alternative method to obtain soluble conductive polymers is the polymerization of aniline derivatives. The most studied aniline derivatives are alkyl [20–21], alkyloxy [22–25], hydroxy [26–27], chloroaniline with the substitution in the *ortho* and *meta* positions [28–30]. Also, substitution at the nitrogen atom was reported by Sayyah et al. [31] to improve the solubility of polyaniline.

Various chemical oxidizing agents, such as potassium dichromate, potassium iodate, ammonium persulphate, hydrogen peroxide, ceric nitrate, ceric sulphate, and ferric chloride have been used for the chemical oxidative polymerization of aniline [7,32].

Polyaniline and polyaniline derivatives have been synthesized and their structure were determined by ^1H -, ^{13}C -NMR, IR spectroscopy [33], scanning tunnelling microscopy [34], DSC, X-ray diffraction, XPS, and conductivity measurements [35–36].

The present work intends, to improve the solubility of polyaniline by oxidative chemical polymerization of *m*-alkyloxyanilines in aqueous HCl medium using sodium dichromate as oxidant at room temperature. The obtained polymer samples were characterized by UV, IR, ^1H -NMR spectroscopy, X-ray diffraction, electron microscopy, thermal analysis, and a.c. conductivity measurements.

EXPERIMENTAL

Materials

3-hydroxyaniline provided by Merck Chemical Co. (Germany) was vacuum distilled and stored in absence of light before use. Concentrated hydrochloric acid and ammonia solution (33%) were chemically pure grade products provided by Prolabo-Chemical Co. (U.K.). Twice distilled water was used as a medium for the polymerization reactions. Sodium dichromate was product of Merck Chemical Co. (Germany). Decyl bromide, dodecyl bromide, and cetyl bromide were products of Merck Chemical Co. (Germany).

Preparation of C10, C12, and C16 Monomers

3-decyloxy aniline (C10), 3-dodecyloxy aniline (C12), and 3-cetyloxy aniline (C16) monomers were prepared by reaction of (1:1)

3-aminophenol with decyl bromide, dodecyl bromide, and cetyl bromide, respectively, in presence of sodium ethoxide as mentioned in a previous article [37].

Oxidative Aqueous Polymerization of the Investigated Monomers

The polymerization reactions were carried out in well-stoppered conical flasks of 250 ml capacity. Addition of the monomer amount followed by the addition of the required amount of sodium dichromate solution (25 ml) to the reaction mixture was carried out under nitrogen atmosphere. The order of addition of the substances was kept constant in all the performed experiments. The stoppered conical flasks were then placed at 25°C temperature in an automatically controlled oven. The flasks were shaken for three hours (15 shakings/10 s/every 15 min) by using an automatic shaker. Then the flasks were left for 2 days at room temperature and then filtered using a buchner funnel after the addition of methanol (30 ml) as nonsolvent. Then the solids were washed with distilled water, and finally dried under vacuum at room temperature until constant weight as reported by Sayyah et al. [37].

Thermal Gravimetric Analysis (TGA) and Differential Thermal Analysis (DTA)

The thermal gravimetric analysis (TGA) and differential thermal Analysis (DTA) of the polymer samples were performed using a SHIMAZU DT-30 thermal analyzer. The weight loss was measured from ambient temperature up to 800°C, at a rate of 20°C per min to determine the degradation rate of the polymers.

Electron Microscopy and X-Ray Diffraction Analysis

The electron microscopic analysis was carried out using a JSM-T20 Scanning electron microscope, JEOL, Japan. The X-ray diffractometer type Philips 1976 Model 1390 was operated at the following conditions which were kept constant all along the analysis processes:

X-ray tube: Cu; Scan speed: 8 (dg/min.); Current: 30 mA

Voltage: 40 kV; Preset time: 10 (s).

Conductivity Measurements

Polymer samples in the form of compressed discs of 1.002 cm diameter and 0.2 cm thickness were used in the present work. Covering the

samples on both sides with air drying electrical conducting silver paste produced good contact. The a.c. conductivity was measured using a Philips RCL bridge (digital and computerized) at a frequency range of 10^2 – 10^5 . The overall error in the a.c. conductivity did not exceed $\pm 2\%$.

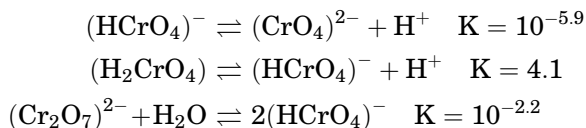
RESULTS AND DISCUSSION

Preparation of the Different Polymers

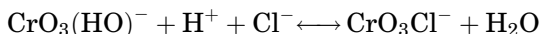
The aqueous oxidative polymerization of C10, C12, and C16 is described in the experimental section, and follows three steps.

The Initial Step

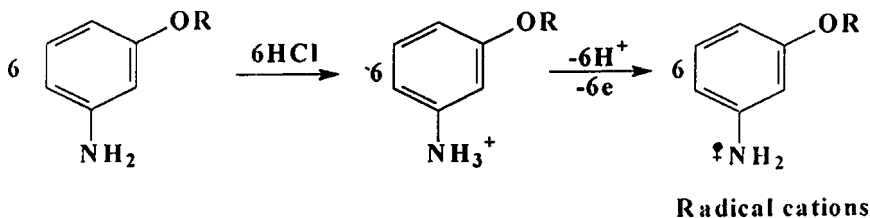
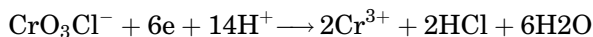
This step is the reaction between dichromate ion and hydrochloric acid, which can be discussed as follows: The orange red dichromate ions $(\text{Cr}_2\text{O}_7)^{2-}$ are in equilibrium with the (HCrO_4) —in the range of pH-values between 2 and 6, but at pH below 1 the main species is (H_2CrO_4) . The equilibria are as follows:



Further, the species present depend on the nature of the acid used [37]. When hydrochloric acid is used, there is an essentially quantitative conversion into the chloro chromate ion as follows:



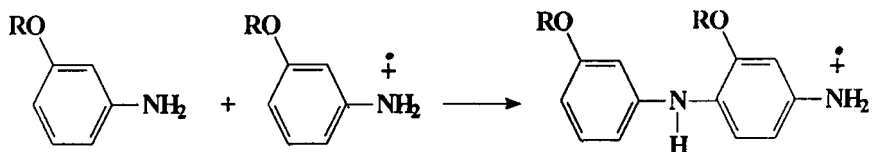
The second step is the oxidation of C10, C12, and C16 by loss of electrons and conversion of the chromium ion to the lower oxidation state in simultaneous steps as follows:



Generally, the initial step is rapid and may occur in short time, 0–5 min (autocatalytic reaction), were no polymeric product is being obtained. After 5 min of the polymerization reaction, the polymeric products were obtained.

Propagation Step

This step involves the interaction between the formed radical cation and the monomer to form a dimer radical cation as follows:

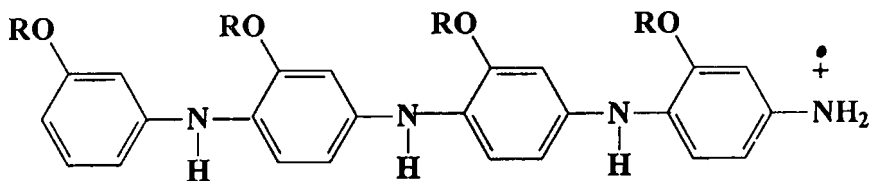


Dimer radical cations

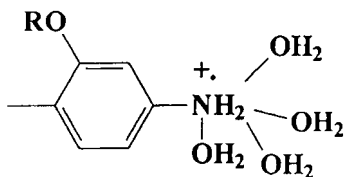
This reaction is followed by further reaction of the formed dimer radical cations with monomer molecules to form trimer radical cation and so on. The degree of polymerization depends on different factors such as dichromate concentration, HCl concentration, monomer concentration, and temperature [21,24,26–27,31,36].

Termination Step

Termination of the reaction occurs by the addition of ammonium hydroxide solution in an equimolar amount to HCl present in the reaction medium (until pH = 7), which leads to cessation of the redox reaction. The reaction could occur as follows:



Solvation of the formed polymer molecules at the end group of polymeric chain (NH₂ group) occurs concomitantly:



Electron Microscope and X-ray Diffraction

The X-ray diffraction patterns of the three prepared polymers (P_{C10} , P_{C12} , and P_{C16}) are represented in Figure 1. The figure shows that the prepared polymer samples are amorphous materials with slight portion of crystallinity in case of P_{C10} and P_{C12} that give a peak at

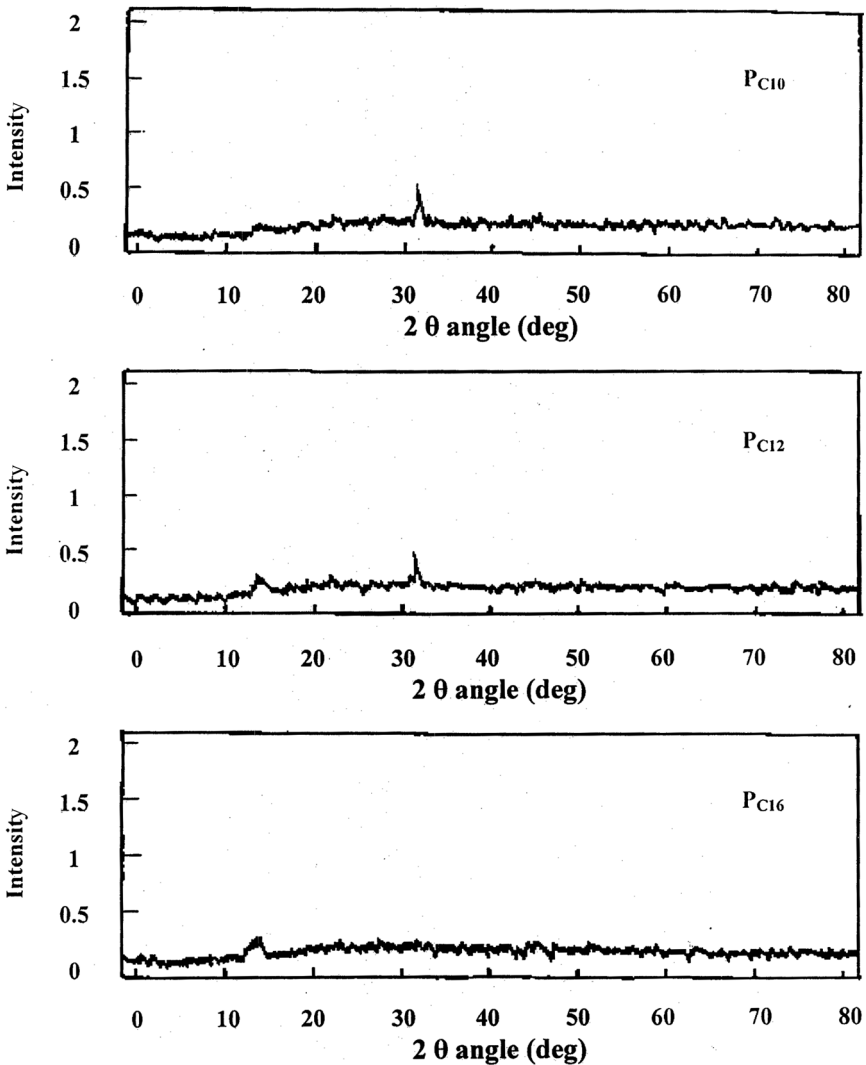


FIGURE 1 X-ray diffraction patterns of the three prepared polymers (P_{C10} , P_{C12} , and P_{C16}).

2 θ equal to 31.623 degree and the same d-spacing (2.8264 Å), but with different intensity, 0.112 KCPs in case of P_{C10} and 0.093 KCPs in case of P_{C12}. The electron micrographs of these polymers are presented in Figure 2, from which the shape and size of the grains can be detected. The shape of the grain particles ranged from spherical to elongated particles, in case of P_{C10} the grain size ranged from 0.45 to 6.0 μm, in case of P_{C12} the grain size ranged from 2.5 to 10 μm and in case of P_{C16} the grain size ranged from 3.2 to 15 μm.

Thermal Analysis

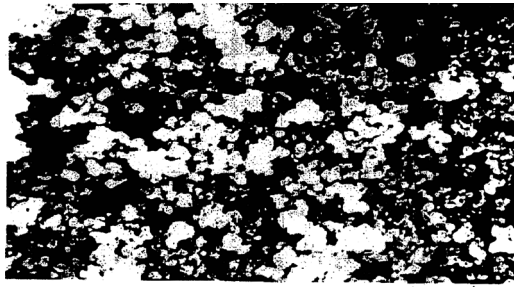
The thermogravimetric analysis (TGA) and differential thermal analysis (DTA) for the prepared polymer samples in Figures 3–5 are investigated. The thermal behavior of the three polymers samples [P_{C10}, P_{C12}, and P_{C16}] is summarized as follows.

The first stage. This stage includes a loss of water of hydration in the three polymer samples in the temperature range from room temperature up to 100°C. The estimated weight losses for this step were found to be 6.4%, 5.98%, and 5.22% for C10, C12, and C16, respectively. The calculated weight losses (based on the repeat units in the suggested structures in Scheme 1) are 6.42%, 5.83%, and 4.94% for P_{C10}, P_{C12}, and P_{C16}, respectively. This stage shows a broad endothermic peak in DTA curves starting at 44.64, 43.77, and 47.27°C for P_{C10}, P_{C12}, and P_{C16}, respectively.

The second stage. In the temperature range between 100 and 200°C. From both TGA and DTA curves, the polymer samples show some thermal stability with no chemical or physical changes.

The third stage. In the temperature range between 200–300°C includes a loss of two HCl molecules from each repeat unit in the three polymer samples associated with an endo-thermic peak on DTA curves. The weight loss in this stage is found to be 6.20, 5.81, and 5.00%, whereas the calculated weight loss is 6.51, 5.92, and 5.01% for P_{C10}, P_{C12}, and P_{C16}, respectively.

The fourth stage. In the temperature range between 300–480°C includes the loss of all benzenoid alkyloxy groups except the OR attached to the quinoid benzene ring. The estimated weight loss of this stage is found to be 38.70, 42.50, and 50.30%, whereas the calculated data are 37.70, 41.09, and 47.50% for P_{C10}, P_{C12}, and P_{C16}, respectively. This stage is associated with an endothermic peak (1) followed by exothermic peak (2) on the DTA curves, which could be attributed to the break of covalent bond between benzene ring and OR (peak 1) and the combination between the alkyloxy radicals respectively (peak 2) (cf. Figure 5).



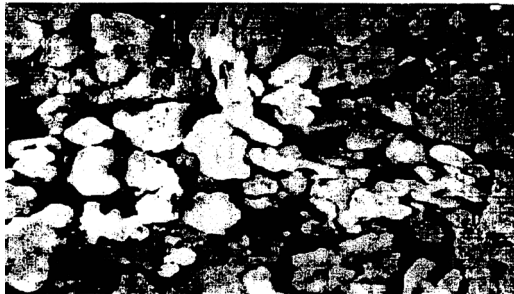
5 μm

[PC₁₀]



5 μm

[PC₁₂]



5 μm

[PC₁₆]

FIGURE 2 Electron microscope pictures of the prepared polymer samples PC₁₀, PC₁₂, and PC₁₆.

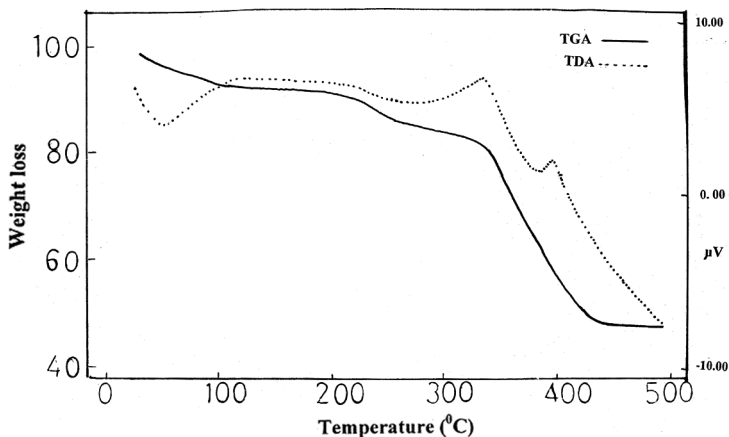


FIGURE 3 The thermogravimetric analysis (TGA) and differential thermal analysis (DTA) for the prepared polymer (P_{C10}).

The last stage. (Above 500°C). In this stage residual weights are found to be 48.7, 46.7, and 39.5%, whereas the calculated data are 49.78, 47.17, and 42.53% for P_{C10} , P_{C12} , P_{C16} , respectively. The aforementioned residual weights in this stage correspond to quinoid unit attached with alkyloxy group, in addition to Chromium oxide residue. From the data the authors suggest a thermal decomposition mechanism for the prepared polymer samples. This mechanism is

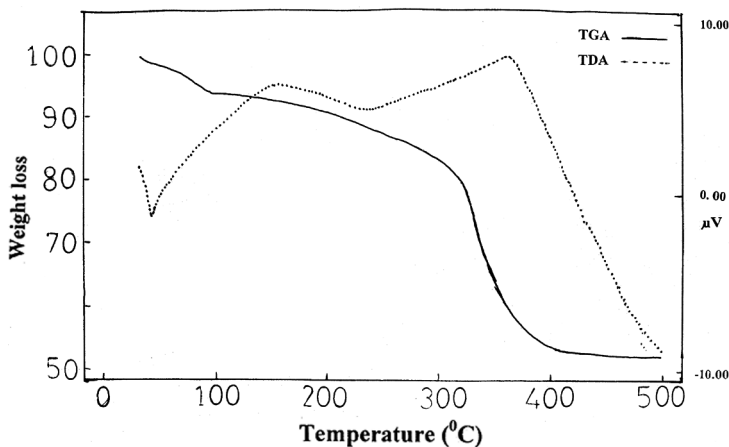


FIGURE 4 The thermogravimetric analysis (TGA) and differential thermal analysis (DTA) for the prepared polymer P_{C12} .

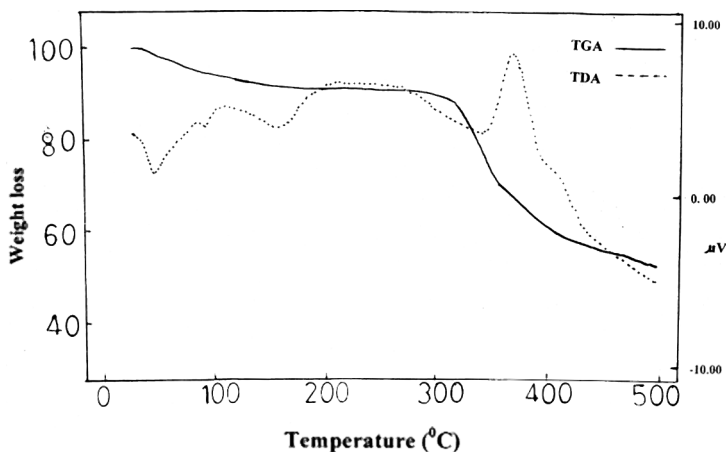


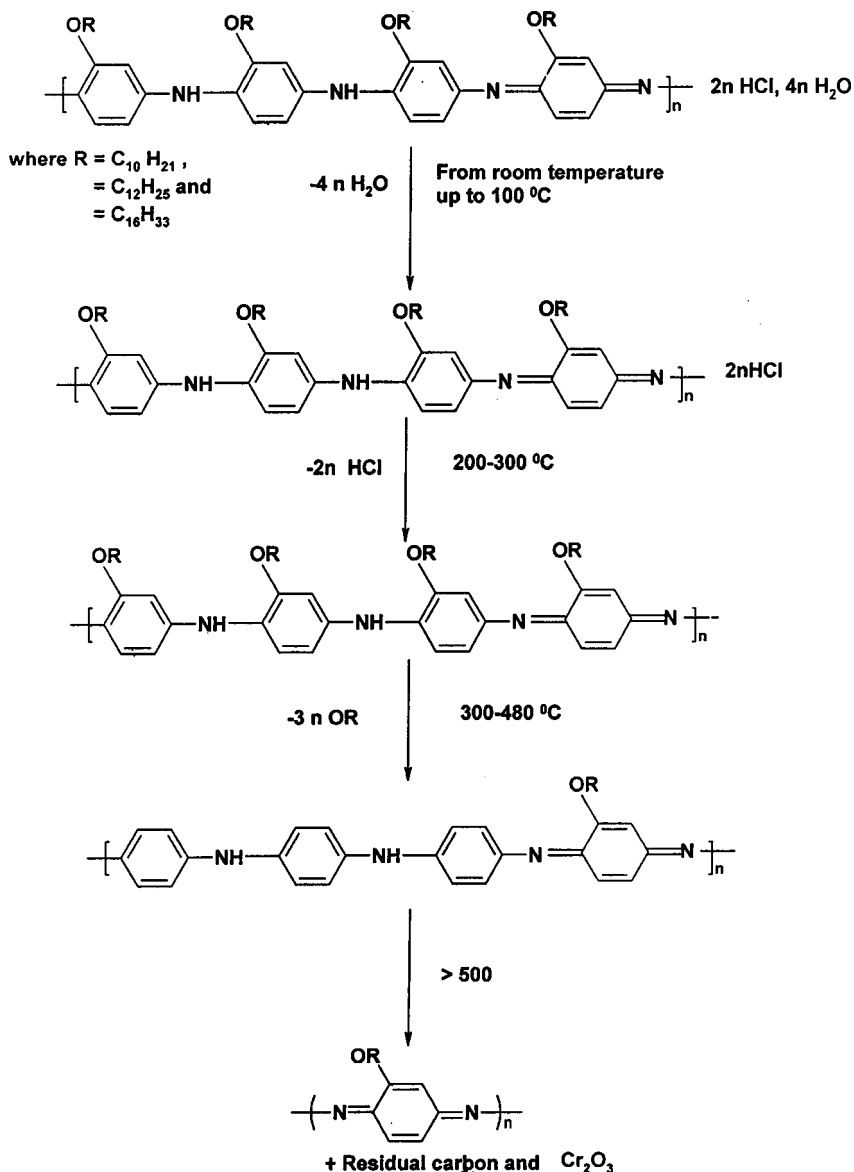
FIGURE 5 The thermogravimetric analysis (TGA) and differential thermal analysis (DTA) for the prepared polymer (P_{C16}).

represented in Scheme 1 and the fragmentation process are also summarized in Table 1, from which it can be concluded that the substitution of H atom in 3-hydroxyaniline by C10, C12, and C16 alkyl chain enhances the thermal stability of the prepared polymer samples.

Electrical Properties of the Prepared Polymeric Surfactants

The pressed samples were used for the investigation of the temperature effect on the electrical properties. The effect of temperature on both dielectric constant and a.c. conductivity of P_{C10} , P_{C12} , and P_{C16} were investigated at different frequencies (120 , 10^3 , 10^4 , and 10^5 Hz). The data are graphically represented in Figures 6–11. From the figures, it is clear that:

1. The dielectric constant (ϵ) increases with raising the temperature up to 350°K in case of P_{C10} , 360°K in case of P_{C12} , and 365°K in case of P_{C16} ; it then decreases up to about 410°K . A phase transition is observed at 350, 360, and 365 K for P_{C10} , P_{C12} , and P_{C16} , respectively. From these figures it can be concluded... that the T_g -value increases with increasing of side chain length. This could be attributed to the increase of the electron donating effect of the substituent side chain (C10, C12, and C16), which leads to more delocalization of π electrons in benzene rings and consequently higher conjugation will take place.



SCHEME 1 Stages in the degradation of the obtained polymers.

2. The high dielectric constant values of the prepared polymeric samples at the maximum point are $\approx 3.2 \times 10^4$, 4.5×10^4 , and 5×10^6 for P_{C10}, P_{C12}, and P_{C16}, respectively (cf. Figures 6, 8, 10). This may be

TABLE 1 Thermal Behavior of the Prepared Conducting Polymers

Fragment part	PAN ± (34)		POH (26)		POCH ₃ (24)		PC10		PC12		PC16	
	Calculated %	Found %	Calculated %	Found %	Calculated %	Found %	Calculated %	Found %	Calculated %	Found %	Calculated %	Found %
4H ₂ O	14.2	14.6	12.61	12.82	11.48	12.103	6.417	6.4	5.834	5.98	4.938	5.22
2HCl	14.4	15.0	24.69	24.72	11.62	11.8	6.506	6.2	5.916	5.81	5.007	5.00
3OR	—	—	—	—	20.01	19.75	37.7	38.7	41.086	42.5	47.53	50.29
Above 500°C	50.69	50.1	39.4	40.2	40.61	40.74	49.377	48.7	47.174	46.71	42.525	39.51

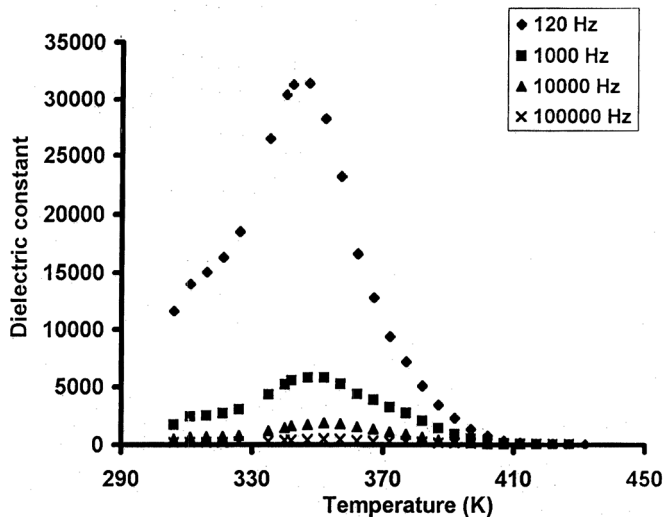


FIGURE 6 The effect of temperature on the dielectric constant of PC10.

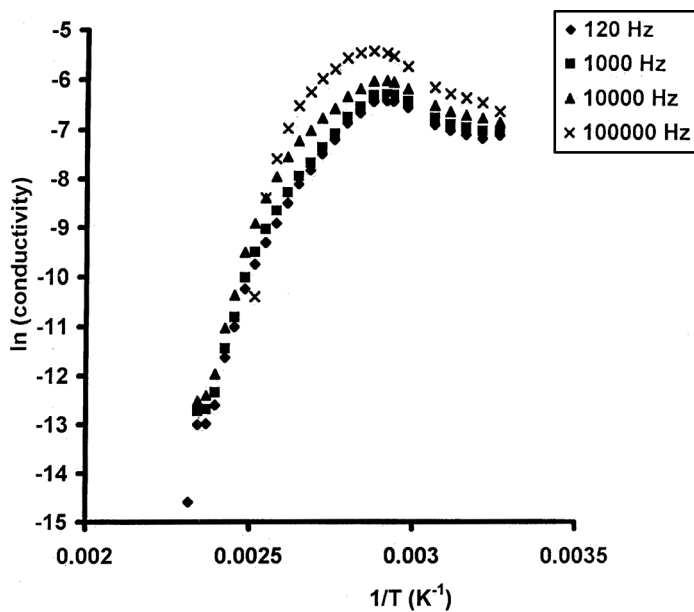


FIGURE 7 The effect of temperature on the a.c. conductivity of PC10.

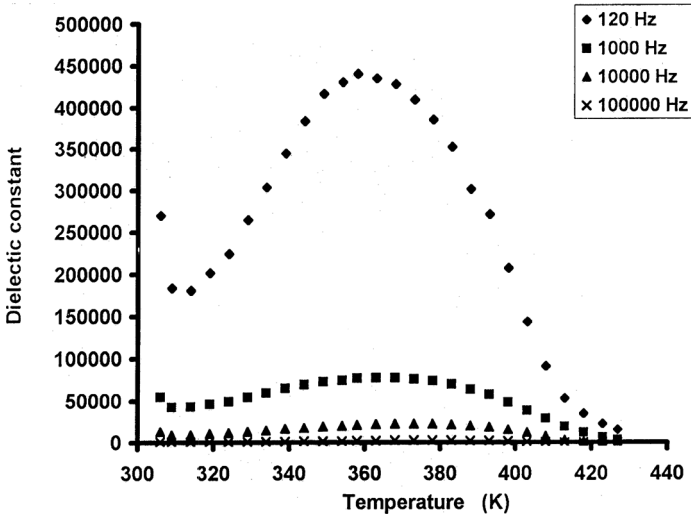


FIGURE 8 The effect of temperature on the dielectric constant of PC12.

due to the presence of small amount of protonation within the insulator region of the polymer samples, which has only limited short range order. These results give an indication for some improvement of the electrical properties [36]. The temperature

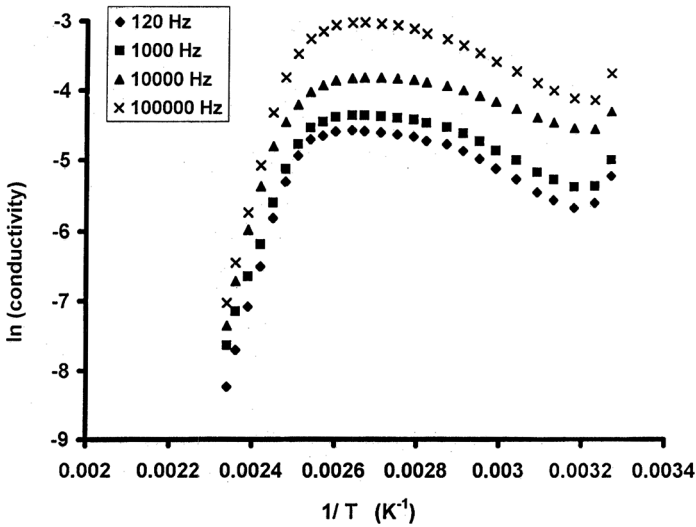


FIGURE 9 The effect of temperature on the conductivity of PC12.

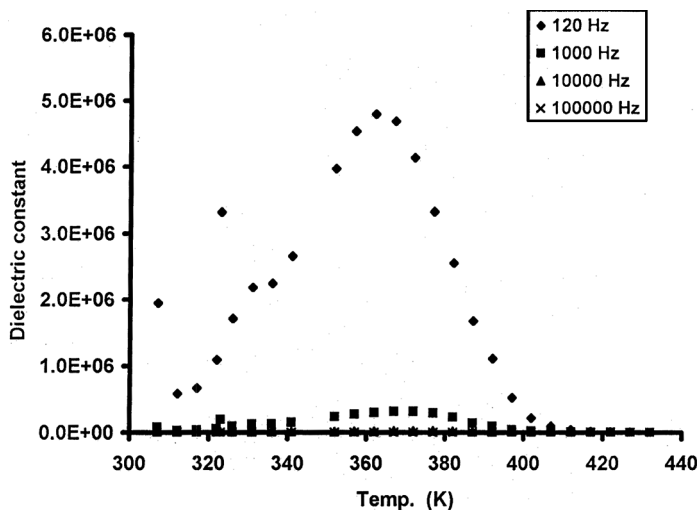


FIGURE 10 The effect of temperature on the dielectric constant of PC16.

corresponding to the peak maximum in each case of the investigated polymeric samples P_{C10} , P_{C12} , and P_{C16} is found to be frequency independent while it becomes sharper at lower frequencies.

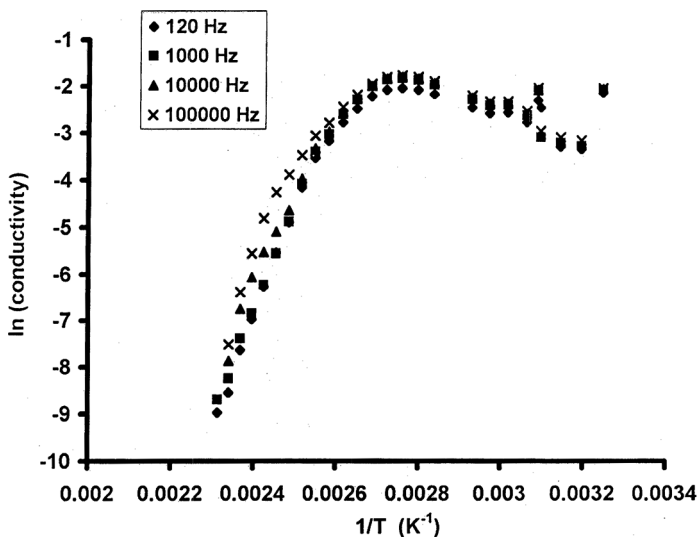


FIGURE 11 The effect of temperature on the conductivity of PC16.

TABLE 2 Dielectric Constant and A.C.-Conductivity of the Prepared Polymers in Comparison with Polyaniline (PANI), Poly 3-Hydroxy Aniline (Pm-HANI), and Poly 3-Methoxy Aniline (Pm-MeOANI)

Parameter	PANI (36)	Pm-HANI (26)	Pm-MeOANI (24)	PC10	PC12	PC16
Dielectric constant max.	6×10^5	6.5×10^5	4×10^4	3.2×10^4	4.5×10^4	5×10^6
Ln a.c. conductivity max.	-2.5	-3	-5	-6	-3	-2

TABLE 3 The Grain Size Values Obtained from Scanning Electron Microscope (SEM) and X-ray Diffraction Parameters

Polymer name	PANI (36)	POH (26)	POCH ₃ (24)	PC10	PC12	PC16
Grain size range (μm)	4.75–14.28	1.3–11.79	2.25–15	0.45–6	2.5–10	3.2–15
2θ	—	32.708	—	31.623	31.623	—
Intensity of the peak at 2θ (KCPs)	—	0.25	—	0.112	0.093	—
d-spacing	—	2.7356	—	2.8269	2.8269	—

3. Also, from Figures 7, 9, and 11 it is clear that the a.c. conductivity increases with raising the temperature at the different frequencies up to 350, 360, and 365°K and then decreases up to $\approx 410^\circ\text{K}$. Also, the a.c. conductivity-1/T curves reveal that the same phase transition present in ϵ -T curves is present at approximately the same temperatures. From the a.c. conductivity-1/T curves, it can also be concluded that the maximum a.c. conductivity values are of about 10^{-6} , 10^{-3} , and 10^{-2} s/m for PC₁₀, PC₁₂, and PC₁₆, respectively at 10^5 Hz. This means that by increasing the length of the substituted alkyloxy group of the side chain, the a.c. conductivity maximum increases.

CONCLUSIONS

From the aforementioned data one can conclude the following:

1. The alkyloxy group enhances the thermal stability, which increases as the number of carbon atoms in the side chain increases.
2. The substituted alkyloxy group enhances the a.c. conductivity of the polymeric samples, which increases with increasing number of hydrophobic moieties in the alkyloxy side group.
3. The substituted alkyloxy group enhances the solubility of the produced polymer in acid medium.

Therefore, there are some indications for the use of this type of polymers as corrosion inhibitors for different metals.

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