

# Ultrasonic Mechanical, Dialectical and Morphology Characterizations of Unsaturated Copolyesters and Terpolyesters

Momen S. A. Abdelaty

Polymer Lap, Chemistry Department, Faculty of Science (Assiut), Al-Azhar University, Assiut, Egypt  
Email: abdelatymomen@yahoo.com

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## Abstract

In a previous study we focused on the synthesis of new family of unsaturated copolyesters and terpolyesters by interfacial condensation polymerization. Here, in this study as complementary work of physical characterizations, we focused on the morphological features using X-ray and SEM investigation. Further, the mechanical properties of solid polymers were also studied by a new technique echo-pulse using ultrasonic impulses. This technique was used for solid polymers to determine the elastic moduli involving, Young's modulus, shear modulus, bulk modulus and micro-hardness modulus. The statistical relation between different kinds of copolyesters and terpolyesters with Poisson's ratio ( $\nu$ ) and micro-hardness were shown. The dielectric constant ( $\epsilon$ ) and the electrical resistance ( $R_p$ ) of the solid copolyesters and terpolyesters including aliphatic, aromatic and azo group in the main chain have been studied at room temperature. Moreover, the relation between frequency and dielectric constant for the selective polymers has been also illustrated.

## Keywords

Unsaturated Copolyesters, Terpolyesters, Ultrasonic Mechanical, Dielectric, Morphology

## 1. Introduction

Because unsaturated polyesters are so widely used, a number of strategies have been suggested [1] [2]. One recent method [3] involves alcoholises of waste resin with propylene glycol, followed by re-esterification with additional maleic anhydride. More styrene is then added to the reconstituted polymer for cross-linking.

Synthesis of polyesters based on biocatalysts and green monomers from re-

newable resources have extensively studied, due to the eco-friendly product, since both monomers and catalysts are from renewable resources; moreover, polymers are biodegradable [4]-[14]. Nowadays, many studies were focused on the formation of unsaturated polyester composites and their mechanical properties [15] [16]. On the other hand, others studied the unsaturated polyesters as layered silicate nanocomposites [17].

The dielectric constant of material ( $\epsilon$ ) can be defined as the ratio of the capacitance of a condenser containing the material to the capacitance under vacuum referred to. The capacitance of a condenser that measures the extent of charges was stored. 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 [18] [19]. Moreover, some conducting fillers to improve the magnitude of electrical conductivity of insulating polymers materials have been published in recent articles [20] [21]. The electrical conductivity of insulating polymers materials can be enhanced several orders of magnitude by incorporating some conducting filler in it [22] [23] [24]. The dielectric constant was measured for a variety of materials like cellulosic, [25] protein, and synthetic fibers [26].

Polymers are usually described as viscous liquid and elastic solids. An ideal linear elastic solid obeys Hooke's law; *i.e.* stress is proportional to the rate of change of strain. An ideal viscous liquid obeys Newton law; *i.e.* stress is proportional to the rate of change of strain [27]. Ultrasonic method and mechanical spectroscopy are non-destructive testing techniques and can be used for the determination of elastic properties of polymers [28]. Poisson's defined the ratio ( $\nu$ ) between transverse strain and longitudinal strain in the elastic loading direction [29]. For most polymer materials Poisson's ratio tending to 0.5. This can be noticed as a shift from elastic to viscoelastic [30]. Nowadays, many studies were focused on the formation of unsaturated polyester composites and their mechanical properties [31].

This study reports on the morphologic, mechanical and electrical properties of new class of unsaturated copolyesters and terpolyesters. They have been synthesized by interfacial condensation polymerization technique. The synthesis, chemical elucidations and some of physical characterizations have been reported previously.

## 2. Experimental

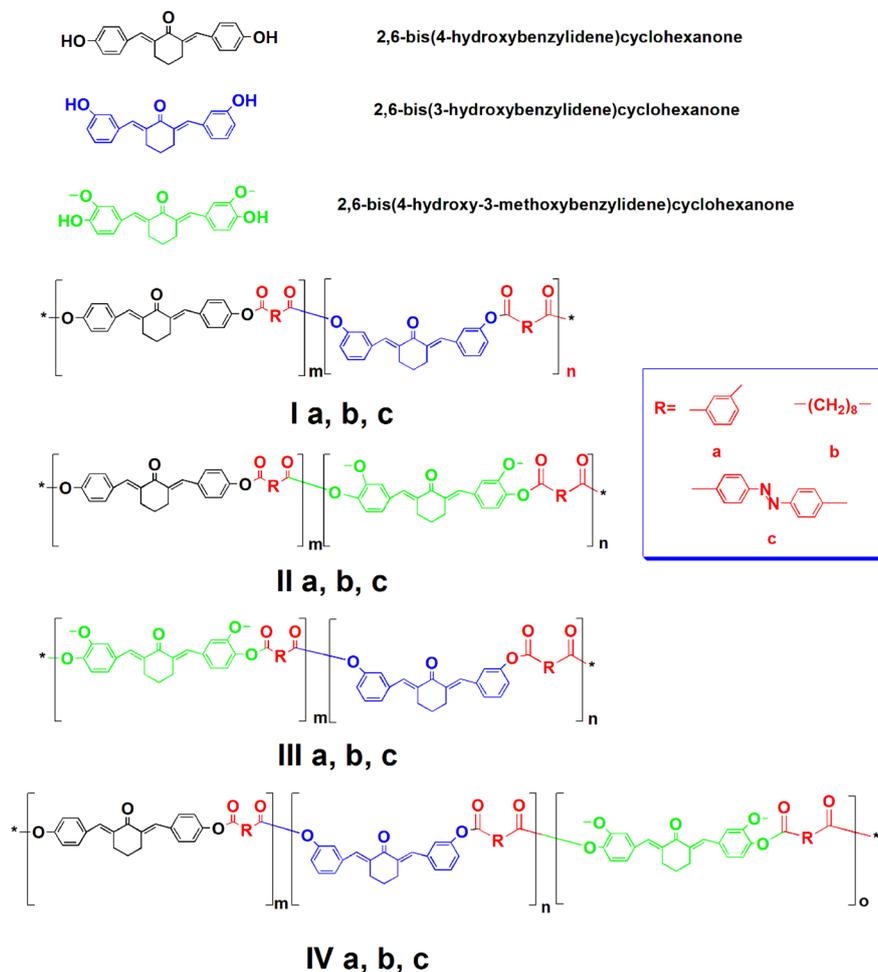
### 2.1. Monomers and Polymers Materials

They have been reported in previous study [1] [2]. The structures of monomers and polymers under investigations were shown in **Scheme 1**.

### 2.2. Instrumentations and Measurements

#### 2.2.1. X-Ray Diffraction Analysis

X-ray diffractographs of the polymer were obtained with Philips X-ray Pw 1710



**Scheme 1.** Structures of monomers and polymers have been synthesized and characterized in previous work and are used in the present study.

diffract-meter, using Ni-filtered CuK $\alpha$  radiation. Please do not revise any of the current designations.

### 2.2.2. Ultrasonic Mechanical Characterization Sample Preparation

#### 1) Formation of Polymer disc

The solid polymer was grinded by manual mortar. The polymer powder was then taken to QwikHandi-Press (used in IR instrument) under pressure of 5 ton to get a uniform pellet of polymer with a thickness 0.9 - 1.2 cm.

#### 2) Density measurements

The densities of polymers were determined at room temperature using the standard displacement method. The polymer sample was hanged by a Teflon wire (0.01 mm in diameter) to one arm of sensitive balance. Water was used as immersion liquid. Densities were calculated according to the following equation;

$$\rho = \rho_0 (W - W_t) / (W - W_t)(W_1 - W_{1t}) \quad (1)$$

where  $\rho_0$  is the density of water (0.9999 kg·m<sup>-3</sup> at RT 25°C),  $W$  and  $W_p$  are the weight of polymer pellet in air and water respectively,  $W_t$  and  $W_{1t}$  are weight of

suspended Teflon wire in air and water respectively.

### 3) Ultrasonic Velocity measurements

Ultrasonic waves travel through any solid material at a specific velocity related to the material characterization and its density. Here, we used the pulse technique which is widely used to generate ultrasonic measurements for solid and fluids. A pulse of sinusoidal voltage is applied to a piezoelectric transducer that is directly contacted with the samples under investigation. The electrical pulse converted to acoustical pulse that transmitted into the medium. The pulse propagates down the medium, and reflected to the opposite face. The time intervals between successive echoes can be measured and velocity of the wave is determined. Once, time intervals duration is obtained and the thickness of the samples determined the velocity of the ultrasonic wave of the sample can be calculated by Equation (2);

$$V = 2d/\Delta t \quad (2)$$

where  $d$  is the thickness of the sample and  $\Delta t$  is the time intervals (31).

In general, the solids are subjected to three types of stressing condition; uniaxial stress, triaxial stress and pure shear. If a uniaxial stress is applied on a body, then elongation in the same direction will be produced. Young's modulus ( $E$ ); defined as the ratio of the linear stress to linear strain. Shear of rigidity ( $G$ ); defined as the ratio of the shear stress to the shear strain. Bulk modulus ( $K$ ); defined as the ratio of the hydrostatic stress to the volumetric strain. Micro-hardness ( $H$ ); is the resistant of material to being penetrated and eroded by another material's shear projection. The four constants held as Equations (3)-(7);

$$L = \rho V_l^2 \quad (3)$$

$$G = \rho V_s^2 \quad (4)$$

$$E = 2(1+\nu)G \quad (5)$$

$$K = L - (4/3)G \quad (6)$$

$$H = (1-2\nu)E/6(1+\nu) \quad (7)$$

where  $\rho$  is the density of the material,  $L$  is the longitudinal elastic modulus,  $V_l$  and  $V_s$  are the longitudinal and transverse ultrasonic velocities and  $\nu$  is Poisson's ratio [32].

#### 2.2.3. Electrical Properties

The dielectric constant ( $\epsilon$ ), parallel resistance ( $R_p$ ) and parallel capacitance ( $C_p$ ) were determined for specimens in the form of discs. They were prepared as discussed lately, the diameter 10 mm and thickness about 7 mm. The measurements have been carried out at room temperature and frequency rang 100 Hz - 1 MHz using HIOKI 3532-50 LCR Hi TESTER.

### 2.3. Scanning Electronic Microscopy

The morphology of the polymer were examined by Scanning Electron Micro-

scopy (SEM) using a JSM-5400 LV instrument; images were recorded with a Pentax Z-50P Camera with Ilford film at an accelerating voltage of 15 KV.

### 3. Results and Discussions

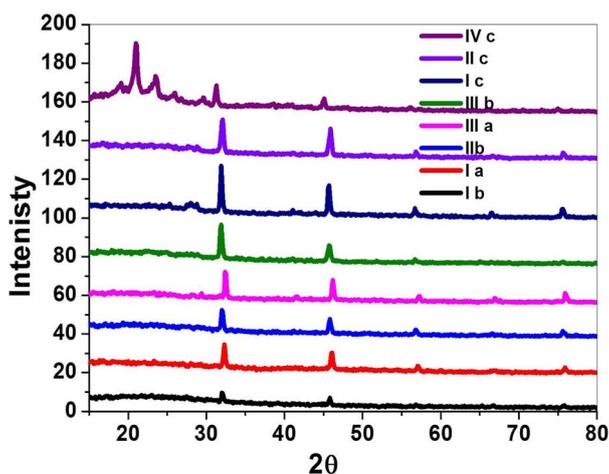
#### 3.1. $^1\text{H}$ NMR and IR

In the previous study we discussed the chemical investigation of these polymers using  $^1\text{H}$ NMR and IR showed good agreement with the chemical structure [1] [2].

#### 3.2. X-Ray Diffraction

X-ray diffractograms of selected examples of copolyesters showed an amorphous patterns in the region  $2\theta = 5^\circ - 30^\circ$ , after this region showed a few reflections of sharpness peaks with an amorphous back ground in the region  $2\theta = 30^\circ - 60^\circ$ , this indicate some degree of polycrystallinity of the polymer. It was noted from **Figure 1** that, the presence of cyclohexyl ring in the polymer chain beside the presence of methylene groups, four or eight, increase the polymer chain flexibility, that urge the approach and mutual attraction of the adjacent chains might be induced some extent of crystallinity. Moreover, it should be noted that the presence of methoxyl groups as substituent in the phenyl ring caused some hindering between the repeating units and enforced it's 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 [33]. Moreover, in copolyesters (IVe, Ve, Vie) the presence of  $-\text{N} = \text{N}-$  as a polar group in addition to  $-\text{C} = \text{C}-$  bonds induces some order between two adjacent chains in the polymer towards some extent of crystallinity [34] as shown in **Figure 1**. X-ray diffractograms of terpolyesters (IVa, c, d) showed an amorphous halo patterns in the region  $2\theta = 5^\circ - 60^\circ$  this indicate a low degree of crystallinity at this region as shown in **Figure 1**.

Terpolyester (IVb) that contain sebacoyl with eight methylene groups in the



**Figure 1.** X-ray diffraction of selective copolyesters and terpolyesters.

main chain is higher crystalline than the other terpolyesters (IVa) due to the approach and mutual attraction of the adjacent chains, which increase the polymer chain flexibility, and induced some extent of crystallinity. Terpolyester (IVc) that contain azo group as a polar group in the main chain have low degree of crystallinity due to the presence of azo group will facilitate the movement of the main chain, therefore it will increase elasticity of polymer [35].

### 3.3. Mechanical Properties

Values of longitudinal, transverse ultrasonic velocities and elastic moduli constants for different copolyesters and terpolyesters based on diarylidencyclohexanone are cleared in **Table 1**. The ultrasonic changes may be explained on the basis of physical properties of copolyesters and terpolyesters demonstrated the elastic moduli parameters as shown in **Table 2**. In polymer (Ib) with aliphatic group (sebacoyl) in the main chain are more elastic than polymer (Ia) with aromatic group (isophthaloyl) in the main chain. This attributed to, the presence of flexible aliphatic units greatly increase the mobility of the copolyesters, and hence have low micro-hardness [35]. The presence of methoxy group in the main chain as in polymer (II a-c), (III a-c) may be increase the intermolecular force of attraction and hinders the three-dimensional packing of the polymer main chain and this copolyesters exhibit low degree of crystallinity [36]. In polymer (Iva-c) terpolyesters, they have higher long chain than copolyesters, elasticity will increase due the effect of the neighbouring group, polymer (IVb) is more elastic than (Iva) as discussed later [37]. Polymer with azo groups in the main chain showed average elasticity between those with aromatic and aliphatic groups, this can be attributed to the mobility of azo group in presence of aromatic chains [35]. The statistical clarifications of different kinds of copolyesters

**Table 1.** Densities, ultrasonic velocities (longitudinal, transverse) and Poisson's ratio for copolyesters and terpolyesters.

Polymer	$\rho^a$ (Kg/m <sup>3</sup> ) 10 <sup>3</sup>	$V_L^b$ (m/s)	$V_s^c$ (m/s)	$\nu^d$
Ia	1.38	5855	3155	0.29
Ib	1.22	6356	3272	0.32
Ic	1.39	5945	3214	0.29
IIa	1.39	5656	3327	0.23
IIb	1.28	6139	3394	0.28
IIc	1.41	5657	3251	0.25
IIIa	1.37	5835	3213	0.28
IIIb	1.23	6262	3285	0.31
IIIc	1.40	5900	3280	0.27
IVa	1.41	6015	3234	0.29
IVb	1.30	6527	3221	0.34
IVc	1.43	6345	3245	0.32

<sup>a</sup>density; <sup>b</sup>longitudinal velocity; <sup>c</sup>transverse velocity; <sup>d</sup>Poisson's ratio.

**Table 2.** Elastic moduli for selected copolyesters and terpolyesters.

Polymer	(L) <sup>a</sup> GPa	(G) <sup>b</sup> GPa	(E) <sup>c</sup> GPa	(K) <sup>d</sup> GPa	(H) <sup>e</sup> GPa
Ia	47.30	13.74	35.45	28.98	1.92
Ib	49.28	13.06	34.47	31.86	1.56
Ic	49.13	14.36	37.05	30.00	2.01
IIa	44.46	15.38	37.83	23.95	2.77
IIb	48.24	14.74	37.73	28.58	2.20
IIc	45.12	14.90	37.25	25.25	2.48
IIIa	46.64	14.14	36.28	27.79	2.08
IIIb	57.57	15.06	39.75	37.50	1.81
IIIc	48.73	15.06	38.25	28.65	2.31
IVa	51.01	14.75	38.05	31.43	2.10
IVb	55.38	13.49	36.15	37.39	1.44
IVc	48.23	13.27	34.77	30.53	1.68

<sup>a</sup>Longitudinal Modulus; <sup>b</sup>Shear Modulus; <sup>c</sup>Young's Modulus; <sup>d</sup>Bulk Modulus; <sup>e</sup>Micro-Hardness.

and terpolyesters with Poisson's ratio ( $\nu$ ) and micro-hardness (H) were shown **Figure 2** and **Figure 3** respectively.

### 3.4. Electrical Properties

The dielectric constant ( $\epsilon$ ) and the electrical resistance  $R_p$  of the two types of copolyesters and terpolyesters including aliphatic, aromatic and azo group in the main chain have been studied carefully at room temperature as clear in **Table 3**.

**Figure 4** showed the relation between frequency and dielectric and these indicating that copolyesters and terpolyesters are good insulator. The difference in the dielectric values can be interpreted to the difference in samples thickness which has a direct relation.

Polymers which contain azo group in main chain show inversely proportional with frequency due to the presence of (-N = N-) group with lone pair of electrons on nitrogen atom and  $\pi - \pi^*$ ,  $n - \pi^*$  transition which increase polarizability for the samples and decrease with frequency (36). Also the values of the electrical resistance  $R_p$  of the two groups are about 107  $\Omega$ . So we can conclude that these copolyesters and terpolyesters can be used as a dielectric medium and as insulator in different applications.

### 3.5. Morphological Features (SEM)

The morphology of the synthesized copolyesters was examined by scanning electron microscopy (SEM). The SEM samples were prepared by putting a smooth part of polymer on a copper holder and subsequently coating it with gold palladium alloy. SEM images were taken on a penta Z50 P Camera with IL ford film at an accelerating voltage of 15 kV using a low-dose technique [37].

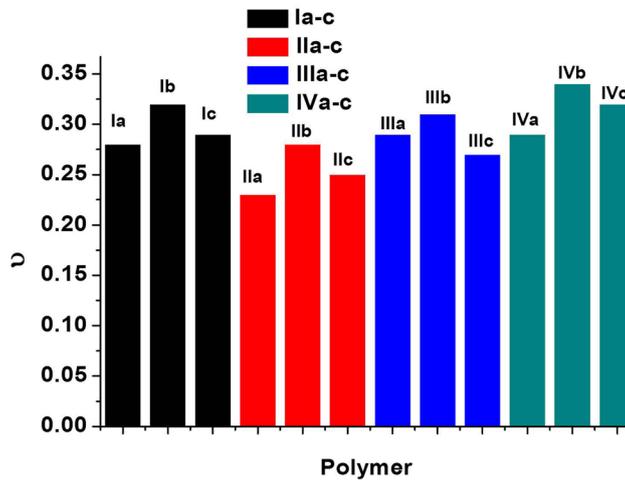


Figure 2. Statistical clarification for copolyesters and terpolyesters with Poisson's ratio ( $\nu$ ).

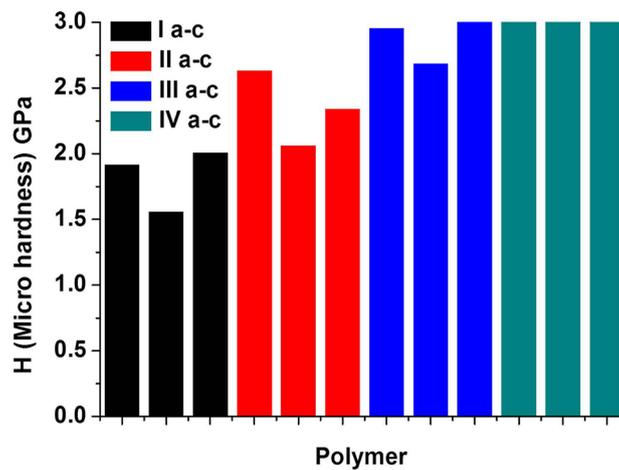


Figure 3. Statistical clarification of copolyesters and terpolyesters with micro-hardness (H).

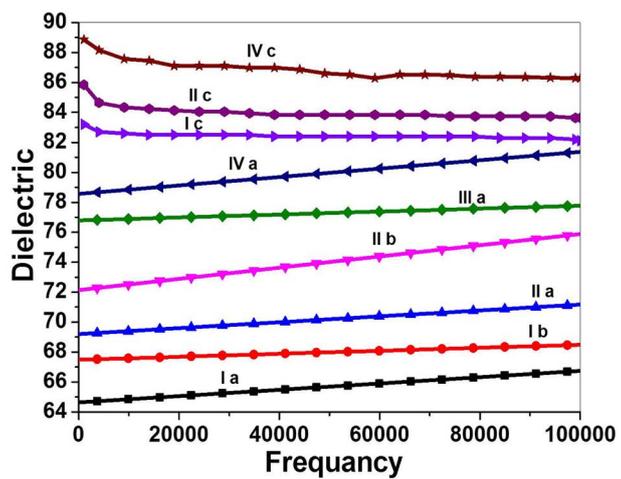


Figure 4. The relation between frequency and dielectric constant for selected copolyesters and terpolyesters.

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

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

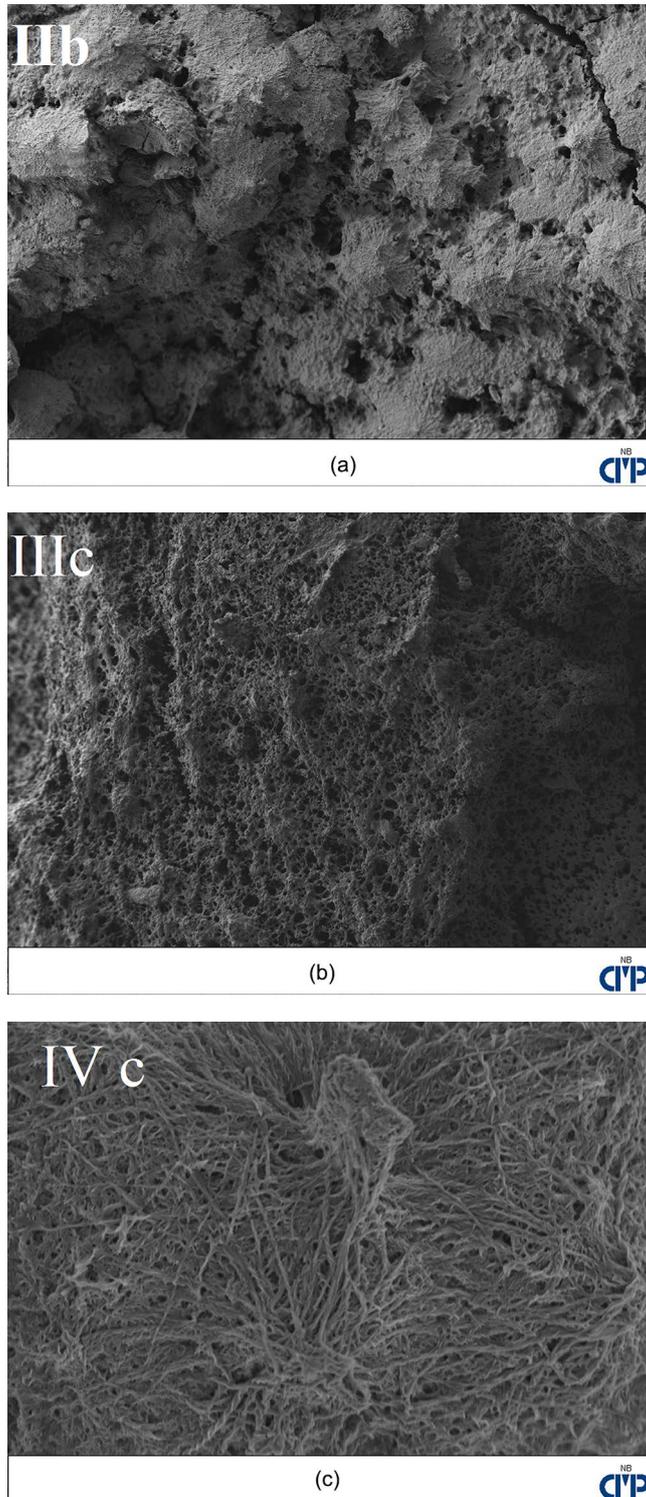
<sup>a</sup>dielectric constant, <sup>b</sup>parallel resistance, <sup>c</sup>parallel capacitance.

The study of selected examples of copolyesters showed that the surface of copolyesters (Ia), magnification of X = 750 likes scapolite shape which appear as fibrous aggregates. For the surface of copolyesters (Ic), magnification of X = 750 likes coral shape. For the surface of copolyesters (IIa), magnification of X = 350 appear as granular and compact waxy-looking masses which likes gypsum. For the surface of copolyesters (IIb), magnification of X = 500, showed granoblastic, mosaic-type, which likes quartzite rock as shown in **Figure 5(a)**. Copolyesters (IIc), magnification of X = 500 appear as big granular. **Figure 5(b)** copolyesters (IIIc), magnification of X = 150 appear as multi-layer of fibrous particles. Terpolyester (IVa), magnification of X = 200 likes fibrous aggregates. **Figure 5(c)** the surface of terpolyesters (IVb), magnification of X = 200 showed coral shape with porous particles; (IVc), magnification of X = 750 fibrous aggregates.

#### 4. Conclusion

In the present study we completed our work for synthesized copolyesters and terpolyesters which have been discussed in our previous work. The chemical and physical characterization has evaluated. Here we focused our interest in the electrical, mechanical with morphology investigation. The X-ray diffraction showed the amorphous state of polymers with some crystallinity in terpolyesters. Scanning Electron Microscopy (SEM) showed the formation of heterogeneous layers. The dielectric constant of copolyesters and terpolyesters with aliphatic or aromatic chains demonstrated the insulator characters. However, others with azo groups in the main chain demonstrated some electrical properties due to highly polarizability. The mechanical properties of copolyesters and terpolyesters were

investigated using ultrasonic waves and kinds of elastic moduli have been calculated. The copolyesters and terpolyesters with aliphatic main chain have more elasticity than others with aromatic chains.



**Figure 5.** SEM of selected copolyesters and terpolyesters at different magnifications.

## Conflicts of Interest

The authors declare no conflict of interest.

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## Graphical Abstract

The present study is accomplishment of our recent work for the synthesis of unsaturated co- and terpolyesters. The aim here is to investigate the electrical and mechanical properties. The dielectric constant of copolyesters and terpolyesters with aliphatic or aromatic chains demonstrated the insulator characters. The mechanical properties of copolyesters and terpolyesters were investigated using ultrasonic waves and kinds of elastic moduli have been calculated. The copolyesters and terpolyesters with aliphatic main chain have more elasticity than others with aromatic chains.

