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Blending a New Polymers Containing Oxadiazole Ring with PVA: Synthesis , Thermal Analysis and Biological Activity

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Abstract

This study focused on the synthesis of novel polymers incorporating the 1,3,4-oxadiazole ring. Four polymers were specifically prepared by blending polymers (6-9) with polyvinyl alcohol (PVA) in defined ratios, resulting in the formation of blended polymers (10-13). The synthesized polymers were characterized using Fourier Transform Infrared (FTIR) spectroscopy and proton nuclear magnetic resonance (^{1}H-NMR). The results showed that the structure aligned with the proposed synthetic polymers. Furthermore, the physical and thermal properties were studied using scanning electron microscopy (SEM), thermogravimetric analysis (TGA) and Differential Scanning Calorimetry (DSC). Additionally, the biological activity was examined against two strains of pathogenic bacteria, Gram-negative (*E. Coli*) and Gram-positive (*S. aureus*). The findings demonstrated greater activity than Amoxicillin as a standard antibiotic.

Keywords: Polymer blends, biological activity, heterocyclic polymers ,1,3,4-oxadiazoles, thermal analysis.

مزج بوليمرات جديدة تحتوي على حلقة أوكسادیازول مع بولي فينيل الكحول: تحضير, تحليل حراري ونشاط بايولوجي

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الخلاصة

ركزت هذه الدراسة على تحضير بوليمرات جديده تحتوي على حلقه 4,3,1- اوكساديازول تم تحضير اربع بوليمرات بشكل خاص عن طريق مزج البوليمرات (6-9) مع بولي فنايل الكحول *(PVA)* بنسب محددة مما ادى الى تكوين مزيج البوليمرات (10-13) تم تشخيص البوليمرات المحضرة باستخدام التحليل الطيفي للأشعة تحت الحمراء *(FTIR)* والرنين المغناطيسي النووي البروتوني *(^{1}H-NMR)*. اظهرت النتائج تطابق التراكيب المقترحة للبوليمرات المحضرة. علاوة على ذلك ، تمت دراسة الخواص الفيزيائية والحرارية باستخدام المجهر الالكتروني الماسح (*SEM*) و التحليل الحراري الوزني (*TGA*) و المسعر الحراري التفاضلي

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(**DSC**) لها. بالإضافة إلى ذلك، تم فحص النشاط البيولوجي ضد سلالتين من البكتيريا المسببة للأمراض،
سلبية الجرام (**E. Coli**) وإيجابية الجرام (**S. aureus**). أظهرت النتائج نشاطًا أكبر من الأموكسيسيلين
كمضاد حيوي قياسي.

1. Introduction

In materials science, a polymer mixture or blend is analogous to metal alloys, where a minimum of two polymers are mixed to produce a new material having distinct physical properties [1]. These blends have gained prominence across various technical fields due to their enhanced superior thermal and mechanical properties [2,3]. The differences in these properties are contingent upon the blend rations and compositions [4,5]. As a synthetic, biodegradable thermoplastic polymer with superior thermal and mechanical properties, polyvinyl alcohol (PVA) is non-toxic and finds use in a variety of industries, including medical, packaging, resins, and building [6]. Polymer blending is a highly effective technique for enhancing or changing the physicochemical characteristics of polymer materials [7]. Numerous studies have utilized various naturally derived fillers, including chitosan, starch, cotton fibers, and microcrystalline cellulose (MCC), to enhance the mechanical properties of PVA. [8-10]. Professionals are aware that a class of heterocyclic polymers which are both thermally and chemically stable are aromatic polymers containing (1,3,4-oxadiazole) units [11-16]. Furthermore, some literature-based research has indicated that a novel class of 1,3,4-oxadiazole polymers exhibits strong biological activity [17-20]. In this study, a new polymers containing a (1,3,4-oxadiazole) ring was synthesized and subsequently mixed with PVA. The thermal properties and biological activity of the generated polymer were studied.

MATERIALS AND METHODS

Materials

Isophthalic acid, 4-amino benzoic acid, $POCl_3$, HCl, thionyl chloride and benzene were supplied from BDH. While, sodium bicarbonate, hydrazine hydrate, sodium nitrite, Polyvinyl alcohol were supplied from Sigma. CDH provided absolute methanol, sulfuric acid, absolute ethanol and diethyl ether.

Instrumentation

FTIR spectra were obtained using a Shimadzu FTIR 8400s with KBr disks in the 400-4000 cm^{-1} range.The ^{1}H-NMR spectra were recorded on Bruker 400 MHz instrument using DMSO-d6 as a solvent and TMS as an internal reference. Measurements were conducted at Central lab, University of Tahran (Iran).

Methods

Preparation of the ester of Isophthalic acid (1)

A mixture of Isophthalic acid (40.846 gm, 0.246 mol) in absolute methanol (200 mL) and sulfuric acid (5.4 mL) was refluxed for 6 hrs. After cooling, the mixture was washed with sodium bicarbonate solution, followed by multiple washes with water. It was then dried, and recrystallized from ethanol. The color of the precipitate white ; yield 80% ; m.p. = 69 ^{0}C. [21]

Preparation the hydrazide of Isophthalic acid (2)

A mixture of ester compound (1)(0.006 mol) and 3 mL of 80% hydrazine hydrate in 5 mL of absolute ethanol was refluxed for 3 hrs. After cooling to room temperature, the solvent was evaporated, and the form solid was recrystallized from diethyl ether. The precipitate was off white; yielding 92%, m.p. = 220 ^{0}C [21].

General Method for Synthesis of Substituted1,3,4-Oxadiazoles (3)

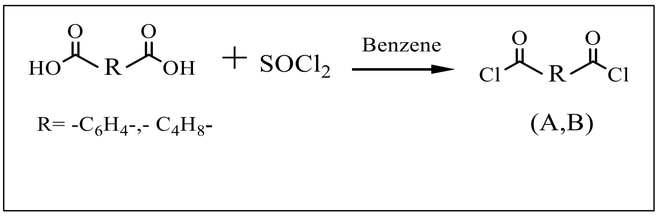
After refluxing for four hours, phosphorus oxychloride (5mL), (5 mmol) of acid hydrazide (2), and 4-amino benzoic acid (10 mmol) were combined and allowed to cool to room temperature. Following the addition of ice and an overnight stirring, the reaction mixture was neutralized with saturated sodium bicarbonate solution. The resulting precipitate was filtered, dried, and crystallized from ethanol to yield the corresponding oxadiazole as a brown precipitate. The yield was 76%, mp.: (180-182) ºC; and FT-IR analysis showed the following peaks: ν (cm^{-1}): (3425-3387) (NH, NH_2), 3055 (C-H aromatic.), 1639 (C=N endocyclic), 1600 (C=C aromatic).

Synthesis of azo compounds (4,5)

In 8 milliliters of 50% HCl, 4,4'-(5,5'-(1,3-phenyl ene) bis(1,3,4-oxadiazole-5,2-diyl)) dianiline (3) was dissolved to form diazonium salt. Subsequently, a solution of sodium nitrite (2.1g, 0.03mol) in 10 mL of water was cooled to a temperature between 0 and 5 ºC. This cooled solution was then missed with a of 0.03 mol solution of phenol or 2-nitrophenol in 25 mL of 10% NaOH. After the mixture was allowed to cool for two hours, hydrochloric acid was added to acidify it. After filtering, the resulting precipitate was cleaned with cold water [23]. This process yielded a brown-red precipitate from compound (4) m.p.: (211-213) ºC; yield (87%) and a yellow precipitate from compound (5) m.p.: (140-142) ºC; yield (90%) FT-IR(KBr), ν (cm^{-1}): (3491,3425) OH, 3057 cm^{-1}(C-H aromatic.), 1630 (C=N endocyclic), 1595 (C=C aromatic), 1419 (N=N azo), ^{1}H-NMR (DMSO-d6) δ(ppm): 8.49-8.50 (m,6H, Ar-NO_2), 8.12-8.16)d.d,8H, Ar-N=N), 7.60-7.64(m,4H,Ar-H) 5.95 (S,2H, H-phenol).

Preparation of acid dichloride (A and B)

0.01 mol of Isophthalic acid or Adipic acid was dissolved in dry benzene 15 mL with an excess of $SOCl_2$. The solution was then refluxed for 8 hours. The excess thionyl chloride and benzene were remove under vacuum after cooling where Isophthalic dichloride (A) precipitate as off-white color; yield (95%), m.p.= (177-179) ºC. So, Adipic dichloride (B), as a white precipitate, yields 93%, m.p. = (184-186) ºC [24].



Equation (1): Synthesis of compounds (A and B)

Synthesis of polymers (6-9)

In 15 milliliters of DMF, compound (4) or (5) (0.01 mol) has been dissolved with a few drops of dry pyridine. After cooling the mixture, one of the compounds (A) or (B) (0.01 mol) was added dropwise while stirring for a full day. Following this, the mixture was poured into 100 milliliters of ice water together with 5 milliliters of concentrated hydrochloric acid. The resulting precipitate was dried and filtered [25]. Polymer (6) was obtained as a yellow color; yield (93%), m.p.= (270-272) ºC. FT-IR analysis showed the following peaks: ν (cm^{-1}): 3051 cm^{-1}(C-H aromatic.),1651 (C=N endocyclic), 1581 (C=C aromatic), 1419 (N=N azo),1710(C=O ester), polymer (7) as off-white precipitate, yield (77%), m.p.= (257-259) ºC. FT-IR(KBr), ν (cm^{-1}): 3051 cm^{-1}(C-H aromatic.), 1651 (C=N endocyclic), 1581 (C=C aromatic), 1419 (N=N azo),1708 (C=Oester), ^{1}H-NMR (DMSO-d6) δ(ppm): 8.43-8.50

(m,8H, Ar-N=N), 8.15-8.19 (m,4H, Ar-H), 7.62-7.66 (m,8H, O-Ar-N=N), 2.75-3.02 (t,4H, CH_2CO), 2.03 -2. 10(t,4H, CH_2-CH_2).

Polymer (8) was obtained as brown precipitate, yield (89%), m.p.= (198-200) ºC. FT-IR analysis showed the following peaks: ν (cm^{-1}): 3062cm^{-1} (C-H aromatic.), 1651 (C=N endocyclic), 1581 (C=C aromatic), 1419 (N=N azo), 1315(NO_2), 1700(C=O ester), polymer(9) as brown precipitate, yield (85%), m.p.= (208-210) ºC. FT-IR(KBr), ν (cm^{-1}): 3055 cm^{-1}(C-H aromatic.), 1651 (C=N endocyclic), 1588 (C=C aromatic), 1419 (N=N azo),1315(NO_2), 1701(C=O ester) ^{1}H-NMR (DMSO-d6) δ(ppm): 8.50-8.64 (m,8H, Ar-NO_2), 7.74-7.82 (d.d,8H, Ar-N=N), 7.53-7.55 (m,8H, Ar-H), 2. 52-3.37 (t,4H, CH_2CO), 1.25 -2.10 (t,4H, CH_2-CH_2).

Synthesis of blend polymers (10-13)

Polymer blends were created by blending polymers (6-9) with PVA in a 1:1 ratio using the solvent casting method. To prepare 5 wt% polymer solutions, PVA was dissolved in hot water and after that mixed with one of the created polymer solutions (6-9). A homogeneous solution was created utilizing a hot plate stirrer for 60 minutes at room temperature after the two polymer solutions had been mixed together [26].

The study of biological activity:

In studies utilizing the agar disc diffusion method, the antibacterial activity of novel blend polymers (10-13) was assessed against two types of pathogenic bacteria: *(G+) (S. aureus) and (G-) (E. Coli)*. DMSO was used as the solvent, and the tests were conducted at a concentration of (100 mg/ml). Agar and potato dextrose agar medium have been used to culture the bacteria, and these plates were incubated for a period of 24 hrs at a temperature of 37 ºC [27-31].

Result and discussions

The scientific route was summarized by Scheme (1), FT-IR Spectrum of this compound shows the stretching vibration band of N-H for NH_2 and NH moiety at (3425-3387) $cm^{-1,}$ so the absorption band at 3055 cm^{-1} attributed to C-H aromatic, in addition, the stretching vibration (ν) band of C=N endocyclic at 1639 cm^{-1} and showed the absorption band of C=C aromatic at 1600 cm^{-1} [32,33].

The azo compounds (4,5) were synthesized by reacting compound (3) with phenol or substituted phenol, resulting in high yields of compounds (4,5). FT-IR spectrum of these monomers displayed abroad band of OH group at (3491,3425) cm^{-1} respectively. The absence of a stretching vibration band of N-H and the appearance of the absorption band of the N=N group is good evidence of the synthesis of these compounds. In addition, the appearance of a single signal in the ^{1}H-NMR spectrum of compound (5) at 5.95 ppm attributed to the proton OH group is a good indication of the matching proposal structure of these monomers. So, the FT-IR spectrum of the polymers (7,9) shows the absence of an absorption band from the OH group, and the ^{1}H-NMR spectrum of these polymers shows the disappearance of the signal for OH proton. These results refer to the success of the synthesis of polymers [34]. In this study, new polymers (6-9) were prepared and blended with PVA to investigate blend polymer properties. The properties regarding the acquired blends showed a good miscibility level between the polymer and PVA (6–9), as evidenced by the blend polymer's FT-IR results, which showed band broadening in the 3600–2400 cm^{-1} range because of strong intermolecular hydrogen bonding between the (OH) of the PVA and (O) of prepared polymers [35].

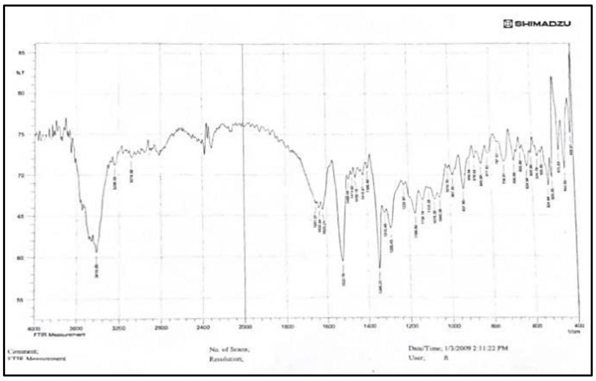


Figure 1 :FTIR of compound (5)

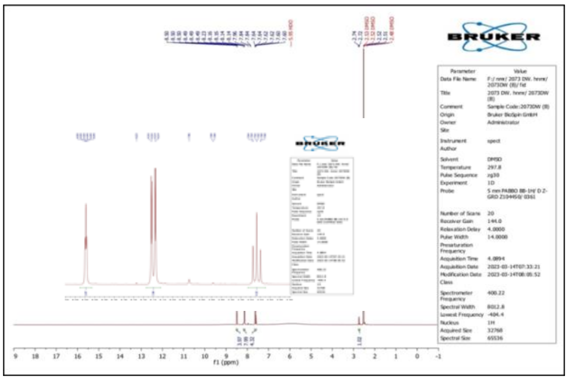


Figure 2 :^{1}H-NMR of compound (5)

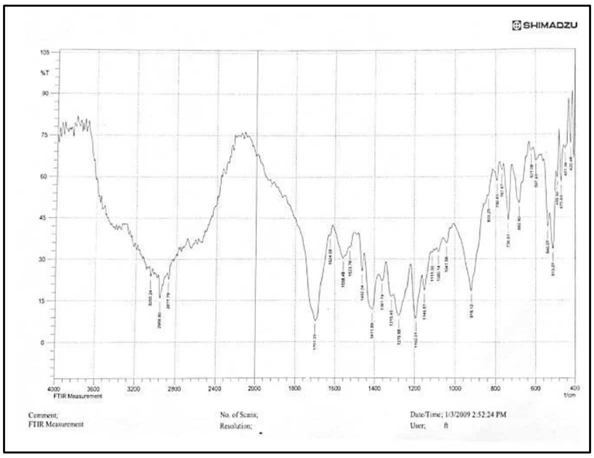


Figure 3 :FTIR of compound (7)

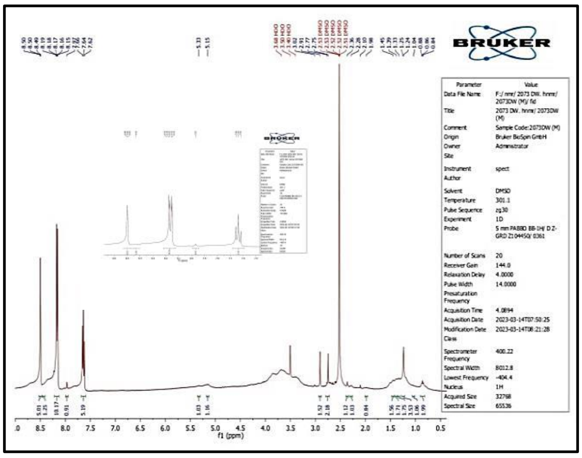


Figure 4 :^{1}H-NMR of compound (7)

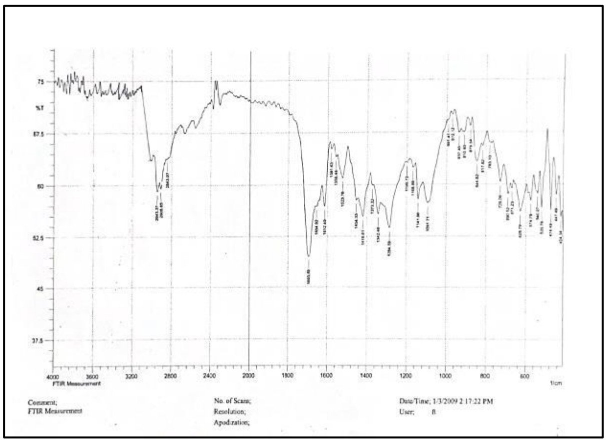


Figure 5 : FTIR of compound (9)

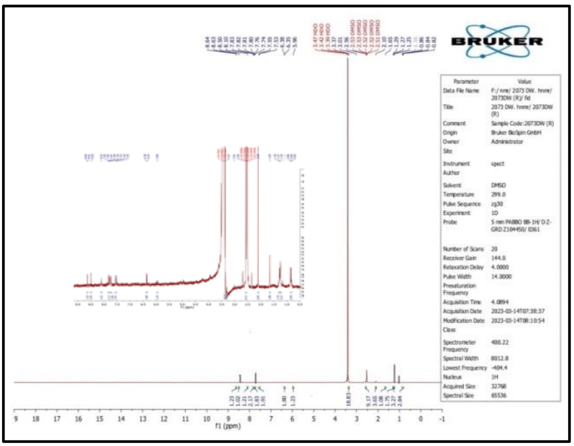
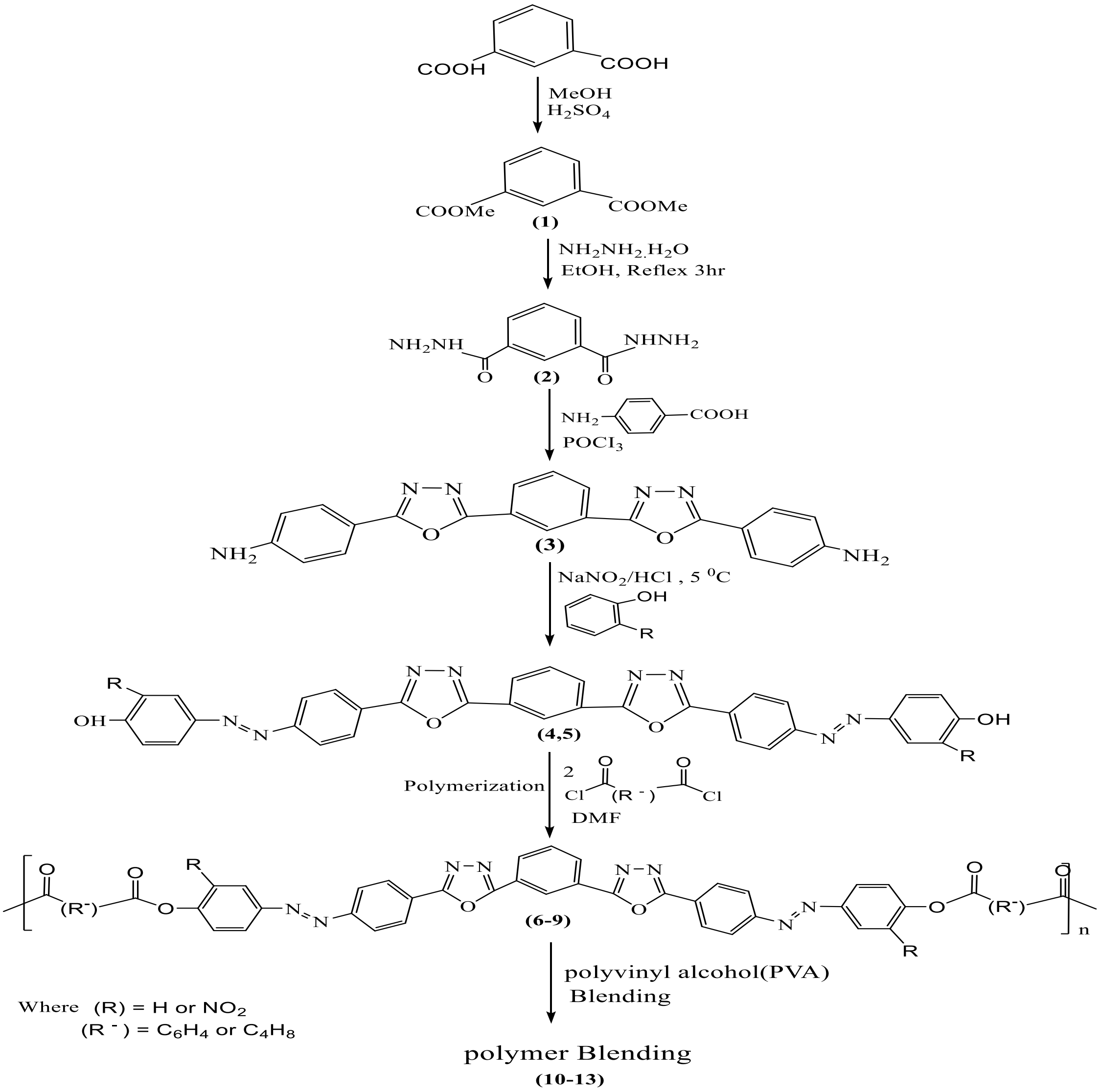


Figure 6 :^{1}H-NMR of compound (9)



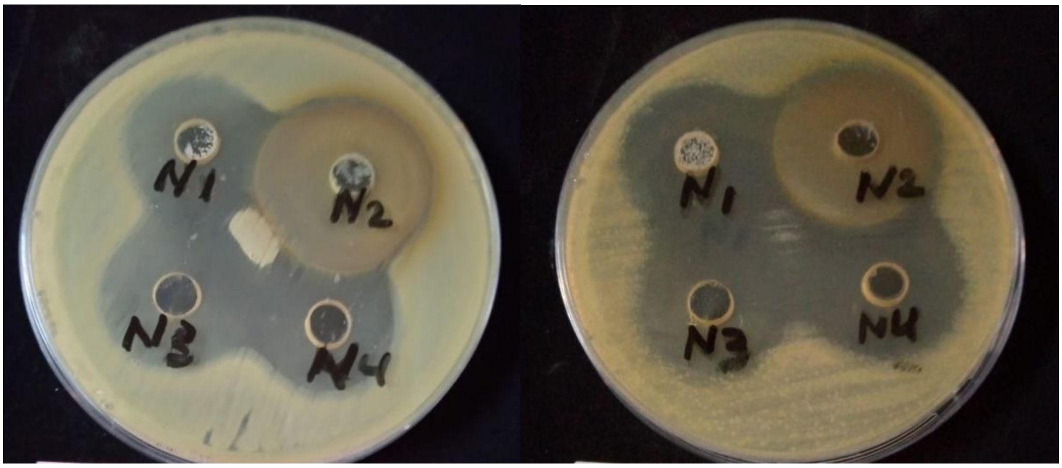
Scheme 1: The synthetic route of the synthesis compounds

Biological activity:

The traditional antibacterial drug Amoxicillin is used in this study to compare its activity with that of the novel blend polymers (10 -13). Table [1] provides a summary of the polymer test results. As seen in Fig 7, all of the polymers display high activity against Gram-positive bacteria *(S. aureus)* and extremely high activity against Gram-negative bacteria *(E. coli)*. This effectiveness is attributed to the presence of the 1,3,4-oxadiazole ring, known for its potent antimicrobial properties, as well as polyvinyl alcohol, which is recognized as green polymer with antimicrobial activity [36].

Table 1: Antimicrobial evaluation of blend polymers (10-13).

Synthesized Compounds	Inhibition zone (mm) at 100 mg/ ml	
	Gram-positive Staphylococcus aureus	*Gram-negative Escherichia Coli*
N_1[10]	31	32
N_2[11]	34	33
N_3[12]	33	30
N_4[13]	32	30
Amoxicillin	23	17



Staphylococcus aureus ***(E. coli)***

Figure7: Antibacterial activity of blend polymers (10-13)

Thermal analysis:

Thermogravimetric (DSC/TG) curves were generated for pure PVA and new blending polymers that blended PVA with oxadiazole ring at a heating rate of 10 °C min^{-1} under argon. The temperature range measured was from 0 °C to 600 °C. Table displays the thermal decomposition of blended PVA. Figure (8) depicts the TGA thermogram for compound (10), which reveals three stages of decomposition: the first stage occurs between 50 and 200°C and causes weight losses of approximately 39.94% because the evaporation of volatile compounds, mainly water, the second stage occurs between 200 and 550°C and causes weight losses of approximately -80.6 due to decomposition of blend polymer's side chains; and the third stage occurs between 550 and 900°C and causes weight losses of approximately 96.17 because a disintegration of main chain of blend polymer. DSC curve displays a crystalline temperature point (Tc) of 85 °C, an exothermic peak at 83 °C reflecting the glass transition temperature (Tg), an endothermic melting point (Tm) of 130 °C for modified PVA, and a degradation point at 300 °C. The TGA curves for compound (12) blend polymer, showing in Figure 9, reveal two stages of a continuous mass loss. The first phase involves a mass loss of volatile compounds of (-25.52) at (45–500) °C. The blended polymer's side chain decomposes around 500-900 °C in the second stage, resulting in a weight loss of roughly (-50.70). A glass Tg of 73 °C was indicated by DSC curve. Exothermic peak at crystalline temperature (Tc°) of (86) °C. The blend polymer melting point (Tm) at 125 °C is the endothermic peak. At (310) °C, the modified PVA starts to degrade. The TGA thermogram for compound (13), depicted in Figure 10, shows two decomposition phases. The first step (50–565) has weight losses of around (–55.21%). The second phase (560-910) resulted in a -97.49% weight decrease. DSC curve revealed that the modified polymer starts to degrade at (236.1) °C, with a glass Tg of (79) °C, a crystalline temperature point (Tc) at (100.3), and a melting point (Tm) at (192.7) °C.

The results show that the Tg of blend polymers is higher than that of pure PVA (PVA with a standard molecular weight of 72,000). The reason is that the presence of solid groups, such as the aromatic structures of the polymer, increases the degree of glass transition of the polymer and the steric hindrance imposed by the groups on the polymer chain. From the Table, we note that the Tc of blend polymer decreased compared with pure PVA because of the existence of a heterogeneous aromatic ring, which causes the disturbance of the hydrogen bonds necessary to form the crystals. As a result, the crystallization decreases Tc. Tm of pure PVA is higher than that of blend polymers because of the presence of OH groups and hydrogen forces in PVA, while in the blend polymer, the OH groups were replaced by heterogeneous rings, which reduces these forces and reduces Tm. [37]

Table 2: TGA characteristic parameters for thermal analysis for polymers

Comp.	**TGA**			
	Step	**Ti/°C**	**Tf/°C**	**Wt.Los%**
(10)	1	50	200	-39.94
	2	200	550	-80.6
	3	550	900	-96.17
(12)	1	45	500	-25.52
	2	500	900	-50.70
(13)	1	50	560	-55.21
	2	560	900	-97.49

Table 3: DSC characteristic parameters for thermal analysis for polymers

Comp.	**DSC**			
	Tg/°C	**Tc/°C**	**Tm/°C**	**Td/°C**
(10)	73.54	85.93	130	300
(12)	73.36	86.03	125	310
(13)	63.56	76.46	100	350

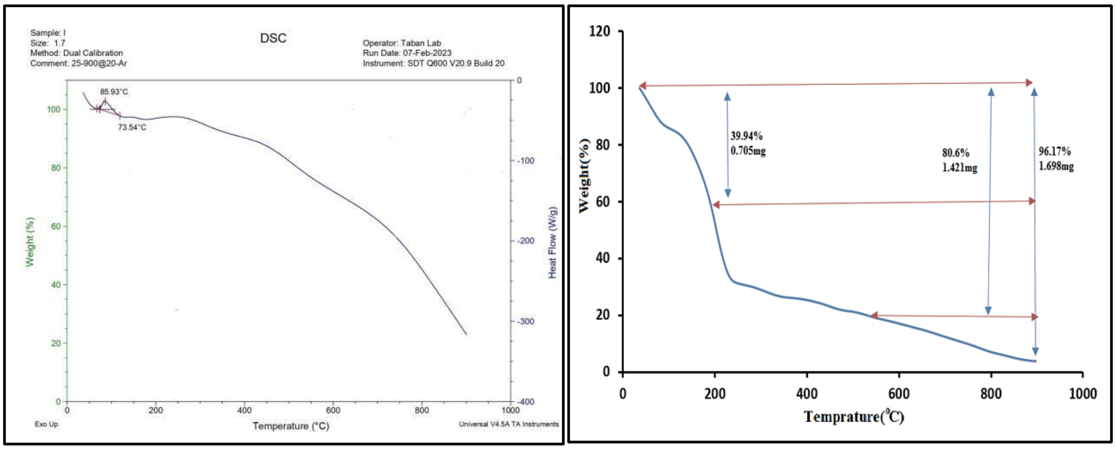


Figure 8 : TGA and DSC of blend polymer (10)

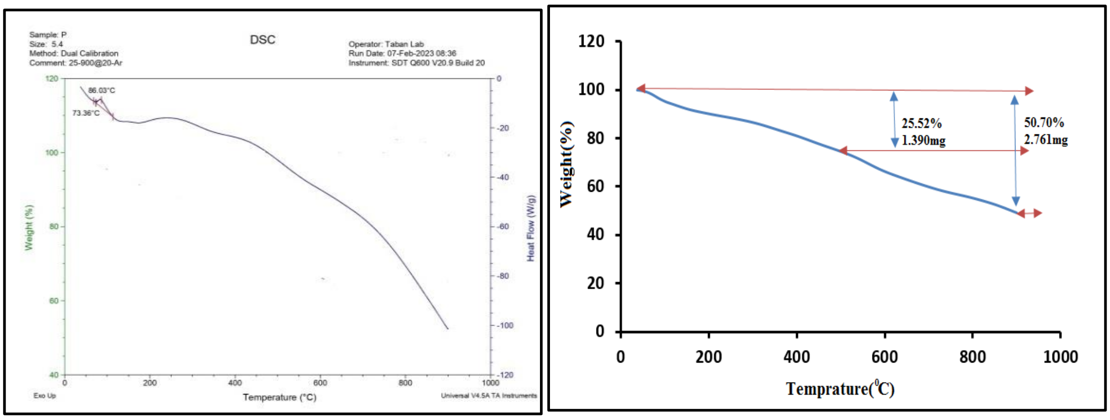


Figure 9 : TGA and DSC of blend polymer (12)

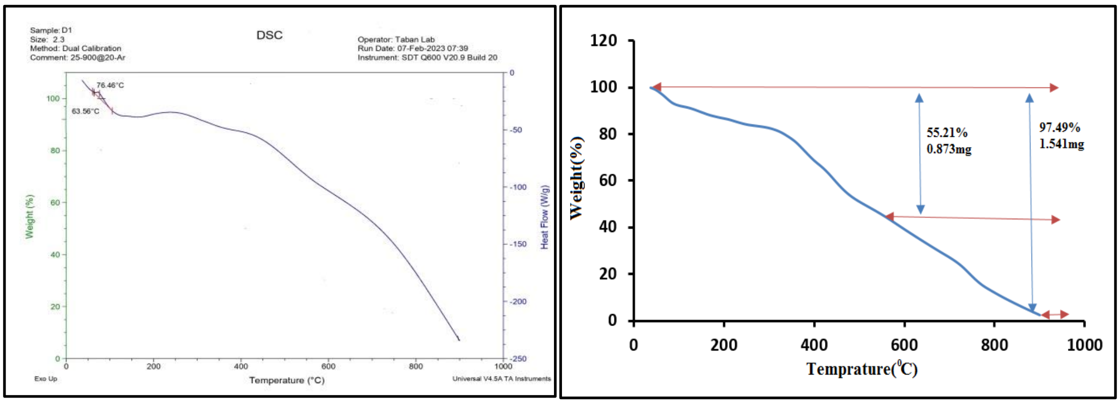


Figure 10: TGA and DSC of blend polymer (13)

SEM Studies

SEM micrographs were employed to examine changes in surface morphology for prepared polymer (6) and blend polymer (10), as shown in Figures 11 and 12, respectively. The SEM images, reveal that the average pore size in the polymer was 62-68 nm, while the pore size in the blend polymer ranged from 100 to 108 nm. These variations of surface morphology result from new bonds in the polymer that have been prepared. Following the polymer-to-polymer interaction, the SEM images showed that the surface of the produced blends had undergone considerable changes as we observed a homogeneous distribution of polymers on the surface of the polymer blend [38].

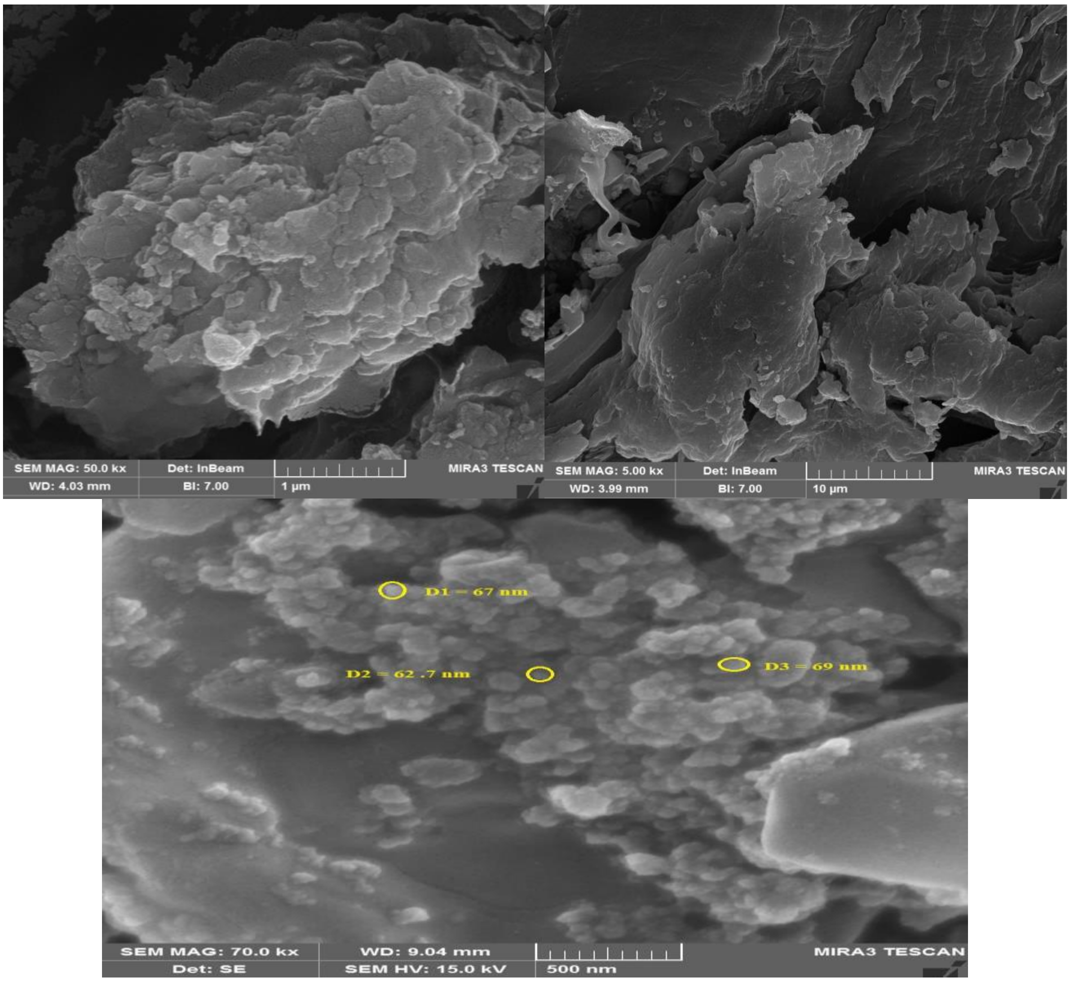


Figure 11 : SEM of polymer (6)

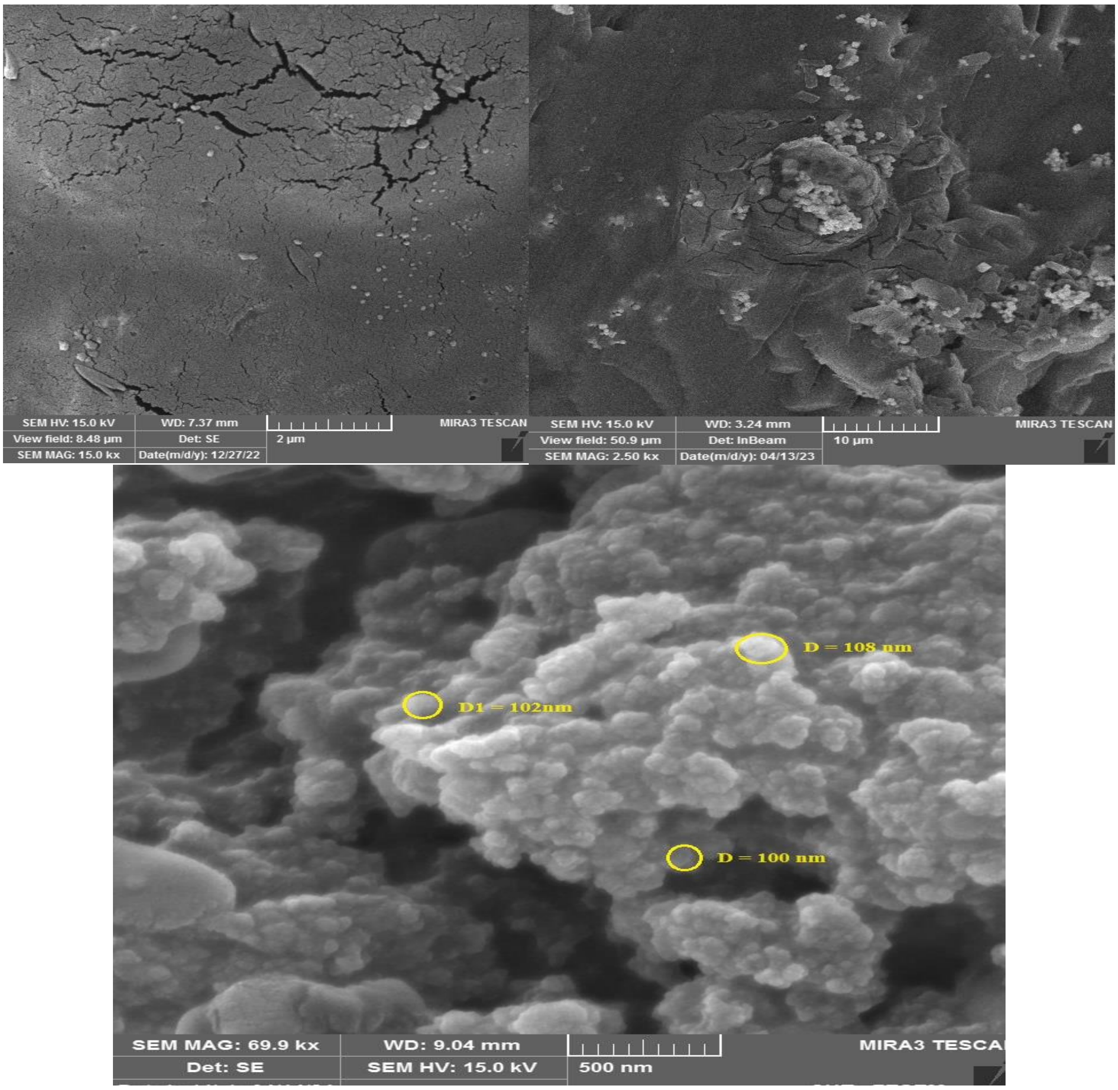


Figure 12 : SEM of blend polymer (10)

Conclusion

The polymers were synthesized with good yield, ranging from 77-93 %, and exhibited high melting point between 198 and 272 °C. The spectral analysis results corresponded to the proposed structure of the polymers. TGA results indicated that the Tg of blend polymers is higher compared to pure PVA, and the new blending polymers demonstrated a fair degree of miscibility between PVA and the polymers produced through strong intermolecular hydrogen bonding, which exists between (OH) of the PVA and (O) of prepared polymers. on the other hand, decreases their Tc of blend polymers comparing with pure PVA, as well as a reduction in their Tm of blend polymers if your comparative with PVA. The SEM images showed an increase in the average size of the pores of blending polymers compared to PVA pore size, which revealed considerable changes on the surface of prepared blends after interaction between polymers. Additionally, the study of biological activity demonstrated that the blending polymers are more active than the Amoxicillin drug against two strains of pathogenic bacteria.

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