



## The Effect of Different Acid Chloride on Physical and Chemical Characterizations of Aryledinecycloalkanone Unsaturated Copolyesters

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### Authors' contributions

This work was carried out in collaboration between all authors. All authors read and approved the final manuscript.

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### ABSTRACT

Unsaturated copolyesters have been prepared and evaluated by chemical and physical instruments. The effect of acid chloride in the general physical characterizations of copolyesters was the aim of study. The solubility of polymers has been tested in different solvents. The viscosity average molecular weight was determined according to Mark-Hounwink equation using some standards polymers. The thermal stability and glass transition temperature was recorded by Thermogravimetric analysis and Differential thermal analysis respectively. The crystallinity has also been investigated by X-ray diffraction. Further, the morphology and shape of polymers were scanned by scanning electron microscopy. The electrical properties especially dielectrical constant of polymer material have been discussed with some detailed.

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## 1. INTRODUCTION

Unsaturated polyester resins are one of the most important resins for commodity glass fiber reinforced composites. They are obtained in a two-step process; first unsaturated and saturated acids or anhydrides are reacted with diols in a polycondensation reaction, secondly the resulting linear polyester prepolymer is dissolved in styrene into syrup like resin. The resin is finally processed into a rigid thermoset in a free radical copolymerisation between styrene and the double bonds in the polyester chain. The basic chemistry of unsaturated polyesters has remained very much unchanged for the last 40 years, although material development has been otherwise rapid [1-5].

The first unsaturated polyester resins of similar type as used today were synthesized in the 1930's. Carlton Ellis found that unsaturated polyester prepolymers could be mixed with styrene, and copolymerized into a rigid polymer. These resins became commercially important the next decade when they were reinforced with glass fibers giving structural products with high mechanical strength and low density. Today unsaturated polyesters are one of the most important matrix resins for composite materials [6-10].

In the composite the unsaturated polyester holds the reinforcement in place; it transfers the external loads to the reinforcement, and protects the reinforcement from the environment. If individual fibers are fractured, the matrix will redistribute the load to the surrounding fibers, thus preventing the complete failure of the material. The composite product will exhibit a broad range of mechanical, chemical, thermal and physical properties, depending on the composition of the unsaturated polyester [10-15].

The basic chemistry of linear unsaturated polyesters is rather simple. A mixture of unsaturated and saturated dicarboxylic acids is reacted with diols in a melt polycondensation. Mono-functional alcohols and acids are also used in some formulations to tailor the properties. The most traditional composition is maleic anhydride, o-phthalic anhydride and 1,2-propanediol, which are cheap raw materials. Other common raw materials are fumaric acid,

isophthalic anhydride, terephthalic acid, adipic acid, ethylene glycol, diethylene glycol, dipropylene glycol, neopentyl glycol and bisphenol A. The unsaturated polyester prepolymer is finally blended with styrene to a reactive resin solution, in which form the resin is sent to the end-user. The styrene acts both as a crosslinking agent, and as a viscosity reducer so that the resin can be processed. In conventional unsaturated polyesters the styrene content varies between 38 and 45 wt % [16-20].

One of the most important features in unsaturated polymers is the electrical properties especially the dielectric constant which defined as the ratio of the capacitance of a condenser containing the material to the capacitance under vacuum and symbolized by ( $\epsilon$ ). An article has focused on studying polymer thin films and their electrical properties with special interest to dielectric behavior for their important applications as insulation, isolation and microelectronics [21,22]. Recent articles have been published some conducting fillers to improve the magnitude of electrical conductivity of insulating polymers materials [23,24]. On the other hand, the dielectric constant was measured for a many kinds of natural polymers like cellulosic, [25] protein, and synthetic fibers [26].

The presented study focused on the preparation and characterization of new unsaturated copolyesters of arylidenealkanone. The influenced of using different acid chloride in the polymer main chain has great interest to discuss. The thermal properties e.g. thermal stability and glass transition temperature of polymers were investigated. The crystallinity, solubility and morphologic properties were also investigated. Further, the dielectrically properties have been measured for the solid unsaturated copolyesters.

## 2. EXPERIMENTAL

### 2.1 Materials

4-Hydroxy-3-methoxybenzaldehyde (vanillin) (99%, Alderich, Germany), 4-Hydroxybenzaldehyde (99%, Alderich, Germany) and 3-Hydroxybenzaldehyde (99%, Alderich, Germany). 4-Nitrobenzoic acid from (98%, Merck, Germany). Adipoyl, sebacoyl, isophthaloyl and terephthaloyl dichlorides (99%, Merck Germany),

Cyclohexanone (98%, Fluka Germany). Other chemicals were purchased from Aldrich, Merck, Fluka or Acrös companies and purified by standard methods.

## 2.2 Instrumentations

Nuclear Magnetic Resonance NMR spectra for monomers, model and polymers were recorded on a Bruker AVANCE 500 (USA) spectrometer (500 MHz) at room temperature in deuterated trifluoroacetic acid ( $\text{CF}_3\text{COOD}$ ). The infrared spectra FT IR were recorded by IR-470 infrared spectrophotometer, Shimadzu; and Pye Unicam SP3-100 spectrophotometer (Japan) milled with dry KBr pellet. 0.02 g of polymer in (3-5 ml) with different solvent was used to test the solubility at room temperature. Ubbelohde VWR (Germany) viscometer was used to determine the inherent viscosities of the polymer solution (0.5%w/v) 1n conc.  $\text{H}_2\text{SO}_4$  at  $30^\circ\text{C}$ . Philips X-ray Pw 1710 (Japan) diffracto-meter, using Ni-filtered  $\text{CuK}\alpha$  radiation was used for crystallinity measurements. Shimadzu TGA-50 and DTA-50 (Japan), thermal analyzer at heating rate  $10^\circ\text{C}/\text{min}$  in air, was used for Thermo-gravimetric analysis (TGA), and differential thermal analysis respectively. Scanning Electron Microscopy (SEM) using a JSM-5400 LV instrument was used for the morphology feature of the polymer, images were recorded with a Pentax Z-50P Camera with Ilford film at an accelerating voltage of 15KV (USA). HIOKI 3532-50LCR Hi TESTER apparatus 4(Japan) in the form of disks with 10 mm diameters and different thickness was used to record the dielectric constant, capacitance, and inductance, resistance of solid polymers.

## 2.3 Preparation of Monomers 1, 2, 3 and Their Models 1a, 2a, 3a

In our previous work [27,28] monomers 1 and 3 and their models were synthesized. Here we prepared new monomer 2 and its model by the same procedure by condensation of 3-hydroxy benzaldehyde with cyclohexanone for 2 hrs. The product has been separated as brown needles from ethanol, yield% = 90%, melting point =  $214^\circ\text{C}$ .

The reaction of 2, 6- bis(3-hydroxybenzylidene) cyclohexanone and benzoyl chloride was used for preparation of model compound **2a**. It was separated as yellow plates from benzene yield, 93%, melting point  $216^\circ\text{C}$ .

$^1\text{H}$  NMR (500 MHz,  $\text{DMSO-d}_6$ ):  $\delta$  (ppm) = 9.77 ppm (s, 2H, 2OH group ), 7.68 ppm (s, 2H of  $2\text{CH}=\text{C}$  ), 6.83 - 7.36 ppm ( m , 8H of Ar -H ) , 2.87 ppm (m , 4H of  $2\text{CH}_2$  ), 1.86 ( p , 2H of  $\text{CH}_2$ , cyclohexanone ).

FT IR (KBr) (**2 monomer**):  $\nu$  = 1695-1710  $\text{cm}^{-1}$  C=O cyclohexanone, 1595-1610  $\text{cm}^{-1}$  C=C stretching, 1595-1620  $\text{cm}^{-1}$  phenylene rings.

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ):  $\delta$ (ppm)= 8.25- 8.60 ppm (m , 4H 30,34,35,39 Ar-H), 7.53 – 8.14 ppm (m , 12H Ar-H), 7.16 –7.27 ppm ( s , 2H , C =CH ), 2.60-2.90 ppm ( t ,4H,  $2\text{CH}_2$  ), 1.30 – 1.87 ppm (P , 2H  $3\text{CH}_2$ , cyclohexanone).

IR (KBr) (**2a model**):  $\nu$  = 1740 (s) (C=O of ester group), 1675 (s) (C=O of cyclohexanone), 1610 (s) (C=C)

## 2.4 Preparation of 4, 4-azodibenzoyldichloride

The general procedure was described in lecture [29,30] .

## 2.5 Preparation of Copolymers

The interfacial condensation polymerization has formed between two phases organic for acid chloride and inorganic for bisphenol monomers. A mixture solution of two different monomers (mol/mol ) of diarylidencyclohexanone, and a suitable quantity of sodium hydroxide; that is stoichiometric quantity (0.02 mol ) of 100% excess (0.04 mol ) dissolved in 100 ml of water was introduced to a three necked, round bottomed flask ( $500\text{ cm}^3$ ) equipped with a mechanical stirrer (2000 rpm/min ), dry nitrogen inlet and out let, and dropping funnel. After 20 min. stirring a mixture (0.02 mol.) of acid chloride dissolved in 40 ml methylene chloride was added over a period 5-10 min. at  $25^\circ\text{C}$  and vigorously stirring. The stirring was continued for 2h., during which yellow solid product separated out. The solid polymer was filtered off, washed by water, hot ethanol, hot acetone, and dried overnight under reduced pressure ( $10^{-2}$  P) at  $100^\circ\text{C}$ .

The actual amounts of each monomer used in the formation of unsaturated copolyesters series 4, 5, 6 have been summarized in Table 1.

**Table 1. Mole ratios of monomers, acid chlorides, physical states, yield%, viscosity average molecular weight and glass temperature of synthetic copolyesters series 4, 5 and 6**

Code	Acid chloride (0.04 mol) in 100 ml dry CH <sub>2</sub> Cl <sub>2</sub>	Yield [%]	Mv 10 <sup>3</sup> g/mol	T <sub>g</sub> [°C]
<b>Series 4</b>				
4a	4.03g	94	9.01	72
4b	4.03g	92	8.79	79
4c	3.67g	88	8.46	56
4d	4.77g	83	10.76	68
4e	6.15g	84	7.44	96
<b>Series 5</b>				
5a	4.03g	89	6.88	66
5b	4.03g	92	8.94	83
5c	3.67g	86	9.39	78
5d	4.77g	95	9.78	82
5e	6.15g	84	6.56	106
<b>Series 6</b>				
6a	4.02g	95	9.74	89
6b	4.02g	91	7.46	94
6c	3.67g	93	7.76	86
6d	4.77g	90	8.33	87
6e	6.15g	88	6.24	116

### 3. RESULTS AND DISCUSSION

Scheme 1 described the preparation of unsaturated copolyester series containing diarylidene-cyclohexanone moiety in the main chain by interfacial condensation polymerization technique. The general procedure used mole/mole of both monomers and two moles of acid chloride [31].

#### 3.1 Polymer Characterizations

##### 3.1.1 <sup>1</sup>H NMR spectra

BRUKER DRX-500 spectrometer was used to record H-proton <sup>1</sup>H-NMR of copolyesters in CF<sub>3</sub>COOD. The <sup>1</sup>H-NMR for unsaturated copolyesters (5a, 5d, 5e, 6a, 6b, 6e) was shown in Fig. 1. Each spectrum recorded the presence of; 0.92-1.07, 1.34-1.67, 3.00-3.52 ppm (m-CH<sub>2</sub>-cyclohexanone), 4.02-4.32 ppm (m-OCH<sub>3</sub>), and 7.02-8.92 (m-Ar-H). Moreover, polymer 5d with sebacate main chain showed at 2.48-2.92 ppm (m-CH<sub>2</sub>).

##### 3.1.2 IR spectra

KBr FT IR spectra showed characteristic bands  $\nu = 1730-1745 \text{ cm}^{-1}$  for C=O ester;  $\nu = 1690-1700 \text{ cm}^{-1}$  C=O cyclohexanone;  $\nu = 1590-1600 \text{ cm}^{-1}$  C=C stretching;  $\nu = 1590-1510 \text{ cm}^{-1}$  phenylene rings; and  $\nu = 1250-1260 \text{ cm}^{-1}$

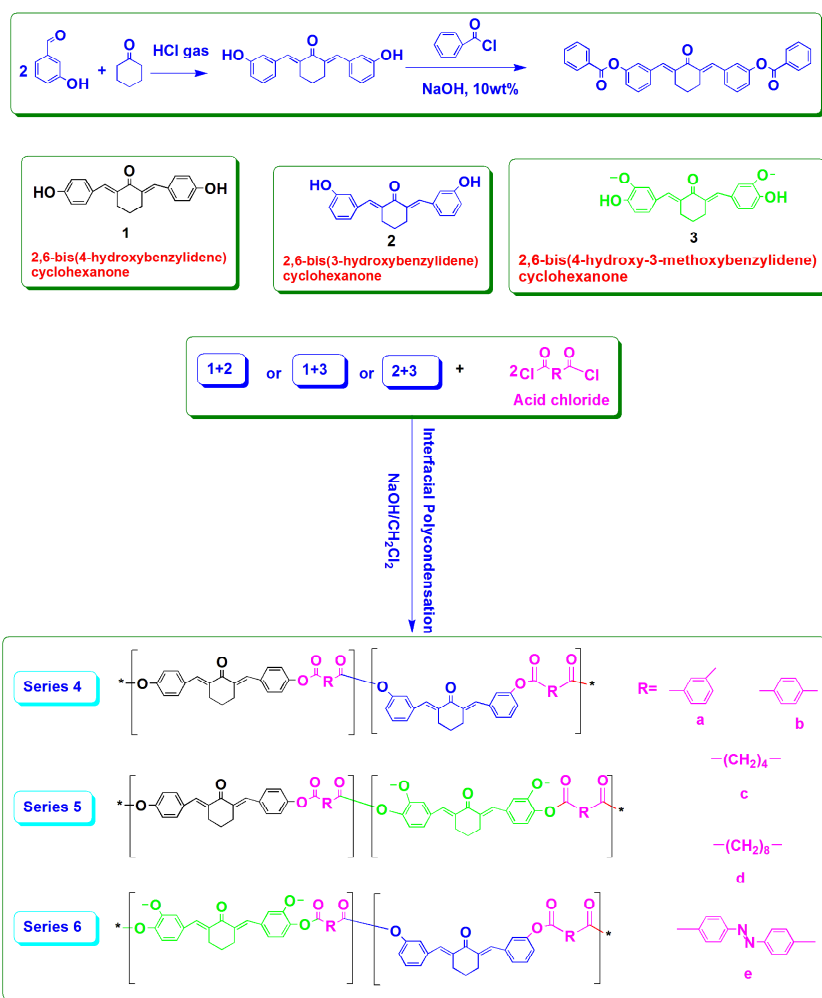
C-O-C bonds (ether linkage). All data was in logic with the chemical structure.

##### 3.1.3 Solubility

All polymers were tested for solubility in different solvents e.g. N-methyl-2-pyrrolidone (NMP), dimethyl formamide (DMF), m-cresol, dimethylsulphoxide (DMSO), CHCl<sub>3</sub>-acetone mixture, dichloroacetic acid (DCA), and concentrated H<sub>2</sub>SO<sub>4</sub>. Table 2, cleared the majority of the copolyesters were completely insoluble in NMP, m-cresol, CHCl<sub>3</sub>-acetone mixture. While, in the protic solvents as (DCA) (Cl<sub>2</sub>CHCOOH) or (TFA) (F<sub>3</sub>CCOOH) copolyesters are completely soluble at room temperature. As similar, in (conc.H<sub>2</sub>SO<sub>4</sub>) all copolymers are completely soluble at room temperature. The change in acid chloride from aliphatic and aromatic affected sharply in the solubility of copolyesters. We detected the slight solubility of aliphatic acid chlorides copolyesters in DMSO and DMF.

##### 3.1.4 The molecular weight (Mv)

Mark-Hounwink equation was used for determination of intrinsic viscosity which further used for the determination of viscosity a average molecular weight of copolyesters as discussed in recent articles and lectures [32-33]. All parameters are summarized Table 1.



Scheme 1. Preparation of monomers, model compound and unsaturated copolyesters 4, 5 and 6

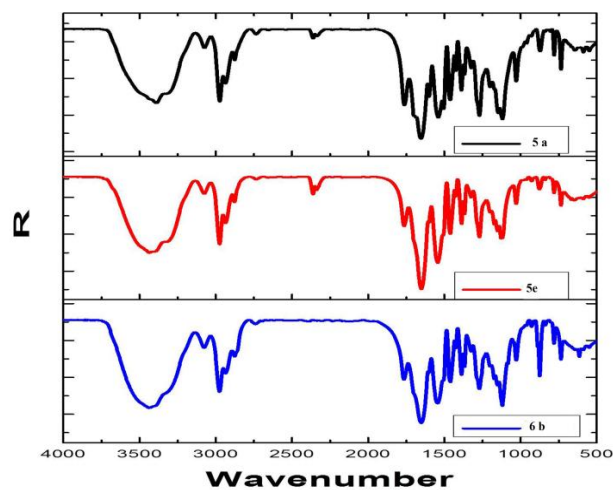


Fig. 1. FT-IR spectra of selected copolyesters

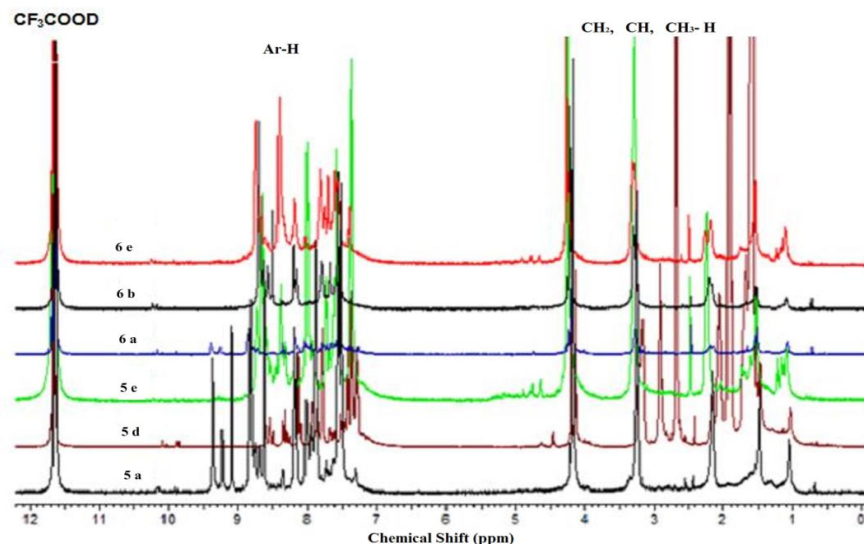


Fig. 2. <sup>1</sup>H NMR (CF<sub>3</sub>COOD) of copolyester

Table 2. Solubility parameters of copolyesters series 4, 5 and 6

Polymer code	1	2	3	4	5	6	7	8
<b>Series 4</b>								
4a	-	-	-	-	-	+	+	+
4b	-	-	-	-	-	+	+	+
4c	±	±	-	-	-	+	+	+
4d	±	±	-	-	-	+	+	+
4e	±	±	-	-	-	+	+	+
<b>Series 5</b>								
5a	-	-	-	-	-	+	+	+
5b	-	-	-	-	-	+	+	+
5c	±	±	-	-	-	+	+	+
5d	±	±	-	-	-	+	+	+
5e	±	±	-	-	-	+	+	+
<b>Series 6</b>								
6a	-	-	-	-	-	+	+	+
6b	-	-	-	-	-	+	+	+
6c	±	±	-	-	-	+	+	+
6d	±	±	-	-	-	+	+	+
6e	±	±	-	-	-	+	+	+

1 (DMF), 2 (DMSO), 3 (NMP), 4 (*m*-cresol), 5 (Chloroform: acetone 1:1), 6 (DCA), 7 (TFA), 8 (Conc. H<sub>2</sub>SO<sub>4</sub>). (+) Soluble at room temperature (RT); (±) partially soluble at RT; (-) insoluble at RT

### 3.1.5 Analysis of the thermal behavior

#### 3.1.5.1 Thermogravimetric analysis (TGA)

Fig. 3A aliphatic chain copolyesters showed a lesser stability than copolyesters with aromatic chains. Other observation was detected for unsaturated copolyesters based on vanillin in which the methoxy group increased the thermal stability.

Fig. 3B demonstrates copolyesters with azo group that decomposed in two stages. Primary is the elimination of azo group, probably as molecular nitrogen as discussed by Back and Black. [34]. The second involved polymer decomposition.

#### 3.1.5.2 Differential thermal analysis (DTA)

The glass transition temperature is one of the most important parameters for characterizing

polymeric material. The selected examples of copolyesters were examined by DTA measurements as shown in Fig. 3. The values of  $T_g$  were summarized in Table 1. From the DTA curves it is easy to determine the glass transition temperature  $T_g$  of these copolyesters. It should be noted that polymers with rigid chains would be expected to have  $T_g$  higher than those with flexible molecules. This is because, on melting, polymers with stiff backbones have lower conformational entropy changes than those with flexible backbones. The chain flexibility is enhanced by the presence of  $-(COO)-$  and by increasing the length of  $-(CH_2)-$  units in the main. The chain rigidity is enhanced by the presence of aromatic groups in the main chains [34].

The presence of acid chloride with azo dye group in the unsaturated copolyesters showed higher  $T_g$ s values than other copolyesters. This fact has interpreted as higher hindrances to internal rotation about primary valance bonds, and hence raises  $T_g$  [35]. Other observation attended to the higher  $T_g$ s values of copolyesters 5e and 6e than 4e, which attributed to the presence of methoxy groups as aside groups that further increase the intermolecular bonding in copolyesters chain [35].

### 3.1.6 X-ray analysis

The X-ray diffractograms (Fig. 5) showed a broad peak at  $2\theta = 20^\circ$ . This peak is associated to the intra-chain segments distance of 0.45 nm (determined using the Bragg's law). Fig. 5 showed some results;

1. The presence eight methylene groups (sebacoyl), increase the polymer chain flexibility
2. The presence of methoxyl groups as substituent in the phenyl ring caused some hindering between the repeating units and enforced its to the unsymmetrical orientation in the polymer chains and reduced the close packed structure and hence these copolyesters exhibit only a low degree of crystallinity [30].
3. Copolyesters (4e, 5e, 6e) with both azo – N=N- group and unsaturation bonds effect side by side in the polymer towards some extent of crystallinity [36-37].

### 3.1.7 Scanning electron microscopy (SEM)

(SEM) is used to study the shape, size and morphology of polymers. The change in surface morphology of copolyesters was investigated by The SEM. The morphological feature of unsaturated copolyesters 4a in Fig. 6, magnification of X=750 likes appear as fibrous aggregates. Higher magnification at X=1500 the surface likes coral shape as shown in Fig. 7. At X=350 magnification in Fig. 8, it seems as waxy-looking masses which likes gypstum; with higher magnification of X= 750. at X=350 magnification, showed fibrous aggregates Fig. 9, higher magnifications X= 750 like scapolite group. In Fig. 10, magnification at X=500, showed granoblastic, which likes quartzite rock. Fig. 11, magnification

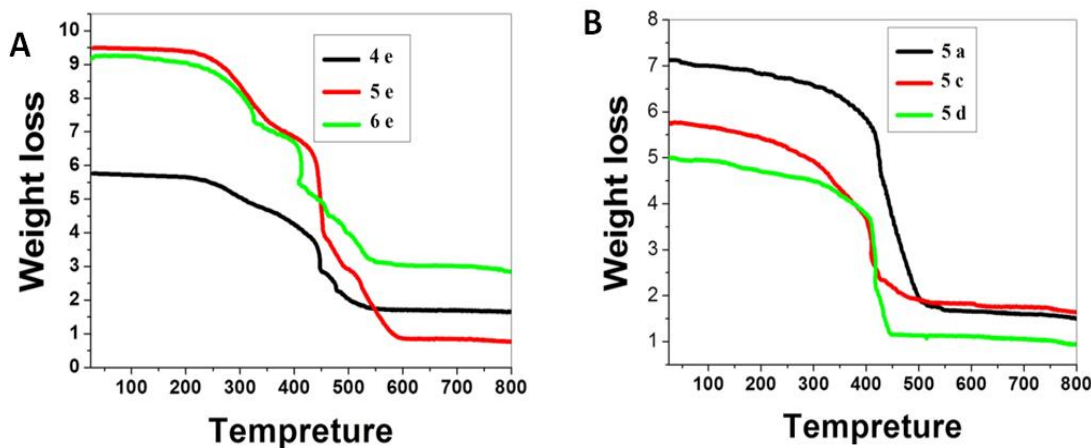


Fig. 3 A, B. TGA of copolyesters

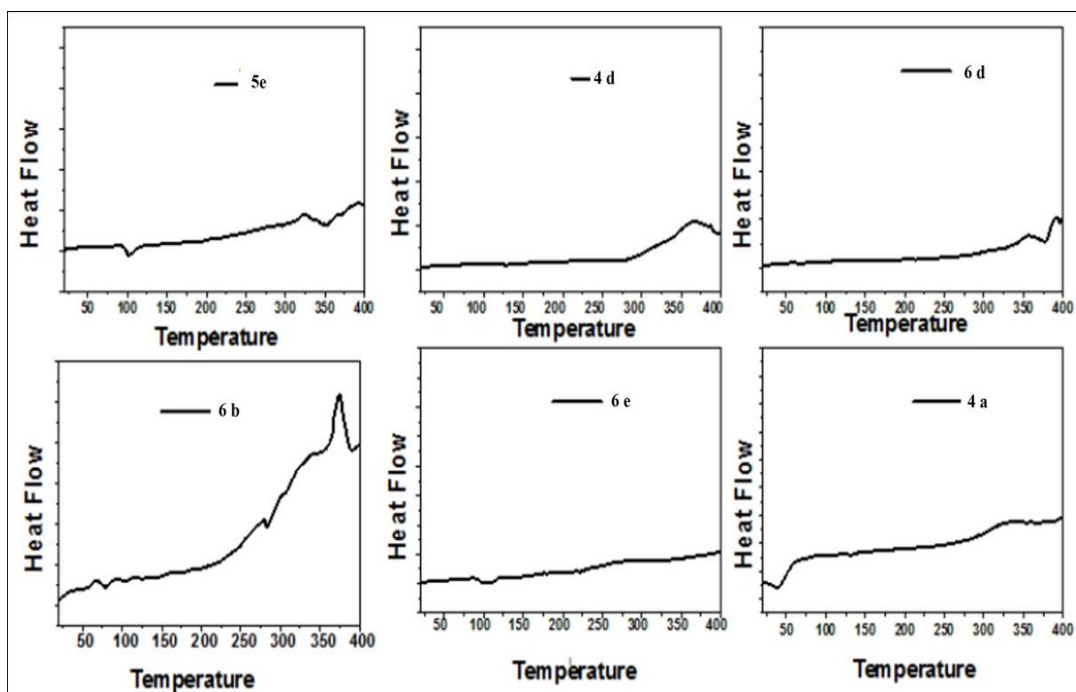


Fig. 4. DTA of copolyesters

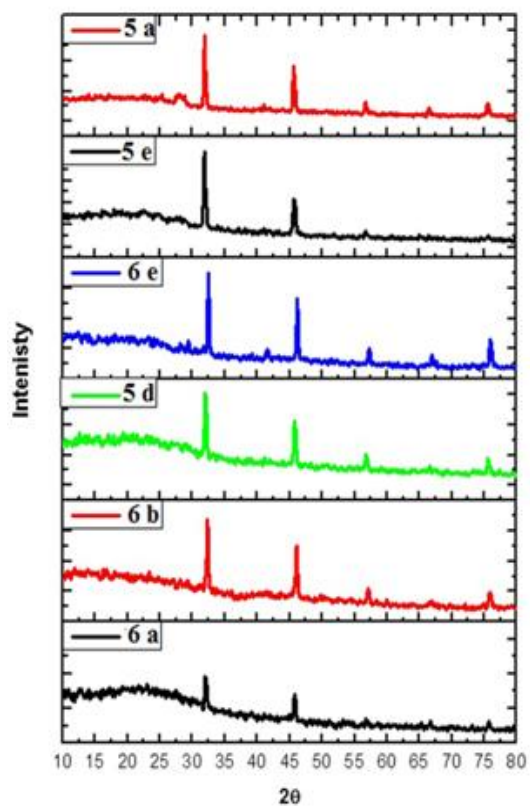


Fig. 5. X-ray diffraction patterns of copolyesters 5 (a,d,e) and 6(a,b,e)

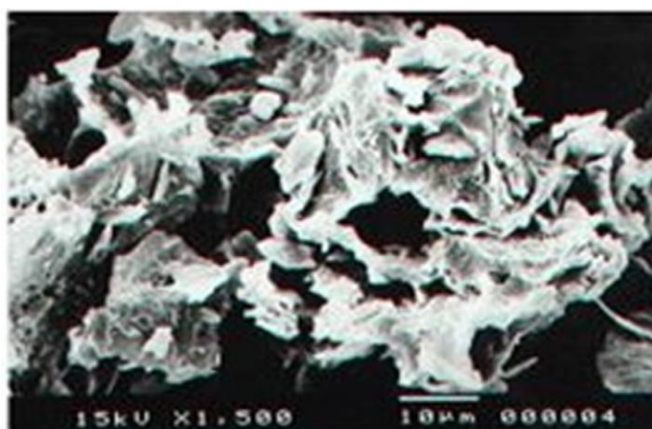
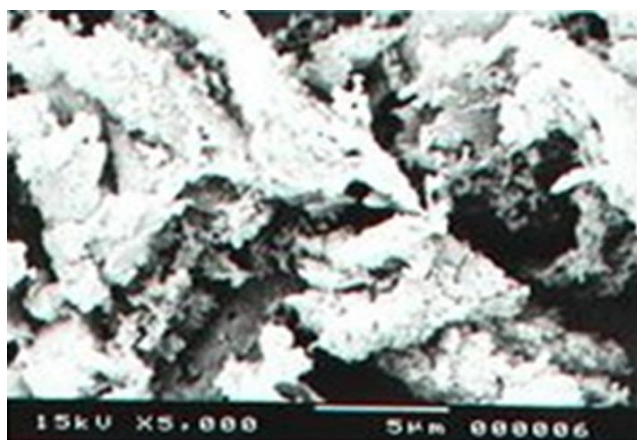
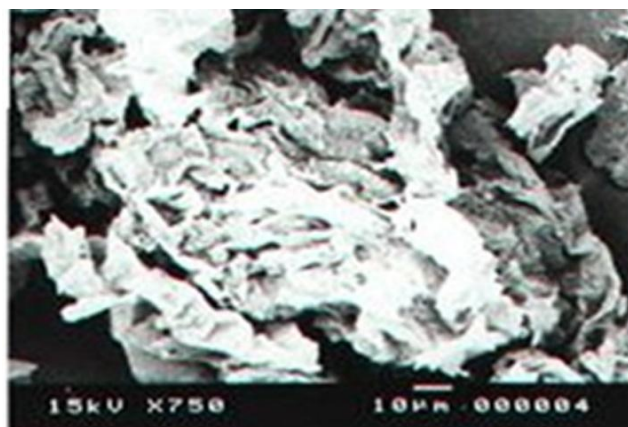


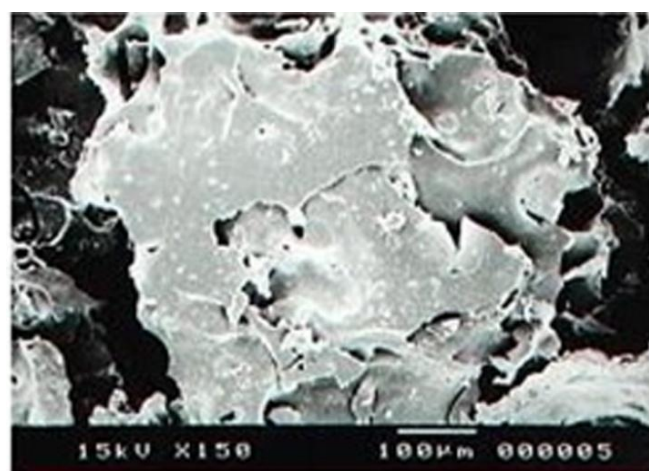
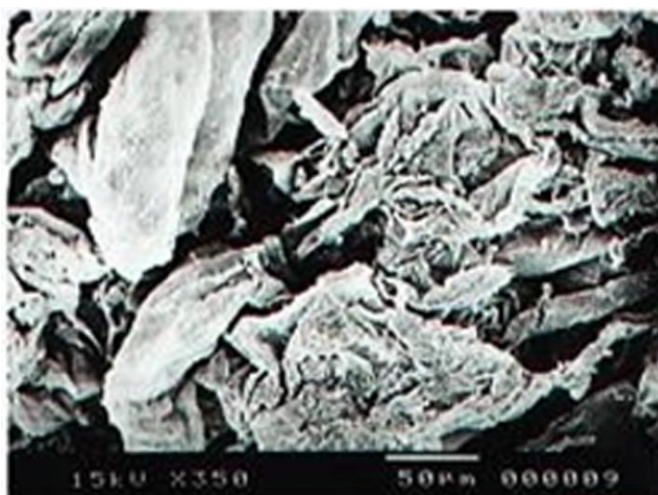
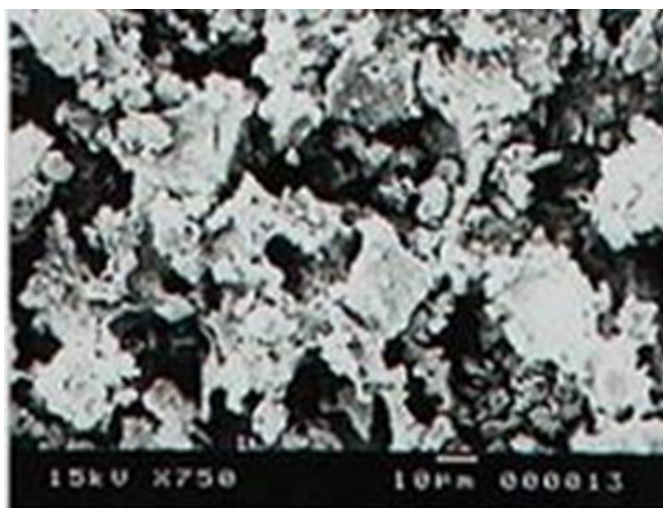
of X=150 appear as multi-layer of fibrous particles.

### **3.1.8 Electrical properties**

The electrical properties of selective unsaturated copolyesters have been evaluated with special attention to dielectrical ( $\epsilon$ ) constant of solid polymers.

Table 3 summarized the electrical parameters of unsaturated copolyesters with different acid chloride in the main chain. From Table 3 and Fig. 12 we can detect those polymers with aliphatic and aromatic acid chloride act as insulators, while others with aromatic azo acid chlorides have some electricity due to the polarization and the electron displacement of the azo group with aromatic conjugation.



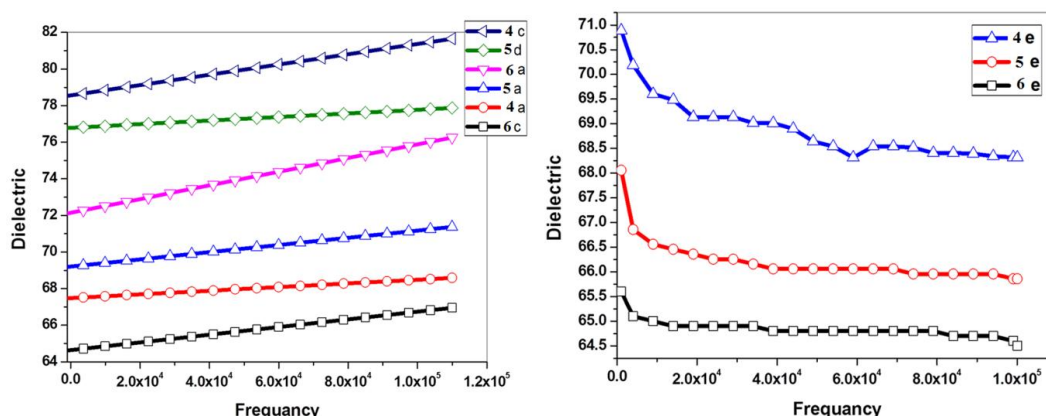


**Figs. 6-11. SEM images of copolyesters 4a, 4d, 6c, 5a, 5d, 4e, 6e surface at different magnifications**

**Table 3. The electrical parameters; relative dielectric constant ( $\epsilon'$ ), parallel resistance ( $R_p$ ) and parallel capacitance ( $C_p$ ) for selected copolyesters and terpolyesters**

Polymer	$d(m)$ $10^{-3}$	$\epsilon^a$	$R_p^b (\Omega)$ $10^7$	$C_p^c$ $10^{-11}$
4a	2.50	62-66	5-8	1.17
4b	3.50	81-87	5-7	1.09
4c	2.50	64-65	4-7	1.14
5a	3.20	80-85	7-9	1.56
5b	4.26	111-116	6-8.50	1.10
5c	1.58	43-45	4-6	1.22
6a	1.59	41-43	6-8	1.19
6b	4.00	98-104	7-9	1.26
6c	5.18	132-135	3-6	1.14
4e	3.20	80-85	7-9	1.55
5e	5.18	100-110	6-8	1.20
6e	1.50	70-73	4-6	1.30

<sup>a</sup> dielectric constant, <sup>b</sup> parallel resistance, <sup>c</sup> parallel capacitance

**Fig. 12. Relation between dielectric constant and frequency of copolyesters**

#### 4. CONCLUSION

The present work has multi-studies started with synthetic of new bis-phenol monomers and the chemical evaluation. The second step is the formation of unsaturated copolyesters from these polymers with different kind of acid chloride. The variation of acid chloride was the target of our study at which all physical characterization has been depended on the kind of acid chloride in the polymer chain. The copolyesters were characterized chemically and physically and all results were in logic case with their chemical structure. The solubility was very poor except of aliphatic acid chloride copolyesters. The thermal stability showed some different results related to acid chloride used in the main chain. Further, the crystallinity demonstrated higher value with aromatic methoxy polymers and lesser with aliphatic chains. The morphology showed the

presence of multi-layered indicating the heterogeneous of polymer surface. The dielectric parameters exhibited the insulator behaviour of the unsaturated copolyesters with some electricity for copolyesters with azo acid chloride. The general characterizations of the synthetic unsaturated copolyesters encourage the using as insulator.

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#### COMPETING INTERESTS

Authors have declared that no competing interests exist.

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