

Journal of Medicinal and Chemical Sciences

Journal homepage: <u>http://www.jmchemsci.com/</u>



Original Article (Special Issue)

Preparation and Characterization and Thermal Study of New Copolymers of Semicarbazide and Fatty Acids Derivatives with Malic Anhydride and Assess their Biological Activity

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ARTICLE INFO

Article history Receive: 2022-06-22 Received in revised: 2022-07-12 Accepted: 2022-09-01 Manuscript ID: JMCS-2208-1661 Checked for Plagiarism: Yes Language Editor: Dr. Behrouz Jamalvandi Editor who approved publication: Dr. Zeinab Arzehgar

ABSTRACT

In this study, new semicarbazide derivatives $[E_1-E_5]$ were prepared by the reaction of semicarbazide with different fatty acids (olic, linolic, linolinic, recenolic and cinnamic acid) in DMF, then this compounds will polymerization with malic anhydride in H₂O₂ as initiator to get $[EP_1-EP_5]$. The prepared compounds were characterized by physical properties, UV-Vis, FT-IR, ¹H-NMR, ¹³C-NMR and TGA analysis.TLC checked the purity for these compounds.The antibacterial activity were studied against different kinds of bacteria, namly*Stophylococcuceaures,Stophylococcuce Saprophytic* and *Protes*.

DOI:10.26655/JMCHEMSCI.2022.7.23

KEYWORDS

Semicarbazide Fatty acids Malic anhydride copolymers Thermal study of fatty acid polymer Assential fatty acid semicarbazide

$$\begin{array}{c|c} & \mathbf{GRAPHICAL ABSTRACT} \\ O & O \\ R-C-OH + H_2N-C-N-NH_2.HCl & H_2SO_4 \\ \hline & DMF, Reflux, 4h \end{array} \qquad \begin{array}{c} O & O \\ H_2SO_4 \\ R-C-N-N-C-NH_2 \\ \hline & R-C-N-$$

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Introduction

Plant oils offer many advantages apart from their renewability. Their world -wide availability and relatively low prices make them industrially attractive and feasible. as daily demonstrated with industrial oleo chemistry. Furthermore diverse chemistry can be applied on them. Leading to a large variety of monomers and polymers [1]. Most importantly, the synthetic potential of nature is very high with this renewable feedstock and consequently. Only a few minor modification reactions (if any at all) have to be performed in order to obtain suitable monomers for many different applications [2]. The double bonds present in fatty acids [FA] make them ideal starting materials for such transformations .Moreover olefin metathesis can not only be used for monomer synthesis but also as polymerization technique [3]. The possibility of polymerization of FA is feasible since in their structure many double bonds are found and available. Therefore, there is favored radical polymerization reaction through the hydrogen in the allyl position, duo to the higher reactivity of this hydrogen creating a distinct monomer [4]. A large number of polymers applications results from their early properties and the way they are processed. In order to increase these applications and also to improve some properties, it is common in polymer industry to under go by synthesis modification [4]. In a recent studies fatty acids demonstrated biological activity [5-18] and some physical properties [19-28] and its polymers [29-41].

Materials and Methods

All chemicals had been used as supplied by (Alfa Assar and Aldrich).

Devices instrument

The melting points were determined by Electro thermal Melting Apparatus 9300 in open capillary tubes that were uncorrected. Thin layer chromatography (TLC) was used for monitory the reaction and check purity.The FT-IR spectra were recorded using FT-IR 8400s shimadzu spectro photometer scale (400-4000 cm⁻¹). The UV-Vis spectra was measured in ethanol using shimadzu 800UV in rang (200-400) nm. ¹H-NMR and ¹³C-NMR spectrum was recorded on Varianoperaing at 400 MHz instrument using DMSO-d⁶ and CDCl₃ as a solvent.Thermal analysis *Tg*was recorded on instrument SDT Q600 V20.9 Build 20 under Nitrogen.

Synthesis methods.

Synthesis of semicarbazide derivatives $[E_1-E_5]$

Semicarbazide (0.0l mol, 0.75 g) was dissolved in (50 mL) of DMF. After complete dissolving, 0.0l mol of one of different fatty acid was add. After adding (3 drops) of diluted H_2SO_4 (1:1), the mixture was then refluxed for 4-5 hours and cooled to room temperature. Filtered, dried and recrystallized physical properties are given in Table 1 [42].

	R-C-N-N-C-NH ₂								
Comp. No.	R	Molecular formula	Color	M.P (°C)	Time (h)	Yield (%)	Rf. EtOH		
E ₁	CH3(CH2)CH=CH(CH)7C=O	M.Wt. (g/mole) C ₁₉ H ₃₇ N ₃ O ₂ 340.53	White	261 dce.	4	82	(cm) 3		
E2	CH ₃ (CH ₂) ₄ CH=CHCH ₂ =CH(CH ₂) ₇ C=O	C ₁₉ H ₃₅ N ₃ O ₂ 338.52	White	265	4	60	2.6		
E3	CH ₃ CH ₂ (CH=CHCH ₂) ₃ CH(CH) ₇ C=O	C ₁₉ H ₃₃ N ₃ O ₂ 332.50	Yellow	268-270	4	75	5		
E4	CH ₃ (CH ₂) ₅ CH(OH)CH ₂ CH=CH(CH ₂) ₇ C= O	C ₁₉ H ₃₇ N ₃ O ₃ 356.52	White	212-214	5	23	1		
E5	Ph-CH=CH-C=O	C ₁₀ H ₁₁ N ₃ O ₂ 221.23	White	160	6	50	5		

 O
 O

 U
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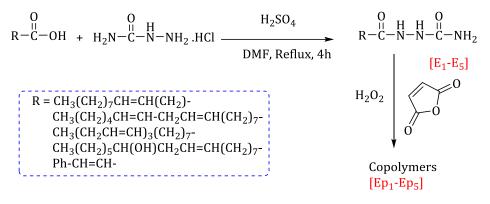
Synthesis of polymer [EP₁-EP₅]

In dry polymerization bottle (0.0l mol) of semicarbzide derivatives $[E_1-E_5]$ and equivalent moles of malic anhydride was dissolved in (10 mL) of DMF then (0.1 L of H_2O_2 was add and bottle was flushed with nitrogen and fimly stoppered.The

mixture was maintained at 80 °C for 3 h. then the resulted solution was poured into 25 mL of ethanol and precipitated polymer was filtered washed with ethanol and dried physical properties of polymer $[EP_1-EP_5]$ are listed in Table 2 [43].

Comp.	R	Color	S.P.	Yield	R.f. EtOH
No.			(°C)	(%)	(cm)
Ep1	CH ₃ (CH ₂) ₇ CH=CH(CH) ₇ C=O	Dark Brown	>300	85	1
Ep ₂	CH ₃ (CH ₂) ₄ CH=CHCH ₂ ch=CH(CH ₂) ₇ C=O	Dark Brown	198 dec.	75	1.5
Ep₃	CH ₃ (CH ₂) ₄ CH=CHCH ₂ CH=CH	Dark Brown	142 dec.	70	1.6
_	CH ₂ CH=CH(CH ₂) ₇ C=O				
Ep4	CH ₃ (CH ₂) ₅ CH(OH)CH ₂ CH=CH(CH ₂) ₇ C=O	Brown	130-137	85	1.2
Ep5	Ph-CH=CH-C=O	Yellow	194 dec.	80	1.8

Table 2: Physical	properties of pr	epared polymer	[EP1-EP5]



Scheme 1: Synthesis of polymer [EP₁-EP₅]

Results and Discussion

Characterization of selecarbazide derivatives $[E_1 - E_5]$

The semicarbazide derivatives $[E_1-E_5]$ were prepared by the reaction semicarbazide with (1mol) of fatty acids (olic, lenolic, lenolinic, recenolic and cinnamic acid) in DMF.

The UV spectra gave absorption band at different wave lengthe for the resulted semicarbazide derivatives (in %99 EtOH) due to $\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ transition and all these transition are listed in Table 3.

The FT-IR spectrum for semicarbazide derivatives showed to appear two band due to primary amid group (3595-3392), (3394-3300) cm⁻¹, beside secondary amid group which appear (3433-3249) cm⁻¹, ν (=CH) alken appear (3086-3010) cm⁻¹, (ν C-

H) aliphatic appear to band (2962-2926), (2860-2818) cm⁻¹, also (C=O) amid group appear (1695-1646) cm⁻¹ and (1600-1608) cm⁻¹ due to (ν C=C) alken, (CH₂ and CH₃) group appear (1492-1402) cm⁻¹, and (769-750), (754-715) cm⁻¹ attributed to ν (C-C)_{asy}, sym. In addition, a band at (3222) cm⁻¹ attributed to (ν OH) alcohol in [E₄] also appearance of bands (3190) cm⁻¹ due to ν (C-H)_{arom}, and (1616, 1525) cm⁻¹ due to ν (C=C)_{arom}. in [E₅],These results are in agreement with recent literature[14]. As shown in Figure S1 E₁. UV and FT-IR spectrum are given in Table 3.

Moreover the ¹H-NMR spectra of [E₁] Figure S2 shows a clear singlet signal at δ =10.66ppm attributed to -CO-NH and δ =10.37 ppm attributed to NH- C=O, triplet signal at δ = 7.23, 7.10,6.97 attributed to NH₂, triplet signal at δ =5.03, 5.02, 4.93, 4.74 ppm attributed to -CH=CH-, triplet signal at δ =4.74, 4.34, 4.33 ppm attributed to CH₂-C=O, multiple signal at arrange δ =4.33-2.01 ppm for CH₂ and singlet signal at δ = 2.01 to CH₃. As well as signal of DMSO-*d*₆ appear at δ = 2.50, 2.51, 2.52 ppm.

The ¹³C-NMR spectrum of [E₁] Figure S3 shows a clear singlet signal at δ =178.89 ppm attributed to-C=O-NH, singlet signal at δ =152.12 ppm attributed to -C=O-NH₂, doublet signal at δ =131.94, 119.34 ppm attributed to -CH=CH-, multiple signal at rang δ =25.50-18.02 ppm due to CH₂ and singlet signal at δ =15.87 ppm attributed to CH₃.

The ¹H-NMR spectra of [E₂] Figure S4 shows singlet signal at $\delta = 10.66$ ppm attributed to C=O-NH, singlet signal at $\delta = 10.37$ ppm attributed to HNC=O, triplet signal at $\delta = 7.23$, 7.10, 6.97 ppm attributed to NH₂, multiple signal an the rang 5.03-4.74 ppm attributed to-CH=CH, triplet signal at $\delta = 4.34$, 4.33, 3.98 ppm attributed to -CH=CH-CH₂*-CH=CH-, also multiple signal at $\delta = 3.58$ -2.01 ppm attributed ta CH₂ group and singlet signal at $\delta = 2.01$ ppm attributed to CH₃ as well as signal of DMSO-*d*₆ appear at $\delta = 2.52$, 2.51, 2.50 ppm.

The ¹³C-NMR spectrum of [E₂] shows singlet signal at δ =167.37 ppm attributed to C=O-NH and singlet signal at δ =155.21ppm attributed to C=ONH₂, Doublet signal at δ =132.17, 129.12 ppm attributed to -CH₌CH-, singlet signal at δ =61.78 ppm due to CH₂, multiple signal an the rang δ =17.90-16.62 ppm attributed CH₂, singlet signal at δ =14.35ppm due to CH₃.

The ¹H-NMR spectra of [E₃] Figure S5 shows a clear singlet signal at δ =11.12 ppm attributed to amin group in CH₂-CO-NH and singlet single at 10.58 ppm attributed to -CO-NH troubled signal appear at δ =7.26, 7.14, 7.01 ppm attributed to NH₂, triplet signal at δ =6.25, 5.83, 4.53 ppm attributed to CH=CH, triplet signal at δ =2.51, 2.44, 2.28 ppm attributed to CH₂ and singlet signal at δ =1.24 ppm attributed to CH₃. DMSO-*d*₆ signal appear at δ =2.51 ppm.

The ¹³C-NMR spectra of [E₃] Figure S6 shows doublet signal at δ =169.76, 163.40 ppm attributed to -CO-NH, and triplet signal at δ =150.45, 147.28, 145.34 ppm attributed to -CO- NH₂ singlet signal at δ =130.11ppm due to HC=CH, triplet signal at δ =79.77,79.44, 79.11 ppm attributed to CH=CHCH₂*CH=CH, multiple signals at rang δ =31.15 -16.66ppm due to CH₂ and singlet signal at δ =16.41 attributed to CH₃ group.

The ¹H-NMR spectra of [E₄] Figure S7 shows doublet signal to NH group at δ =9.90, 9.71 ppm and doublet signal for NH₂ group at δ =8.70, 8.58 ppm multiple signal at ring δ =7.90-7.01ppm attributed to -HC=CH-doublet signal at δ =3.39, 3.37 ppm duo to OH alcohol. Singlet signal at δ =2.29 ppm attributed to CH. multiple signal at rang δ =2.10-1.39 ppm duo to CH₂ and singlet signal at δ =0.85 ppm attributed to CH₃ as well as signal of DMSO-*d*₆ appear at δ =2.51 ppm.

The ¹³C-NMR spectrum of [E₄] shows singlet signal at δ =163.02 ppm attributed to C=O-NH and singlet signal at δ =154.17ppm attributed to C=ONH₂, Doublet signal at δ =1143.46, 135.41 ppm attributed to -CH₌CH-, singlet signal at δ =60.92 ppm due to C-OH, singlet signal at δ =53.21ppm due to CH₂, multiple signal an the rang δ =25.08-16.62 ppm attributed CH₂, and singlet signal at δ =16.41ppm due to CH₃.

The ¹H-NMR spectra of [E₅] shows doublet signal at $\delta = 10.91$, 10.73 ppm attributed to C=O-NH, $\delta = 10.37$ ppm attributed to C=ONH, multiple signal at $\delta = 7.75$ -7.61 ppm attributed to CH aromatic, doublet signal at $\delta = 7.68$, 7.67 ppm due to NH₂ and multiple signal at 6.67 -7.63ppm attributed to -CH=CH- aleph. As well as signal of DMSO-d⁶ appear at $\delta = 2.57$, 2.50 ppm.

The ¹³C-NMR spectrum of [E₅] shows singlet signal at δ =169.15 ppm attributed to C=O-NH and singlet signal at δ =159.08ppm attributed to C=ONH₂, multiple signal at δ =133.28-128.80ppm attributed to CH aromatic, doublet signal at δ =127.63, 125.60 ppm due to -CH=CH-

Characterization of polymers [Ep1-Ep5]

Semicarbazid derivatives copolymers $[Ep_1-Ep_5]$ were prepared from the reaction semicarbazide derivatives $[E_1-E_5]$ and malice hydride in DMF with H_2O_2 as initiator. The FT-IR spectrum showed to appear two band due to primary amid group (3450-3393), (3496-3340) cm⁻¹, beside secondary amid group which appear (3327-3192) cm⁻¹.

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Beside new bands appear at 1728-1701 cm⁻¹ attributed to the (C=O) of malice anhydride as well as the appearance of the bond at 1116-1105 cm⁻¹ due to (C-O) malic anhydride. The rest of the **Table 3:** FT-IR & UV-Vis data of pr

packages kept their positions as shown in Figure S8 and S9, Ep_1 , Ep_5 and FT-IR spectrum are given in Table 3.

Comp.					IR (KBr			1 5		
No.	ν (NH ₂) Asy. Sym.	ν (NH)	v (=CH) Alkene	v (CH ₂) (CH ₃) Alep. Asy.	v (C=O) Amid	N (C=C) Alkene	δ(CH ₂) (CH ₃) Asy. Sym.	ν (C-C)	other	λmax EtOH
				Sym.						
E1	3488	3288	3041	2962	1646	1600	1479	769	-	246
	3394			2918			1355	754		260
E2	3450	3249	3016	2948	1695	1608	1456	763	-	254
	3390			2858			1367	727		250
	0.40 7	0.400	0.000	0000		1 6 9 9	1.170			260
E3	3495	3433	3086	2926	1674	1600	1452	750	-	250
	3450			2860			1367	715		260 280
E ₄	4360	3250	3018	2927	1697	1608	1464	763	ν O-H Alcohol	251
24	3420	0200	0010	2850	2077	1000	1370	721	3222	265
E5	3392	3288	3010	2928	1690	1600	1492	769	ν C-H Ar.	278
	3300			2854			1381	719	3190	
									ν C=CAr.	
									1616	
									1525	
CoPoly			I		IR	(KBr) cm	1	1		
No.	ν	ν	ν	ν	δ (CH ₂)	N			other	
	(NH ₂)	(NH)	(CH ₂)	(C=O)	(CH ₃)	(C-O)				
	Asy.		(CH ₃)	Acid	Asy.					
	Sym.		Alep.		Sym.					
			Asy.							
			Sym.							
Ep_1	3414	3327	2912	1722	1460	1114			-	
	3373		2850		1373					
Ep ₂	3433	3271	2995	1718	1450	1105			-	
	3340		2880		1396					
Ep3	3450	3271	2926	1701	1421	1105			-	
	3496	00.17	2850	4 8 9 9	1380			~	1 1 1 0 : 2 2	
Ep4	3417	3265	2924	1730	1450	1116		OH a	lcohol 3433	
	3394	0400	2840	4500	1370			0.4		50
Ep_5	3394	3192	2930	1728	1460	1114	C=	C Aromat	ic. 1631,1595,14	58
	3350		2835		1367					

able 3: FT-IR & UV-V	is data of prepa	ared compounds a	nd Copolymers
	is until of prepa	n cu compounds a	ind copolymers

Thermal characterization of copolymers [Ep₂-Ep₅] The thermal behavior data of copolymers [Ep₂-Ep₅] is summarized in Table 5. Thermogram of $[Ep_3]$ show a broad single stage weight loss because of linkages in its structure. Is higher than other copolymers that may emanate from higher cross linking in copolymer backbond (Figure S11). Thermogram of [Ep₄] show a double stage weight loss because of double bond and OH alcohol in its structure that come from cross linking in copolymer bacbbond (Figure S12). Thermograms of [Ep₂] and [Ep₃] have three and four stage decomposition straight in the rate of 97.33%, 89.57%. Char yield of Ep₂ is higher than other copolymers (Figure S10 and S13).

Also the prepared copolymers showed good thermal stability through their thermal analysis, softening points and this due to the presence of semicarbazide and fatty acids and malic anhydride in their repeating units since insertion of these components in copolymeric chains exhibit the copolymer good thermal and chemical stability.

		Table 4: ¹ H& ¹³ C data of prepared compounds [E ₁ -E ₅]
Comp.		
No.		
E1	$^{1}\mathrm{H}$	¹ <i>H-NMR Spectrum, δppm, 300Hz, δ40 (DMSO-d</i> ₆), 10.66 (1H, NH), 10.37 (1H, NH), 7.23, 7.10,
		6.97 (2H, NH ₂), 5.03-4.74 (4H, CH=CH), 4.74, 4.33 (3H, CH ₂ C=O), 3.96, 3.58 (28H, CH ₂), 1.01
		(3H, CH ₃).
	¹³ C	¹³ C-NMR Spectrum, δppm, 300H _Z , δ40 (DMSO), 178.89 (O=C-NH), 152.12 (O=C-NH ₂) 131.94,
		119.34 (C=C), 25.50-18.02 (CH ₂), 15.87 (CH ₃).
E2	$^{1}\mathrm{H}$	¹ <i>H-NMR Spectrum, δppm, 300Hz, δ40 (DMSO-d</i> ₆), 10.66 (1H,NH-C=O), 10.37 (1H,NH-C=O),
		7.23, 7.10, 6.97 (2H, NH ₂), 5.03-4.74 (CH=CH), 4.34, 4.33, 3.98 (2H, CH ₂), 3.58, 2.01 (22H,
		CH ₂), 2.01 (3H, CH ₃), (28H, CH ₂), 1.01 (3H, CH ₃).
	¹³ C	¹³ C-NMR Spectrum, δppm, 300Hz, δ40 (DMSO), 167.37 (0=C-NH), 155.21 (0=C-NH ₂), 132.17,
		129.12 (C=C), 61.78 (CH ₂), 17.90-16.62 (CH ₂), 14.35 (CH ₃).
E3	$^{1}\mathrm{H}$	¹ H-NMR Spectrum, δppm, 300Hz, δ40 (DMSO-d6), 11.12(1H, NH), 10.58 (1H, NH-C=O), 7.26,
		7.14, 7.01 (2H, NH ₂), 6.25, 5.83, 4.53 (22H, CH ₂ -CH=CH), 2.51, 2.44, 2.28 (2H, CH ₂), 1.24 (3H,
		CH ₃).
	¹³ C	¹³ C-NMR Spectrum, δppm, 300H _Z , δ40 (DMSO), 169.76, 163.40 (0=C-NH), 150.45, 147.28,
		145.34 (O=C-NH ₂), 130.11 (C=C), 79.77, 79.44, 79.11 (CH=CH-CH ₂ *-CH=CH), 31.15-16.66
		(CH ₂), 16.41 (CH ₃).
E ₄	$^{1}\mathrm{H}$	¹ <i>H-NMR Spectrum, δppm, 300Hz, δ40 (DMSO-d</i> ₆), 9.90,(1H, NH), 9.71 (1H, NH), 8.7, 8.58, (2H,
		NH ₂), 7.90, 7.01 (2H, CH=CH), 3.39, 3.37 (1H, OH), 2.92 (1H, CH), 2.10-1.39 (15H, CH ₂), 0.85
		(3H, CH ₃).
	¹³ C	¹³ C-NMR Spectrum, δppm, 300Hz, δ40 (DMSO), 163.02, (0=C-NH), 154.17, (0=C-NH ₂), 143.46,
		135.41 (C=C), 60.92 (C-OH), 53.21 (CH ₂), 25.08-16.62 (CH ₂), 16.41 (CH ₃).
E5	$^{1}\mathrm{H}$	¹ H-NMR Spectrum, δppm, 300Hz, δ40 (DMSO-d ₆), 10.91, (1H, NH), 10.73 (1H, NH), 7.75-7.71
		(5H, CH-Ar), 7.68, 7.67, 7.65-6.97 (2H, CH=CH).
	¹³ C	¹³ C-NMR Spectrum, δppm, 300H _Z , δ40 (DMSO), 169.15 (0=C-NH), 159.08, (0=C-NH ₂), 133.28-
		128.80 (C-H _{Arom.}), 127.63, 125.60 (C=C).

Table 4: ¹ H& ¹³ C data of prepared compounds [H	E1-E5]	
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Table 5: Thermal properties of copolymers	[Ep ₂ -Ep ₅]
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CoPoly.	Step	TG%					
		$T_i C^o$	$T_f C^o$	Wt loss %			
Ep ₂	1	25	230	6.165			
	2	230	350	34.66			
	3	350	600	56.51			
Ep3	1	25	600	80.58			
Ep_4	1	250	290	33.07			
	2	290	600	12.87			
Ep5	1	25	150	3.655			
	2	150	225	10.37			
	3	225	350	70.53			
	4	350	600	5.027			

Antibacterial activity

The effect of the prepared compounds [Ep₁-Ep₅] on the growth of bacterla, namely *Staphylococcus auras, Staphylococcus staprophyticus* gram (+ve), *Proteus* gram (-ve). Antibacterial activity of the

prepared compounds were studied and the results showed that some of the prepared compounds possess good antibacterial activity. The results of in hibition zone diameter (IZD) in millimeter are shown in Table 5 [16].

Table 6 : Growth inhibition zone diameter (mm) of seven heterocyclic compounds against two pathogenic
hacteria species

		bacteria species		
Comp. No.	Conc. (mg/mmol)	Staphylococcus auras	Staphylococcus saprophytic	Protes
P1	0.01	17	20	11
	0.001	14	14	13
	0.0001	19	13	16
P2	0.01	20	15	12
	0.001	23	25	14
	0.0001	20	20	11
Р3	0.01	25	10	31
	0.001	22	14	28
	0.0001	25	10	29
P4	0.01	21	14	21
	0.001	23	19	20
	0.0001	27	14	24
P5	0.01	21	14	19
	0.001	22	17	21
	0.0001	21	10	16
Ep1	0.01	20	21	16
	0.001	24	20	18
	0.0001	21	25	15
Ep ₂	0.01	21	15	10
	0.001	21	21	11
	0.0001	20	25	10
Ep3	0.01	23	24	21
	0.001	18	25	19
	0.0001	22	25	23
Ep4	0.01	26	16	21
	0.001	23	10	19
	0.0001	16	15	20
Ep5	0.01	11	14	27
	0.001	26	15	26
	0.0001	15	21	30
Amoxicillin	0.01	12	11	15
	0.001	13	14	10
	0.0001	14	13	11
Ampicillin	0.01	22	22	23
-	0.001	23	21	33
	0.0001	44	24	40
Ciprofluxacin	0.01	20	21	23
	0.001	36	26	41
	0.0001	34	30	43
Blank dish	0.01	0	0	0
	0.001	0	0	0
	0.0001	0	0	0

Conclusion

The aim of the research was to preparation of some new compounds and polymers for some

fatty acids and study of their thermal stability, which showed high stability. The antibacterial activity of all of the synthesized compounds [E1-EP5] was tested in vitro. It showed good activity against selected gram-negative and gram-positive bacteria.

Funding

This research did not receive any specific grant from funding agencies in the public, commercial, or not-for-profit sectors.

Authors' contributions

All authors contributed toward data analysis, drafting and revising the paper and agreed to responsible for all the aspects of this work.

Conflict of Interest

Authors have declared that they have no known competing financial interests or non-financial

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HOW TO CITE THIS ARTICLE

Iman Aywob Yass. Preparation and Characterization and Thermal Study of New Copolymers of Semicarbazide and Fatty Acids Derivatives with Malic Anhydride and Assess their Biological Activity, *J. Med. Chem. Sci.*, 2022, 5(7) 1347-1356 https://doi.org/10.26655/JMCHEMSCI.2022.7.23 URL: http://www.imchemsci.com/article 155761.html