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RESEARCH ARTICLE

Synthesis, Electro Conductivity, Thermal Stability and Antibacterial Activity of New Bis 1, 3, 4-Oxadiazole Copolymers

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Abstract

A series of bis oxadiazole copolymer were synthesized. The first series was synthesized by the reaction the resorcinol with ethylbromacetate to obtain diethyl 2,2'-[benzene-1,3-diylbis(oxy)]diacetate (1). Compound 1 was converted to their corresponding di hydrazide (2). This compound was reacted with oxalyl chloride in pyridine and N-Methyl-2-pyrrolidone (NMP) to obtained P1. This polymer was converted to P2 by cyclized it in the presence of polypesporic acid (PPA) as dehydrating agent to obtain P2 as bis oxadiazole copolymer. P3-P7 was synthesized by the reaction of compound 2 with five various di carboxylic acid in PPA as depicted in Scheme 1. These copolymers were characterized by IR and ^{1}H-NMR. The electro conductivity of synthesized copolymer were tested at 20, 20000 Hz at 25, 35, 45, 55, 65, 75, 85 and 95 ℃. Various positive and negative conductivity were recorded in most copolymers at different temperature. Thermal stability was studied for all copolymers. Copolymers 6 and 7 exhibited significant stability at 450 C. The antibacterial activity for these copolymers was tested against gram negative and gram positive bacteria. Copolymer P1, P2, P5, P6 and P7 showed antibacterial activity against all bacteria under test and P5 exhibited highest inhibition zone (23 mm) against *Shigella dysentery,* while P7 showed same inhibition against *Bacillus subtilis*. Furthermore, P7 exhibited inhibition zone equal 19 mm against *Esherichia Coli.*

Keyword: *Copolymer, Bis oxadiazole, Electro conductivity thermal.*

Introduction

In Generally, the 2,5-disubstituted-1,3,4-oxadiazole and 2-alkyl or aryl 1,3, 4-oxadiazole-5-thione and their thioalkyl derivatives besides to 2-amino-5-aryl or alkyl oxadiazoles displayed a various biological activities such as Anti-urease[1], Antibacterial [2], Anticancer[3, 4], Anti-fungal [5], Anti-allergic[6], Anti-inflammatory activity [7, 8] and antioxidant properties[9, 10]. 1, 3, 4-Oxadiazole derivatives are known as pharmacophores according to their metabolic profile and ability to engage in hydrogen bonding.

In particular , marketed antihypertensive agents such as tiodazosin[11, 12] and nesapidil [13]. Furthermore, it exhibited fascinating physical properties. For instance, electrochemical properties[14, 15], electro-optical properties[16] and luminescence properties[17, 18] and photophysical properties[19]. Poly oxadiazoles also, showed interested physical properties such light emitted properties as [20]. Aromatic poly (1, 3, 4-oxadiazole) has been utilized in several applications such as fibers with high technical, a system of aerospace engine, and gas separation membranes and fuel cells. As well as, polyoxadiazoles exhibits blue photoluminescence due to the existence of conjugated oxadiazole and p-phenylene rings in its backbone. It can be used for organic transistors, polymer light-emitting devices, and photovoltaic cell [21] as well as good hydrolytic stability, electroluminescence and high glass transition temperature[22],[23]. Due to these significant properties this work presented synthesis new bis -1, 3, 4-oxadiazole and investigated their electro conductivity in various temperature as well investigated their thermal stability.

Experimental

General

Chemicals and solvents utilized for synthesis

were purchased from Sigma-Aldrich, Fisherand besides to Merck. The Melting point was determined utilizing open capillary tube method by OMEGA MPS10 apparatus and it is uncorrected. The IR spectra were assigned by Perkin Elmer 400 Fourier Transform Infrared (FTIR) Spectrometer. The NMR spectra were recorded on Bruker AVN 300 MHz (AL-Bayt University, Jordan), using DMSO-d_6 as a solvent with TMS as internal standard, measurements.

The electro conductivity was screened by LCR meter at 50-3000 Hz. Differential Scanning Calorimetry (DSC) analysis was carried out using Perkin Elmer DSC, equipped with an internal cooler 2P-cooling accessory, and some of them were performed in Ibn –Al-Haitham College.

Synthesis of diethyl 2, 2'-[benzene-1, 3-diylbis (oxy)] diacetate

Bromoethyl acetate (10.02 g, 60 mmol) in 15 mL dry acetone was added dropwise to stirring solution of resorcinol (3.3 g, 30 mmol) in 25mL of dry aceton and 4.14 g, 30 mmol of unhydros putassium carbonate. The mixture was heated under reflux for 48 hrs. The solvent was removed under reduced pressure and then extracted three time with 25 mL ethyl acetate.

The combined organic layer was washed by solution of sodium hydroxide (5%) several time and moniterd by TLC uttilizing hexane: ethyl acetate (3:1). After disposal from the starting material and mono ester. The organic layer was washed by distilled water. The organic layer was dried under unhdrous sodium sulfate then evaporated under reduced pressure to afford pale yellow liquid. Yield 77%

Synthesis of 2,2'-[benzene-1,3-diylbis(oxy)]diacetohydrazide

Excess of hydrazine hydrate (80%) was added to warm solution of diethyl 2,2'-[benzene-1,3-diylbis(oxy)]diacetate(5.16 g, 18.2 mmol) in ethanol (25 mL). The mixture was heated under reflux for two hours. Upon cooling the precipitated filtrated and washed with cold ethanol then recrystallized from aqueous methanol to afford white precipitate Mp: 228 °C [lit 229 °C] Yield 81 %.

Synthesis of P1

Oxalyl chloride 0.22 g, 2 mmol was added drop wise through additional funnel within 20 mints to a solution of di hydrazide (0.5 g , 2 mmol) in dry pyridine and NMP (3: 2) at ambient temperature . After completion the addition the mixture was refluxed for 2 hrs at 60 °C. Upon cooling the mixture was poured into 100mL crashed ice and left to stirring for 1 hour. Hydrochloric acid (2%, 10 mL) was added to the mixture before evaporated under reduced pressure to afford pale brown amorphous.

Synthesis of P2

P1 (0.25 g) was added to stirring poly phosphoric acid at 150-160 °C. The mixture was left stirring under this temperature for 4 hours. The mixture was poured into 50 mL crashed ice the resulting product evaporated under reduced pressure to afford off white. Mp = up 300℃

General Synthesis bis 1, 3, 4-OxadiazolePolymerization

Dihydrazide **2** (0.5 g, 2 mmol) and 2 mmole of dicarboxylic acid was mixed and grinding to finny powder then poured into hot stirring liquid of poly phosphoric acid at 140-150 °C. the mixture lift under heating and stirring for 3 hrs. Upon cooling the mixture poured into 50 mL crashed ice. The PH of solution was neutralized by sodium hydroxide 10 %. The mixture was evaporated under reduced pressure to afforded the target polymer. The resulting copolymers were tabulated in Table 1.

Screening the Electrical Conductivity

The samples of copolymers under test were formed by pressing stabile weight from the pure copolymer to thickness 0.08 cm and diameter equal 1cm under 20 MPa. (Mega pascal) . The conductivity was screened by LCR meter at 50-3000 Hz at 25, 35, 45, 55, 65, 75, 85 and 95 ◦ C. The Electrical capacity and the Tangent of loss angle were also determined by Electrical Conductivity as shown below [24].

$C_p=\varepsilon_0 A/d$……(1)

$\varepsilon_r'=C_P/C_0 \longrightarrow \varepsilon_0 A/d/ C_0$ ….(2)

$\varepsilon_r''=\varepsilon_r' \tan(\delta) \longrightarrow \varepsilon_0 A/d/ C_0 . \tan(\delta)$….(3)

$\sigma_{a.c}=\omega \varepsilon_r'' \varepsilon_0$ ……(4)

C_0 =Capacity in vacuum in F

ω = Angular frequency…..2πf in f

σa.c=Alternating electrical conductivity in s/m

whereas
C_p = Capacity materials in F
A = Area…..πr^2 in m^2
d = Thickness in m

tan(δ) = Tangent of loss angle
ε_0 = Permittivity of vacuum $8.85*10^{-12}$ in F/m
$\varepsilon r'$ = Permittivity of the material in F/m
$\varepsilon r''$ = Relative permittivity in F/m

Result and Discussion

The **1** was synthesized by the reaction of resorcinol with bromoethyl acetate in the presence of potassium carbonate in acetone according to procedure that reported by Shakir, RM[25]. The melting point was compared with the literature. The FTIR spectrum displayed that no phenolic hydroxyl group band which is consist good evidence for success the reaction. Furthermore, the spectrum showed new band at 2983 and 2937 cm^{-1} attributed to aliphatic CH besides to band at 3080 cm^{-1} for the aromatic CH. The band of carbonyl ester was located at 1755 cm^{-1} and the C=C was located at 1603 and 1491 cm^{-1}. The band of ArOC was assigned at 1159 $cm^{-1.}$

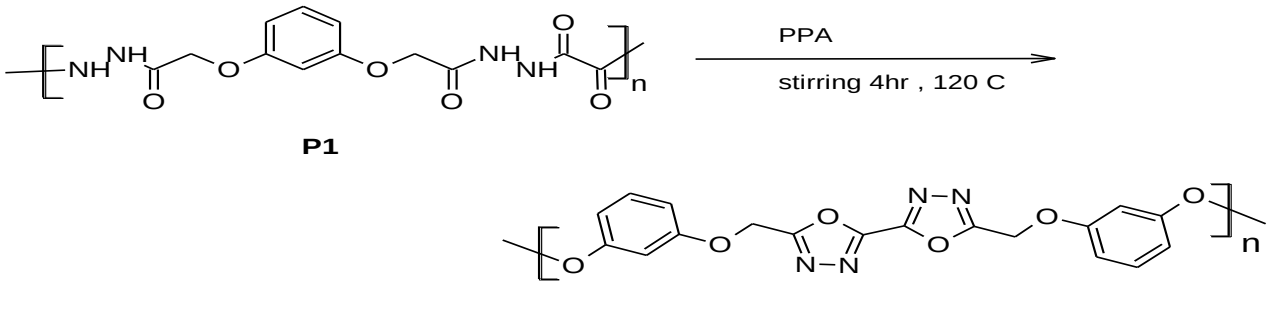
The ^{1}H-NMR spectrum shows disappearance the phenolic hydroxyl and it also shows existence of singlet peak at4.70 for OCH2CO with integral equal to for protons. The protons of two ethoxyl groups were located at 1.16 ppm as triplet peak for six protons and quartet peak at 4.07ppm for four protons. The shoulder in the triplet peak for the CH_3 group could be attributed to the geometrical isomers [26].

The aromatic protons were located as multiplet peak at 6.44 -6.55 for three protons (H_2, H4, H6). The proton of H_5 was appeared as triplet peak at 7.14 ppm. The CHN analysis of this compound showed that the practical percentage was in agreement with the theoretical percentage. 2, 2'-[benzene-1, 3-diylbis (oxy)] diacetate (**1**) was heated under reflux with excess of hydrazine hydrazide (80%) to obtain the bis acid hydrazide (**2**). The melting point of this compound was compared with literature[25] and characterized from their FTIR and ^{1}H-NMR spectrum. The FTIR spectrum exhibited three band at 3315, 3326 and 3205 cm^{-1}.

This attributed to NH_2 and NH. The CH aromatic and aliphatic bands were located at 3080, 2947 and 2912 cm^{-1} respectively. The ^{1}H-NMR spectrum exhibited disappearing of ten protons for two groups of OCH2CH3 and remaining two group of OCH2CO. Furthermore, the spectrum shows broad singlet band at 4.28 for two NH_2 as well the protons of two NH groups for the hydrazide was located at 9.26 ppm with integration for two protons.

The aromatic protons assigned as multiplet peak at 6.24, 6.56 ppm for H_2, H4, H6 and the H_5 was located as triplet peak at 7.13 ppm with coupling constant equal to 8.22 Hz. As well the practical percentage in CHN analysis was in agreement with the theoretical percentage. The copolymer (P1) was synthesized to be starting material for synthesized **P2**.

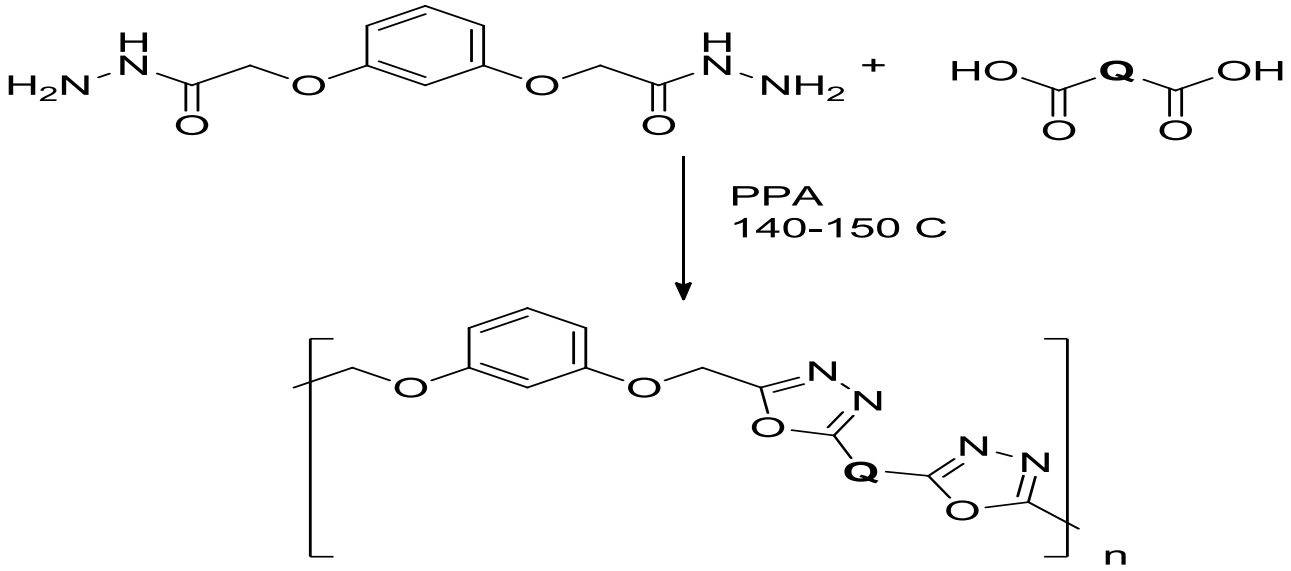
Reaction of di acid hydrazide (**2**) with oxalyl chloride in equal mille moles at ambient temperature and existence of pyridine as scavenger for chloride ion lead to afford **P1**. Furthermore, heating this reaction for two hours could increase the polymerization process. The product of copolymer was characterized by FTIR and ^{1}H-NMR.



P1 **P 2**

Scheme 1: Synthesis of P2

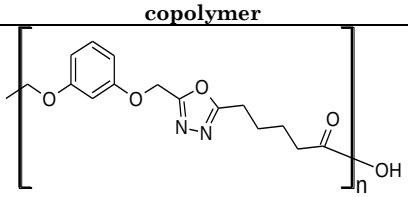
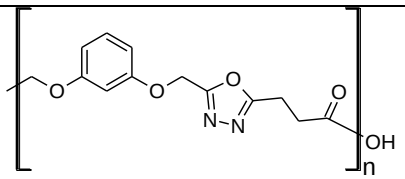
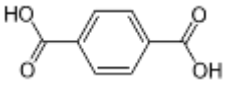
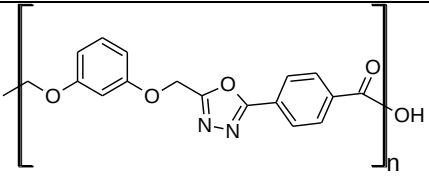
The FTIR spectrum of this copolymer exhibited two bands for NH at 3396 and 3234 cm^{-1}. It's clearly known that the linear polymer takes different shapes such as zikzak shape [27]. These differences in shape lead to differences absorption in FTIR spectrum due to existence different electronic environments[28]. Furthermore, the FTIR spectrum showed the medium band for CH aromatic at 3030 cm^{-1} and band at 295 4, 2918, 2850 for CH aliphatic. Appearance of these two peaks strongly than the monomer considered good evidence for increase the repetitions of this group and success the polymerizations.[29, 30]. The FTIR spectrum shows two carbonyl group one of them at 1705 cm^{-1} , which attributed to carboxylic carbonyl and it could be one end of the copolymer or the two end. This could be attributed to the dispersity of this copolymer [31]. The second carbonyl was assigned at 1662 cm^{-1}. The ^{1}H-NMR spectrum of **P1** showed two peaks for OCH2CO at 10.14,10.25 and 10.38 ppm and that was good evidence for success the polymerization and indicated existence of the repetitions[32, 33]. The NH as well, appeared as multi peaks at 7.17 ppm and small peak at 8.5 and 8.6 could be attributed to dispersity of copolymer or to end of copolymers. These bis-1,3,4-oxadiazole polymers were synthesized by reaction of the di acid hydrazide (**2**) with five different di acids in the presence of poly phosphoric acid as dehydration agent at 140-150 ℃.

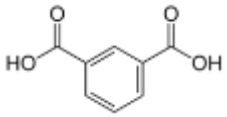
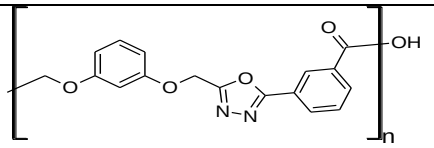
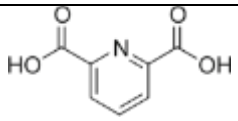
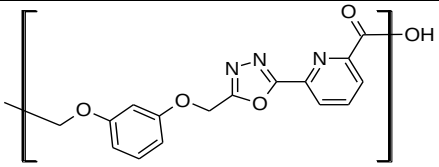


Scheme 2: Synthesis of cop3-cop7

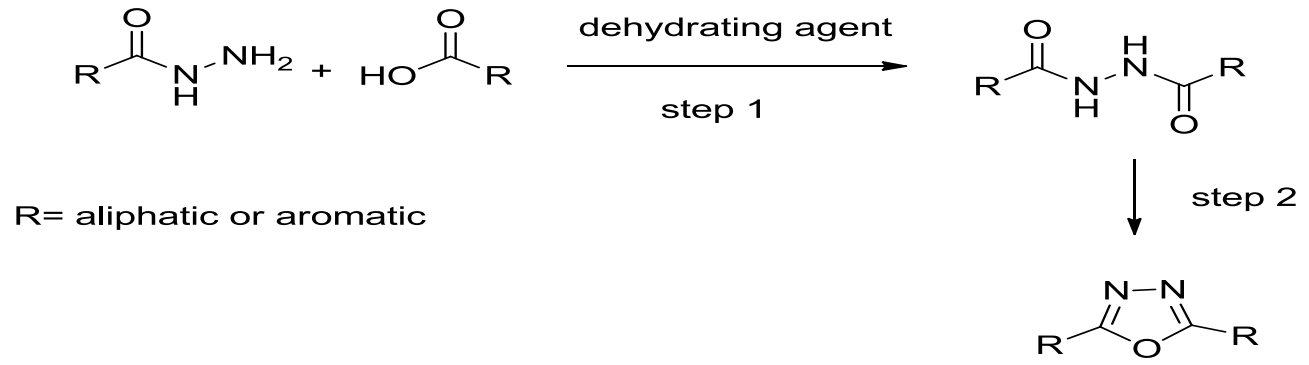
These polymers were identified by FTIR besides to ^{1}H and ^{13}C NMR for **P7**.

Table 1: **The diacids useful and its polymers**

No.	Di acid	copolymer
P3	$HOOC(CH_2)_4COOH$	
P4	$HOOC(CH_2)_2COOH$	
P5		

P6		
P7		

The FTIR spectrum for **P3** exhibited disappearance the bands of NH_2 and NH as well broad band was located at 3400 cm^{-1}, which attributed to one or two ends of the copolymer with COOH group. The band at 1689 cm^{-1} was referred to carboxylic carbonyl. The interested peak for formation 1, 3, 4-oxadiazole ring was located at 1612 cm^{-1} attributed to C=N. The same result was exhibited with **P4**, whereas the broad peak of carboxylic acid was located at 3433 cm^{-1} and the C=N of oxadiazole was assigned at 1606 cm^{-1}. The FTIR spectrum of **P5** shows broad band at 3435 cm^{-1} as well exhibited the aromatic and aliphatic CH. . The band of carboxylic carbonyl was located at 1695 cm^{-1} and the band of C=N was located at 1660 cm^{-1}. This high wave number for C=N could be attributed to the long range conjugated between the two oxadiazole rings and 1,4-phenyl group [34]. The band of C=C was located at 1597 cm^{-1}. FTIR spectrum of **P6** exhibited band at 3236 cm^{-1} and this attributed to CONHNHCO. The mechanism of reaction the acid hydrazide with carboxylic acid formed oxadiazole ring in the presence of dehydrating agent such as PPA, P_2O_5, $POCl_3$ and constricted H_2SO_4 clearly known it take place with two steps. The first one formation is aryl or acyl hydrazide, while the second step is cyclization the acyl the aryl or acyl hydrazide to oxadiazole [35-37] as demonstrated in Scheme 1.



Scheme 1: The mechanism of reaction the acid hydrazide with carboxylic acid formed oxadiazole rin

In other word, the NH could be existence in the structure of copolymer and some aryl hydrazide did not complete cyclization to oxadiazole ring or it could be existence disparity of copolymer. Furthermore, the spectrum exhibited the aromatic and aliphatic CH. On the other hand, assigned band at 1670 cm^{-1} for carbonyl harmonize with existence of NH. Furthermore, the peak at 1610 cm^{-1} confirms the formation of the 1, 3, 4-oxadiazole ring. The FTIR spectrum of **P7** shows broad band for NH as mention earlier and it in agreement with existence of small band at 1678 cm^{-1}. The bands aliphatic and aromatic CH was located at 3026, 2962 and 2848 cm^{-1} respectively. The band of C=N was located at 1610 cm^{-1} as well the C=C was located at 1543, 1493 cm^{-1} The ^{1}H-NMR spectrum of **P7** shows the OCH_2 as multiplet peak due to repetition of the monomer and to existence more than one possible system[32]. For instance, the OCH_2 in this copolymer can be attached with oxadiazole ring as well with carbonyl for non-cyclized in copolymer or in the dispersity. As well the two multiplet peak at aromatic rang harmonised with proposed structure. Furthermore, the peak at 9.3 ppm was in agreement with the FTIR which is referring to existence of NHCO. Besides to the FTIR and 1H-NMR this polymer was characterized by ^{13}C-NMR. The ^{13}C-NMR exhibited more than one peak at 66.25-66.81 ppm.

This peaks corresponding to repetition of OCH_2 which can attributed to the polymerization[38]. Furthermore, the multi peak at 144.07-144.45 ppm which attributed to quaternary aromatic carbon attached with OCH_2 express to the repetition of 1,3-phenyl group. The interesting area at 159.56-159.20 ppm and 167.49-166.89 ppm represented two C=N of 1, 3,4-oxadiazole and two C=N of 2,6-pyridine besides to CONH. These peaks can reflect the degree of polymerization [39, 40].

Electro Conductivity of P1-P7

The electro conductivity for **P1** was tested at 25, 35, 45, 55, 65, 75, 85 and 95°C. The electro conductivity at 25°C exhibited positive electro conductivity equal to 6.6×10^{-6} (s/m) at 827.2727 Hz as well it exhibited negative electro conductivity -3.6 × 10-7 (s/m) at 1029.091 Hz. The negative conductivity is known as negative resistance, which is express to an enhance in voltage across the device's terminals to outcome diminution in electric current through it [41].

This is in disparity into an regular resistor in which an enhance of applied voltage lead to a proportional increase in current due to Ohm's law, resulting in a positive resistance[42]. However, a positive resistance expends the power from current passing through it, while a negative resistance produces power[43]. With these conditions it can enhance the power of an electrical signal, amplifying it [44]. The Electro conductivity at 35-85 °C did not showed much differences comparing to 25 °C.

The electro conductivity for P1 at 95 °C displayed interesting conductivity at positive and at negative values. At the positive conductivity it revealed 1.45×10^{-6}, $2{,}05 \times 10^{-6}$ and 1.03×10^{-6} (s/m) at 423.63Hz ,1230.90 and 1432.72 Hz , as shown in Figure 1. The copolymer P2 showed increases of electro conductivity with increasing the temperature as well with increasing the frequency as shown in Figure 2. The highest conductivity was at 95°C with linear increases.

The copolymer P3 exhibited highest electro conductivity at 55° C, which decreasing slightly with increases the frequency. The conductivity at 65 °C also showed interesting conductivity and linear increasing with increasing frequency as demonstrated in Figure 3. However, the conductivity of P3 at 95 and 75 °C was too closed together. This result indicated that the conductivity of this copolymer depend on certain temperature to obtain the highest resistance as depicted in Figure 4. The resistance of P4 at 45, 65 and 75° C possess nearly similar electro conductivity attitude as depicted in Figure 5.

Furthermore, it exhibited interesting electro conductivity at 85 °C, whereas exhibited positive conductivity at 221.8 Hz the decreases to negative resistance at 423.6 Hz. The next maximum increasing with increases the frequency was located at 625.45 Hz. The second interesting electro conductivity was located at 827.27 Hz to obtain negative value then advanced to positive value at 2441.8 Hz then stabilized until near to 5000 Hz, as demonstrated in Figure 6. Same sequence attitude was appeared at 95° C as depicted in Figure 1.

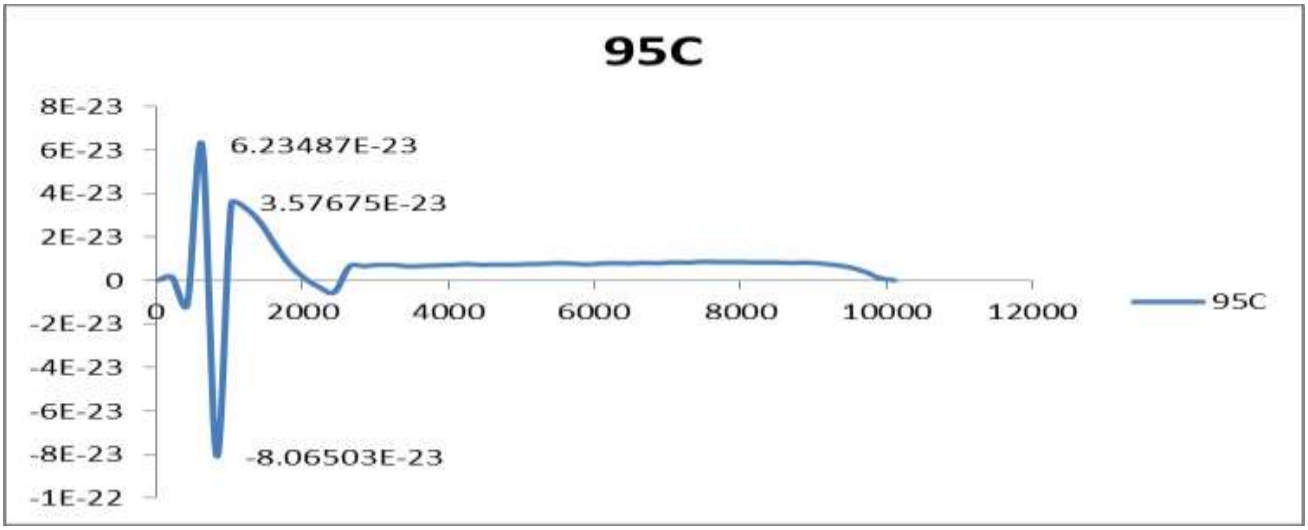


Figure 1: Electro conductivity of P4at 95° C

The overall conductivity at all temperatures is shown in Figure 2.

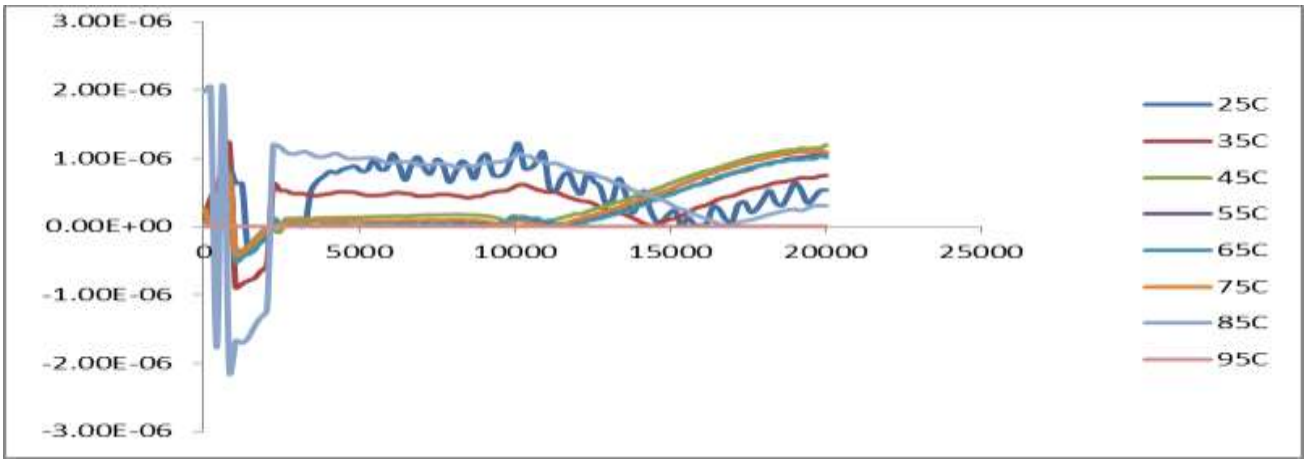


Figure 2: Electro conductivity of P4at all temperature

The conductivity of P5 at 45, 75, 85 and 95 °C respectively exhibited linear increasing with increasing the frequency as depicted in Figure 3.

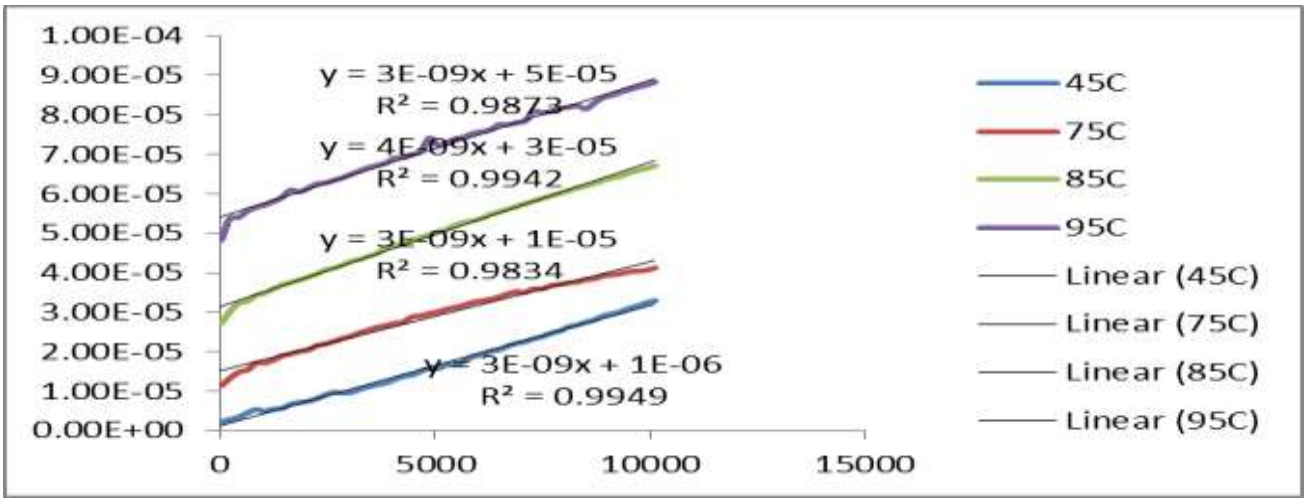


Figure 3: Electro conductivity of P5at 45, 75, 85, and 95 °C

Even though, the conductivity at 65° C was linear, the conductivity at this temperature was less than the conductivity at 45° C. The P5 showed lowest conductivity at 25 than 35 and 55°C respectively as demonstrated in Figure 4.

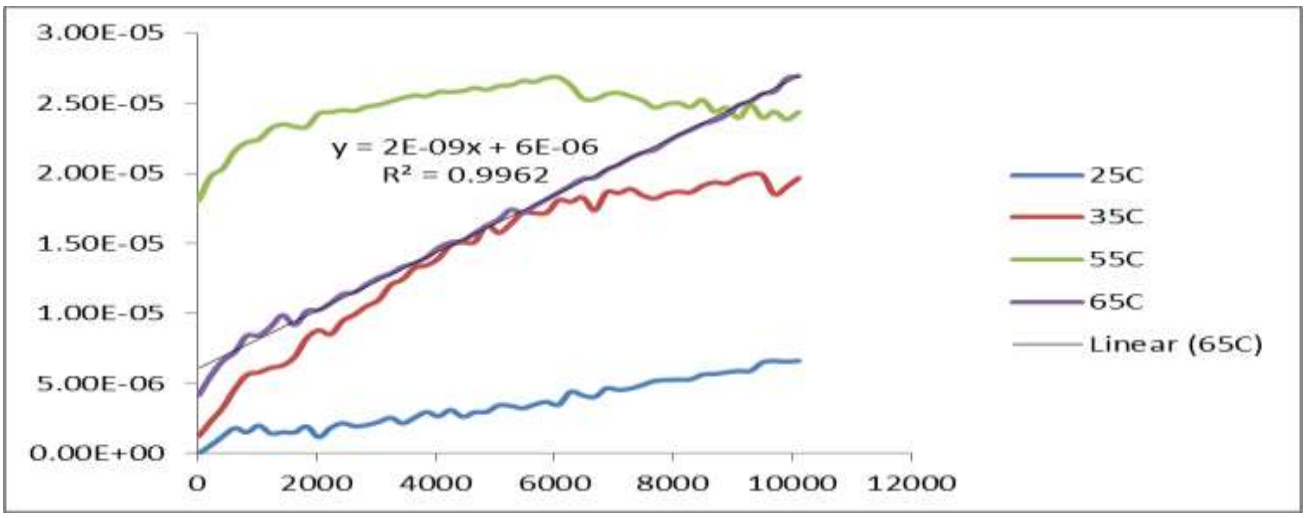


Figure 4: Electro conductivity of P5at 25, 35, 55, and 65° C

Copolymer P6 exhibited highest conductivity at 75°C. The attitude of the conductivity was linear direct proportion. Same attitude was located at 85 °C and the other temperatures as depicted in Figure 5. Furthermore, the conductivity at 85 °C was high compared to 95 °C.

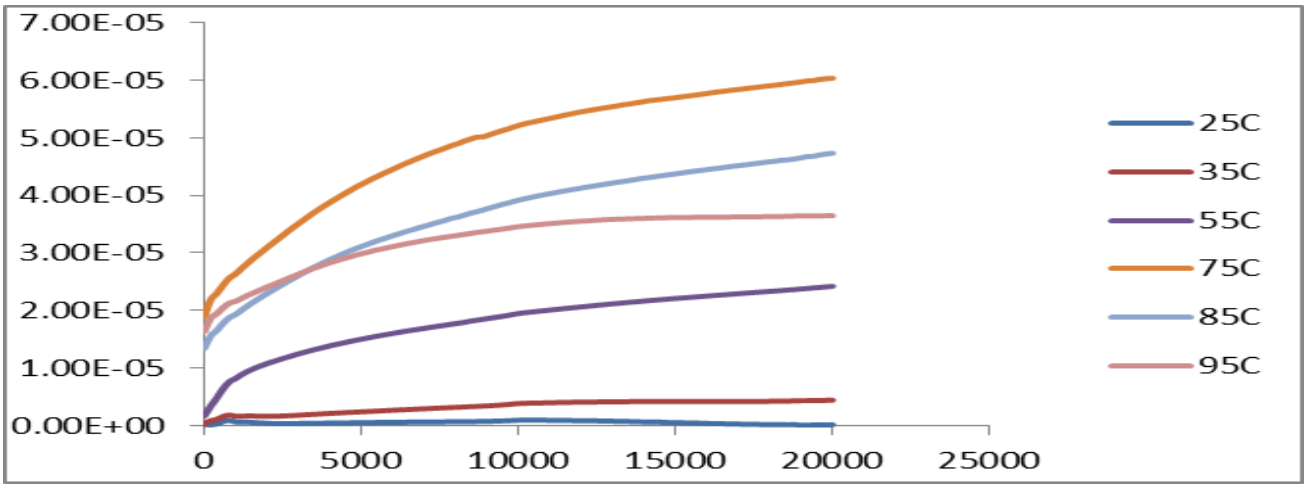


Figure 5: Electro conductivity of P6 at 25, 35, 55, 75, 85, and 95 °C

Unique attitude was located at 45 °C. The electro conductivity of P6 enhances with increase the frequency and showed linear attitude until 15000 Hz, Figure 6.

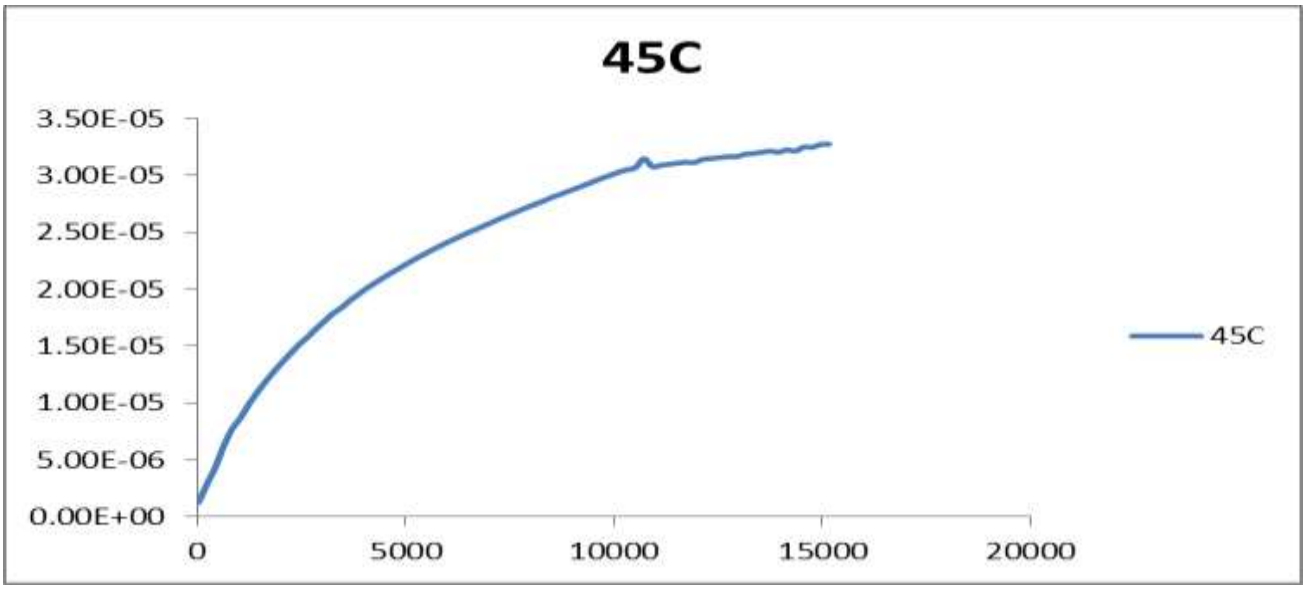


Figure 6: Electro conductivity of P6at 65 °C until 15000Hz

After that, frequency the conductivity decline to minimal value at 1798.83 Hz then raised to maximum conductivity at 18183.65 Hz, as displayed in Figure 7.

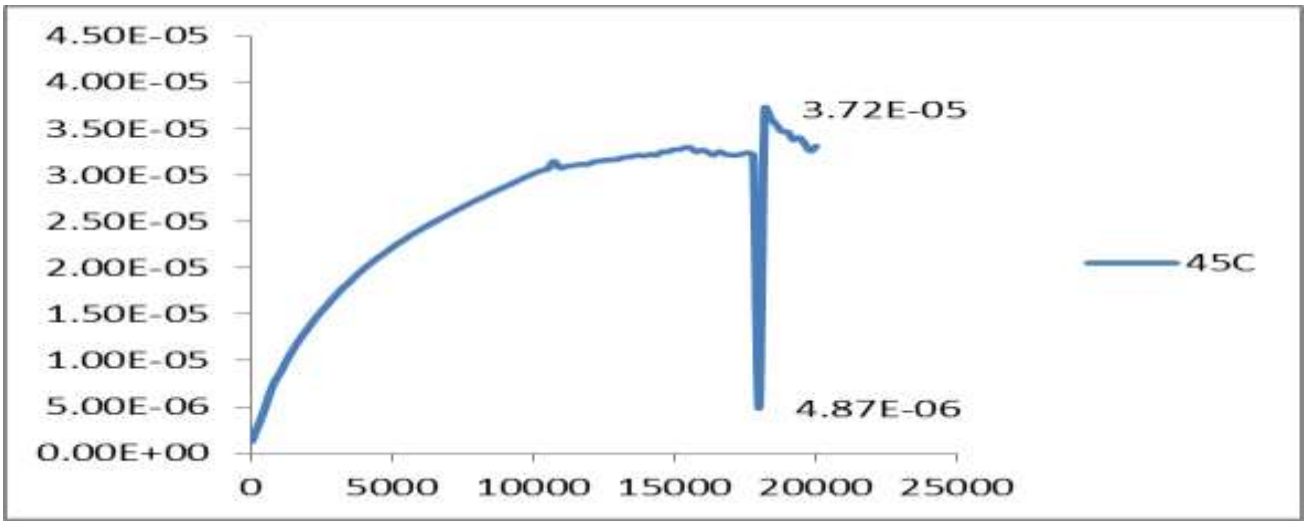


Figure 7: Electro conductivity of P6at 65 °C, 20 to 20000Hz

The electro conductivity of P7 showed various electro conductivity behaviours at different temperatures. The highest conductivity assigned at 25 °C then at 35 °C.

Furthermore, this copolymer recorded negative conductivity at 1230.9 Hz with 95°C, as shown in Figure 8.

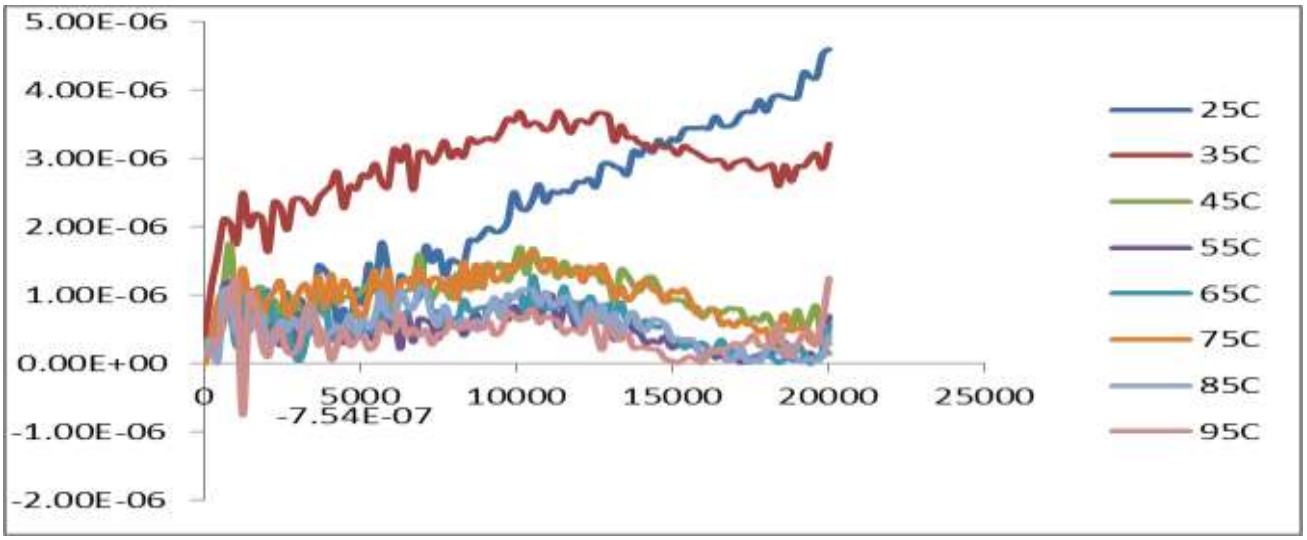


Figure 8: Electro conductivity of P7 at all temperature

Thermal Stability of the Synthesised Polymers

The thermo gravimetric analysis (Tga) of the synthesized copolymers (**P1-P7**) was exhibited by diverse thermal stability at ambient temperature to 450 °C. Using of argon with heating rate 5 °C per minute. Most copolymer did not exhibited significant thermos stability accept **P6** and **P7** which were exhibited by significant thermos stability. The thermo gram of both copolymers demonstrated resistance to thermal degradation to 450 °C as depicted in Figure 9 and Figure 10.

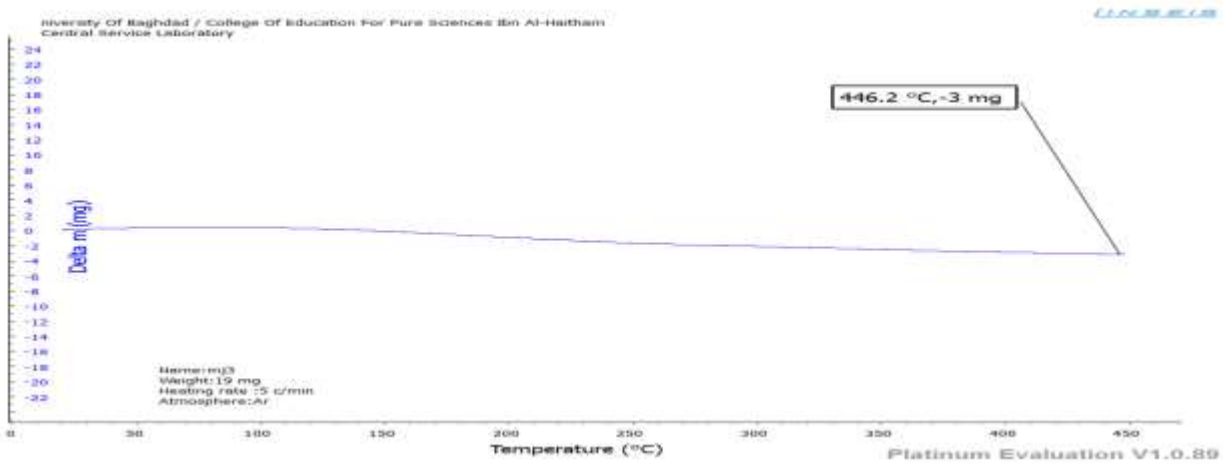


Figure 9: Tga of P6

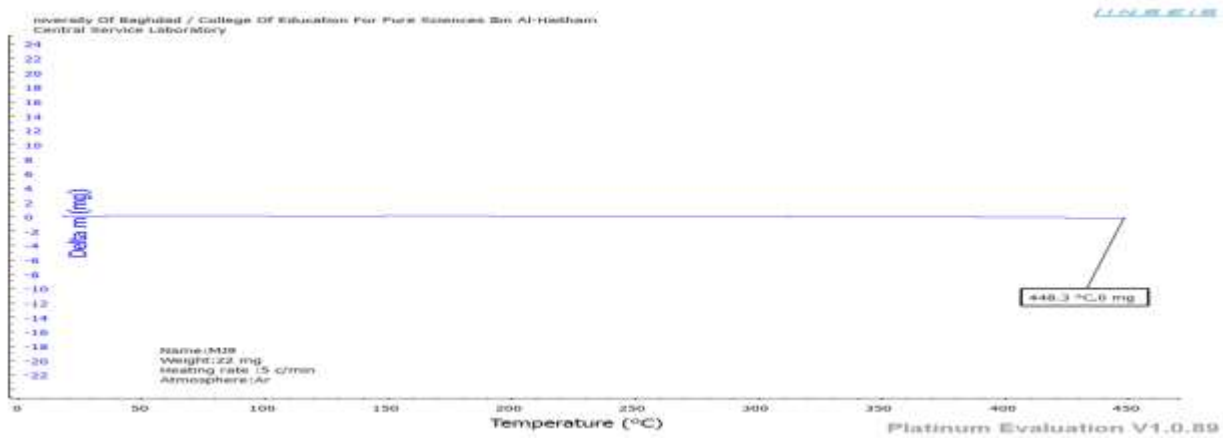


Figure 10: Tga of P7

Furthermore, the differential scanning calorimeter (DSC) for these copolymers (P6 & P7) showed exothermic peaks 180.9-202.3 °C and 73.2-98.9 °C respectively for P6. Copolymers 7 exhibited their exothermic peak at 302.3-324.3 °C and 101.2-116.8 °C respectively, Figures 11 & 12.

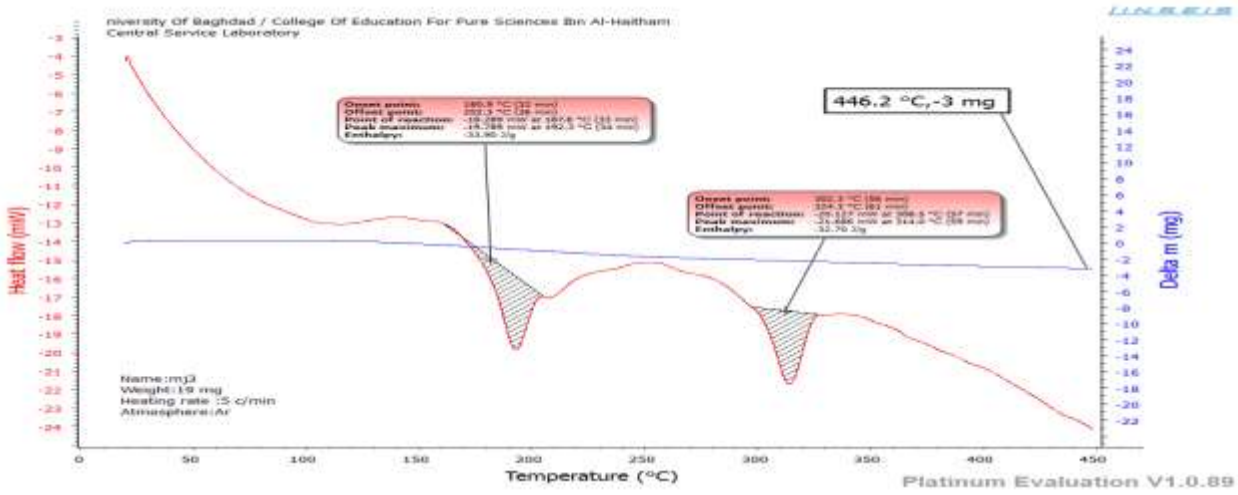


Figure 11: DSC of P6

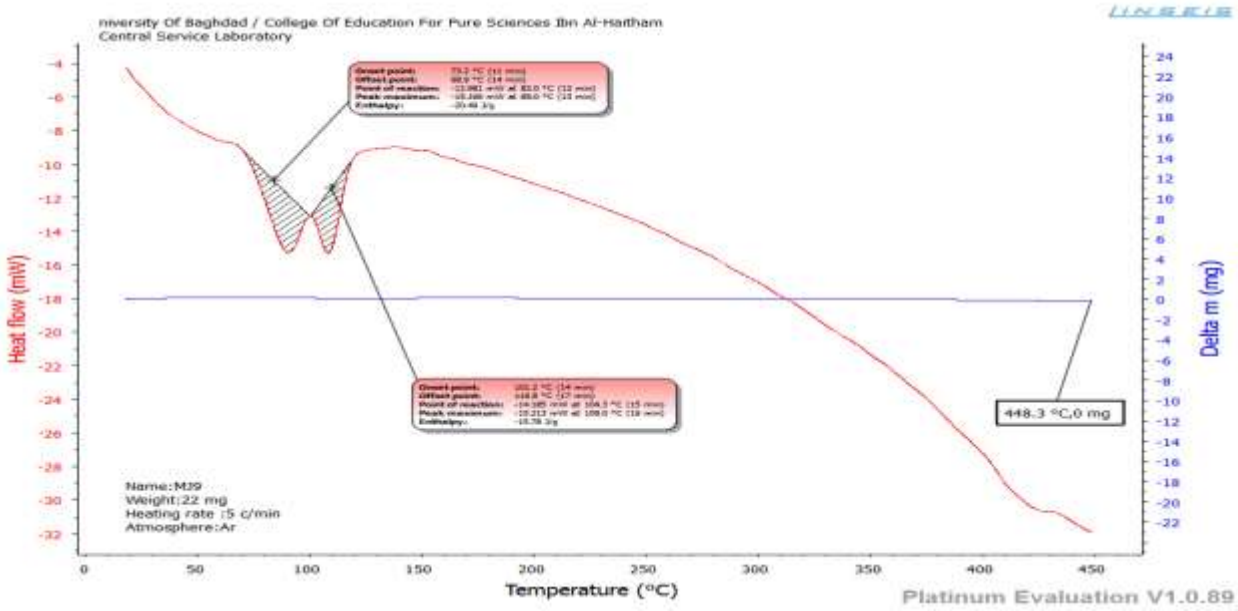


Figure 12: DSC of P7

Antibacterial Activity of the Synthesised Polymers

The antibacterial activity of the synthesized copolymer was screened by the a gar diffusion method. *Serratia marcescens (-), Pseudo monas aerogenosa(-) , Bacillus(+) , and Entero bacter Cloacae* as gram- and gram + pathological bacteria have been used in this test.

The samples were remedied by prepared a gar on a petri dishes after autoclaving it for 15 min at 121 °C. The surface of the gar was vaccinated uniformly. the microorganisms, bacteria were activated in a nutrient growth medium at 37 °C for 24 her. microorganisms , the above bacteria were activated in a nutrient growth medium at 37°C for 24 hrs. Dimethyl sulfoxide was used as a solvent as well as controller. Three concentrations 0.01, 0.05 and 0.025 M of copolymer under test have been utilized millimeter to measure the inhibition zone.

The best results were recorded at 0.01 M. P1, P2, P5, P6 and P7 showed antibacterial activity against all bacteria as displayed in Table 2. P5 exhibited highest inhibition zone (23 mm) against *Shigella dysentery,* while P7 showed same inhibition against *Bacillus subtilis*. Furthermore, P7 exhibited inhibition zone equal 19 mm against *Esherichia Coli.*

Table 2: Inhibition zone for P1-P13 at 0.01M

No.	*Bacillus subtilis*	*Shigella dysenteria*	*Klebsiella pneumoniae*	*Staphylococcus aureus*	*Esherichia Coli*	*Bacillus subtilis*
P1	**15**	**13**	**14**	**15**	**14**	**15**
P2	**15**	**16**	**-**	**14**	**14**	**15**
P3	**-**	**-**	**-**	**-**	**14**	**-**
P4	**-**	**-**	**13**	**-**	**14**	**-**
P5	**16**	**23**	**14**	**15**	**14**	**16**
P6	**13**	**14**	**12**	**13**	**14**	**13**
P7	**23**	**14**	**14**	**13**	**19**	**23**

Conclusion

A series of bis 1, 3, 4-oxadiazole copolymers were synthesized. These copolymers show diverse electro conductivity at different temperature. Copolymers 6 and 7 exhibited significant thermal stability at 450 C. Copolymer P1, P2, P5, P6 and P7 showed antibacterial activity against all bacteria under test and P5 exhibited highest inhibition zone (23 mm) against *Shigella dysentery,* while P7 showed same inhibition against *Bacillus subtilis*. Furthermore, P7 exhibited inhibition zone equal 19 mm against *Esherichia Coli.*

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