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# Preparation and characterization of new compounds for esters and polymers derived from furfural, study of some physical applications and evaluation of their biological activity

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**Abstract**---This research includes the preparation of esters and loading of polymers through the reaction of amino acids derived from furfural with alcohols and polyvinyl alcohol by the traditional method (sublimation). 1:1 mol) and then reacts the product of Schiff's bases with the anhydride. The compounds were diagnosed using infrared spectroscopy (IR), nuclear magnetic resonance ( $^1\text{H}$ ,  $^{13}\text{C}$ -NMR), UV-Vis spectroscopy, and mass spectrometry (Mass). Laser bombardment was also performed using a scanning electron microscope (SEM) to determine the shape of the particle and whether it was a nanoparticle or not. Also, the biological activity was evaluated on four types of karma-positive and gram-negative pathogenic bacteria, which showed moderate to high inhibitory ratios and measured the molar electrical conductivity of some of them. The products were confirmed by the physical changes that occurred to them, such as color, molecular weight, and melting point.

**Keywords**---Furfural, Schiff's bases, Amic acids, Esters, Pseudomonas Putida, Klebsiella Pneumoniae, Staphylococcus Aureus, Enterococcus.

## 1. Introduction

Furfural (a heterocyclic compound) and its derivatives have been used extensively in industrial and applied fields (nematicides, fungicides, and insecticides),

plastics, flavor enhancers for food and beverages, resins, decolorizing agents, and transportation fuels. Aviation fuel stocks, gas line additives, lubricants, and medicines [1]. In addition, it has wide industrial applications, including its tensile strength and a high degree of transmission to glass, which makes it suitable for the manufacture of compressed glass fibers exceeding its power (349 MPa), its corrosion resistance, and as a catalyst in cellulosic kinin compounds, but it is not strong when used as an adhesive; Therefore, it is strengthened by adding it to epoxy resins, maleic anhydride, and ethylenediamine to improve its mechanical properties [2]. Furfural has been linked to several mono- and diamine compounds to form Schiff bases through simple condensation to form the azomethane group (C=N). Through a simple condensation between ketones or aldehydes with primary amines, which are among the famous and essential organic chemical compounds in organic preparation [3,4]. Amic acids are distinguished by the diversity of their compounds and the multiplicity of methods of use; This is due to its many benefits, whether in organic preparations for imides or isoimides or in the field of polymer science, and it has also been used in the field of pharmacology and the pharmaceutical industry [5]. It has been used to treat epilepsy [6,7], and arthritis [8]. As anti-tuberculosis bacilli and nematodes [9]. It was also used in medical diagnosis [10]. It was also used as an insecticide and fungicide; Because of its high ability to annihilate [11, 12]. And extinguishing fires and preventing the spread of flames [13]. Esters are organic compounds (R-CO-OR'), and they are one of the derivatives of carboxylic acids that can be saturated, unsaturated or aromatic aliphatic. Because esters contain a carbonyl group, they are polar and have a molecular weight close to aldehydes and ketones; Therefore, the boiling point of esters is lower than theirs [14]. Esters are used in many industries; Because they are easy to form, whether cyclic or noncyclic. In addition to that, they are bases that protect the hydroxyl and carbonyl groups, and their formation results in a group of natural products, including sugars and peptides, which are more prevalent than others [15]. It may also be included in the composition of drugs, such as the anti-tumor drug Quadron, a compound (methyl taxol) used to treat ovarian cancer [16], and breast cancer [17].

## 2. Experimental

**2.1. Chemicals used:** All chemicals used in this work were purchased from BDH, Aldrich and Fluka companies and were used without further purification.

**2.2. Devices used:** The melting points were measured using Electrothermal Melting Apparatus 9300. The FT-IR spectra were captured using a Shimadzu FT-IR 8400S spectrophotometer with a (400-4000)  $\text{cm}^{-1}$  by KBr disc. DMSO- $d_6$  as solvents were used to capture  $^1\text{H-NMR}$  and  $^{13}\text{C-NMR}$  spectra on Bruker instruments running at 400 MHz.

### 2.3. Preparation method [18]:

In a circular flask (250 mL) (0.01 mol) of dissolved alcohol is placed in a suitable solvent according to each compound mentioned in the table below, and then (7-5 drops) of acid are added. Concentrated hydrochloric to it with continuous stirring and the same number of moles of prepared amino acids dissolved in the same type of solvent for each alcohol was placed in a separating funnel and added in the form of drops to the alcohol so that no precipitate formed, as it was

sublimated in a water bath with a degree of (100). °C for (3 hours), after which the mixture was placed in ice for (30 minutes) and no precipitate formed, so the solution was evaporated and a deposit was obtained.

Table (1): shows some physical properties of the prepared compounds

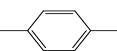

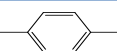
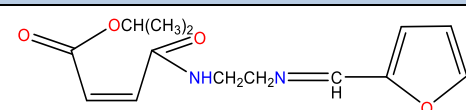
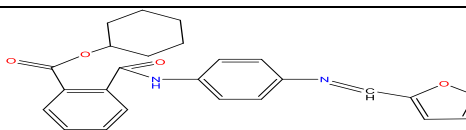
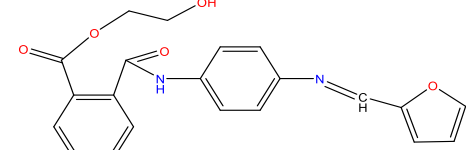
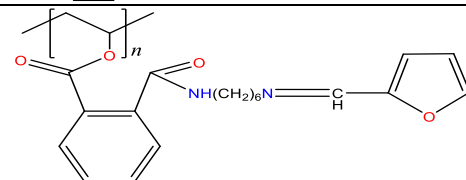
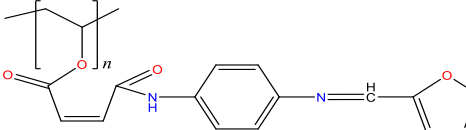
Comp. No.	Furfural-Ar/R-Anhydride-Alcohol	Anhydride	Alcohol	Yield%	M.P °C	Color	Recr. Solvent
WS-35	-CH <sub>2</sub> -CH <sub>2</sub> -	Maleic	Isopropyl	70	186-188	Gray	Water
WS-49		Phthalic	Cyclohexanol	65	153-155	Black	THF
WS-50		Phthalic	Ethylene glycol	63	172-174	Black	THF
WS-45	-(CH <sub>2</sub> ) <sub>6</sub> -	Phthalic	PVA	66	Gum	Brown	Water
WS-48		Maleic	PVA	65	Gum	Red	Formaldehyde

Table (2): Prepared Compounds

Comp. No.	Structure	Name
WS.Z1		isopropyl (Z)-4-((2-(((E)-furan-2-ylmethylene)amino)ethyl)amino)-4-oxobut-2-enoate
WS.Z2		cyclohexyl (E)-2-((4-((furan-2-ylmethylene)amino)phenyl)carbamoyl)benzoate
WS.Z3		2-hydroxyethyl (E)-2-((4-((furan-2-ylmethylene)amino)phenyl)carbamoyl)benzoate
WS.Z4		sec-butyl 2-((6-((furan-2-ylmethylene)amino)hexyl)carbamoyl)benzoate
WS.Z5		sec-butyl (2Z)-4-((4-((furan-2-ylmethylene)amino)phenyl)amino)-4-oxobut-2-enoate

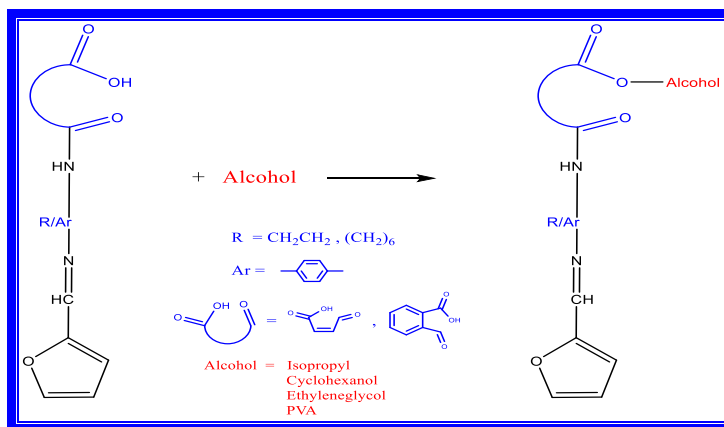
#### 2.4. Biological efficacy assessment [19]

When studying the biological activity of some prepared compounds, four types of pathogenic bacterial isolates were used, two are Gram-negative (Gr-Ve) and they

are [*Pseudomonas putida* (P-P-), *Klebsiella Pneumoniae* (K-)], and two Gram-positive (Gr +Ve) [*Staphylococcus Aureus* (S.A+), *Enterococcus Faecalis* (E.F+)], which is very important in the medical field; This is due to its high resistance to antibiotics. These bacteria were obtained from the College of Education for Pure Sciences - Department of Life Sciences laboratories. The culture medium used is Mueller-Hinton Agar, which is a transparent nutrient medium with a yellow color and is very useful in testing the sensitivity of microorganisms against antibiotics; Because it contains an animal infusion extracted from starch and casein, as it has a high ability to grow most microorganisms and microbes, and it is widely used in measuring the biological effectiveness of antibiotics in addition to chemicals with medical uses and is also used in measuring and determining the minimum inhibitory concentration (MIC). The effectiveness of the prepared compounds on bacteria was tested by the agar well diffusion method (18), and chemical solutions of the compounds were prepared [WS.Z2, WS.Z4, WS.Z5]. ] using a solvent (DMSO), and each compound in four concentrations (10<sup>-5</sup>, 10<sup>-4</sup>, 10<sup>-3</sup>, 10<sup>-2</sup> g/ml). The culture medium Müller-Hinton Agar was prepared using a pressure oven for sterilization (Autoclave) and then distributed on Petri dishes and left to solidify. Then four holes were worked in each dish and then placed in the incubator for (24 hours). at a degree of (37 °C), the results were read to show the sensitivity of the compounds used, which depends on measuring the diameter of the damping around the holes visible in the dishes. This is compared with the size of the inhibition diameter for the standard antibiotics used.

### 3. Results and Discussion

Esters were prepared from the reaction of amino acids derived from furfural with alcohols in a ratio of (1:1 mol), as shown in the following equation:



#### 3.1. Spectroscopic interpretation (IR, <sup>1</sup>H-NMR, <sup>13</sup>C-NMR, UV, Mass)

When studying the infrared spectrum of the compound [WS.Z1], an absorption band for the (NH) group appeared at 3522 cm<sup>-1</sup>, and a band appeared at 1778 cm<sup>-1</sup> belongs to the stretching group (C=O), and a stretching band appeared at 1697 cm<sup>-1</sup> belonging to the (C=O) group. Amide and a stretching band appeared at 1274 cm<sup>-1</sup> belonging to the (C-O) group of the furan ring, and a bundle at 1240 cm<sup>-1</sup> belonged to the (C-O) group of the furan ring. Stretchable (C-O) steric. By

studying the infrared spectrum of the compound [WS.Z5], an absorption beam for a group (NH) appeared at  $3407\text{ cm}^{-1}$ , and a stretching beam appeared for a group (C=O) esterified at  $1733\text{ cm}^{-1}$ . A band appeared at ( $1654\text{ cm}^{-1}$ ) belonging to a stretch group (C=O) amide, a band appearing at  $1279\text{ cm}^{-1}$  belonging to the stretching group (C-O) of the furan ring, and a band appearing at  $1240\text{ cm}^{-1}$  stretching (C-O) Istrian [20].

Table (3): Values of FT-IR spectrum in  $\text{cm}^{-1}$  units for compounds [WS.Z1-WS.Z5]

Comp. No.	IR (KBr) $\text{cm}^{-1}$				
	$\nu\text{NH}$	$\nu\text{C-H}$ Aromatic	$\nu\text{C=O}$ Ester	$\nu\text{C=C}$ Olephenic	$\nu\text{C-N}$
	$\nu\text{OH}$	$\nu\text{C-H}$ Alkyl	$\nu\text{C=O}$ Amid	$\nu\text{C=C}$ Aromatic	$\nu\text{C-O}$ Furan Ring
	$\nu\text{C-H}$ Olephenic	$\sigma\text{C-H}$ Alkyl	$\nu\text{C=N}$	$\nu\text{C=C}$ Aromatic	$\nu\text{C-O}$ Ester
WS.Z1	3522	3064	1778	1560	1325
	/	2987	1697	1500	1274
	3292	1392,1371	1627	1438	1240
WS.Z2	3406	3066	1707	/	1319
	/	2929	1656	1550,1506	1261
	/	1406	1604	1452	1222,1203
WS.Z3	3418	3055	1703	/	1311
	3319	2933	1654	1556,1504	1261
	/	1398	1622	1454	1226,1203
WS.Z4	3398	3068	1714	/	1398,1377
	3342	2937,2862	1637	1552	1247
	/	1438	1616	1469	1221
WS.Z5	3407	3053	1733	1562	1329
	3369	2921,2862	1654	1517	1279
	3253	1396	1620	1473	1240

By studying the proton nuclear magnetic resonance spectrum ( $^1\text{H-NMR}$ ) of the compound [WS.Z1], we note that there is a signal at (8.01) ppm that belongs to the group (HC=N), as we note that Signals appear at (ppm 7.84, 6.33, 6.23) belong to the group (HC=CH) of the furan ring. The two signals appear at (ppm 6.35, 6.21) belong to the group (HC=CH) for anhydride, as well as the signals appearing at (3.28, 3.27, 1.74, 1.08 ppm), belong to the aliphatic (CH) groups, and the signal at (9.04 ppm) belongs to the (NH) group [21].

The carbon nuclear magnetic resonance spectrum ( $^{13}\text{C-NMR}$ ) of the same compound showed a signal at (ppm 175.96) belonging to the carbon of the carbonyl group, and the signal at (ppm 159.03) belongs to the (HC = N) group. The two signals at (ppm 145.99 and 114.35) belong to the carbon groups in the furan ring, while the signal at (ppm 133.75) belongs to the (HC=CH) group of anhydrides. The two signals at (ppm 68.14) and 23.32) belong to the isopropyl group, and the reference at (ppm 43.19) belongs to the aliphatic (CH<sub>2</sub>) group.

When ultraviolet spectroscopy was performed in foresters, we observed the appearance of an absorption band ( $\pi \rightarrow \pi^*$ ) of small wavelength and greater intensity; Due to the presence of (C=C) pathologically shifted (C=C) bonds for the esters within the range (238 nm). Also, an absorption band ( $n \rightarrow \pi^*$ ) with a large wavelength and less intensity appeared because of the unshared electron pairs on the oxygen and nitrogen atoms and the bathochromic displacement of the presence of oxo and succession groups within the range (414 nm). By mass spectrometry, it was found that the molecular weight of the compound [WS.Z1] is (278.2), and the calculated molecular weight of the same compound is (278.31) [22].

$$\begin{aligned}
 [M/e]^{\dagger}: & \text{C}_{13}\text{H}_{17}\text{N}_2\text{O}_3 = 250.0 \quad , \quad \text{C}_{11}\text{H}_{11}\text{N}_2\text{O}_3 = 219.9 \quad , \quad \text{C}_9\text{H}_{14}\text{NO}_3 = 183.9 \\
 & \text{C}_7\text{H}_{10}\text{NO}_3 = 155.9 \quad , \quad \text{C}_7\text{H}_9\text{N}_2\text{O} = 136.9 \quad , \quad \text{C}_7\text{H}_8\text{NO} = 122.1 \\
 & \text{C}_6\text{H}_6\text{NO} = 108.0 \quad , \quad \text{C}_5\text{H}_4\text{NO} = 94.2 \quad , \quad \text{C}_5\text{H}_4\text{O} = 80.0 \\
 & \text{C}_3\text{H}_2\text{O} = 53.9
 \end{aligned}$$

The mass spectrometry also found that the molecular weight of the compound [WS.Z3] is (378.1), and the calculated molecular weight of the same compound is (378.38) [23].

$$\begin{aligned}
 [M/e]^{\dagger}: & \text{C}_{18}\text{H}_{16}\text{N}_2\text{O}_4 = 323.8 \quad , \quad \text{C}_{18}\text{H}_{13}\text{N}_2\text{O}_2 = 289.2 \quad , \quad \text{C}_{10}\text{H}_9\text{O}_4 = 193.1 \\
 & \text{C}_5\text{H}_4\text{NO} = 94.1 \quad , \quad \text{C}_5\text{H}_4\text{O} = 79.9
 \end{aligned}$$

### 3.2. Electrical conductivity

Electrical conductivity is widely used in coordination chemistry; This is to know the ionic formulas that can be obtained in the compound, whether a solid or a solution. The degree of conductivity increases as the number of ions liberated in the answer increases, while the degree of conductivity in the complex that does not ionize is low.

Table (5) The molar electrical conductivity values of the compounds [WS.Z2, WS.Z4, WS.Z5] in a solvent (DMSO) at a concentration ( $10^{-3}$ ) molarly

Test Comp.	Conductivity	T/°C
WS.Z2	7.1	22.9
WS.Z4	12.1	21.3
WS.Z5	12.9	21.0

### 3.3. Elemental Analysis (C.H.N):

The spectrum of elemental analysis of carbon, hydrogen, and nitrogen (C, H, N) showed the conformity of the structural formula of the two compounds [WS.Z1, WS.Z3] [24], as shown in the table below:

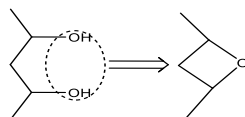
Table (4): Elemental analysis statement (C.H.N) for compounds [WS.Z1, WS.Z3]

Comp.	Found & Cal.	Carbon%	Hydrogen%	Nitrogen%
WS.Z1	Found	60.57764053	6.098001957	9.850709922

	Calculate	60.418167	6.518314	10.065538
WS.Z3	Found	66.88191605	4.340785503	7.360085487
	Calculate	66.659099	4.794418	7.40351

### 3.4. Thermolysis (DSC)

What is meant by thermal decomposition (Differential Scanning Calorimetry - DSC) is what occurs on crystallized polymers or transformed into glass, and it was found from the thermal analyzes of the two compounds [WS.Z4, WS.Z5] that they had high stability at temperatures exceeding ( 600 °C), and water molecules were lost from the unlinked PVA polymeric chains at (225-150 °C), which leads to the formation of quaternary rings of the corresponding epoxide, This was confirmed by the shape of a crystal of epoxide [25].



### 3.5. Scanning Electron Microscopy (SEM) Analysis:

The effect of laser bombardment by scanning electron microscopy (SEM) of the two compounds [WS.Z1, WS.Z4], the compound [WS.Z1] appears at (10 μm) in the form of sheets of Small scattered rock masses containing cracks, fissures, grooves and deep trenches that trap gases within them. Simultaneously, the compound [WS.Z4] appears as a hardened piece of plastic (similar to chocolate) with zigzags and scattered holes like volcanic holes and cracks of varying sizes. The surface also contained deep trenches that trap gases inside them.

At (1 μm), [WS.Z1] appears like an iceberg with fissures and deep trenches. Compound [WS.Z4] appears at the same distance in the form of a large piece of plastic containing large cracks, cavities, voids between them, and deep trenches. At (500 nm), [WS.Z1] appears like an iceberg with cracks and spurs with deep trenches. At the same distance, the boat [WS.Z4] appears in the form of a giant snowflake, on which small pieces of snow are stuck, interspersed with voids, cracks, and deep caves. At (200 nm), [WS.Z1] appears like an iceberg containing cracks and fissures with deep gaps, and trenches,, and nanoparticles. The compound [WS.Z4] appears at the same distance in the form of a giant snowflake that seems to be floating on which small pieces of snow are stuck, interspersed with voids, cracks, and deep caves the appearance of the nanoparticle electric [26].

### 3.6. Biological activity:

Through the biological activity of some of the prepared compounds, it was found that most of these compounds have antibacterial activity compared to some pre-manufactured antibiotics for these types of bacteria, such as Ampicillin, Cefixime, and Ceftriaxone) which are highly effective antibacterial that are widely used in the treatment of many infections caused by these bacteria, in addition to having a sizeable inhibitory diameter; Therefore, it gives a high selectivity when studying the sensitivity of bacteria to some of the prepared compounds. Since the compounds [WS.Z2, WS.Z4, WS.Z5] in this research have shown moderate to high efficacy on different types of chromium-positive and chromium-negative bacteria

compared to the mentioned antibiotics, it is possible to use these Compounds as a treatment for the same infections and pathological conditions of antibiotics, after ascertaining the biological pathway of these compounds and their side effects, and the amount of their accumulation in animal tissues. The results are shown in table (6) below indicate that most prepared compounds can inhibit bacteria through different concentrations (10<sup>-5</sup>, 10<sup>-4</sup>, 10<sup>-3</sup>, 10<sup>-2</sup>). g/ml, as the diameter of the damping ranges between (0-20 mm). Figures (12-14) show that the brake values vary according to the compound; This is due to the low nasalization and the presence of resonance [27,28].

Table (6): The inhibitory activity of the compounds [WS.Z2, WS.Z4, WS.Z5] on positive and negative bacteria (inhibition diameter measured in mm)

Comp.	C (g/ml)	K -	P.P -	S.A +	E.F +
<b>WS.Z2</b>	10 <sup>-2</sup>	0	0	18	6
<b>A</b>	10 <sup>-3</sup>	0	0	20	0
<b>B</b>	10 <sup>-4</sup>	0	0	14	10
<b>C</b>	10 <sup>-5</sup>	0	0	20	8
<b>K/d</b>		d=0	K=0	d=14	K=0
<b>WS.Z4</b>	10 <sup>-2</sup>	0	0	20	6
<b>A</b>	10 <sup>-3</sup>	0	0	16	2
<b>B</b>	10 <sup>-4</sup>	0	0	18	8
<b>C</b>	10 <sup>-5</sup>	0	0	16	14
<b>K/d</b>		K=26	K=0	K=0	d=4
<b>WS.Z5</b>	10 <sup>-2</sup>	12	14	16	14
<b>A</b>	10 <sup>-3</sup>	0	4	20	4
<b>B</b>	10 <sup>-4</sup>	0	0	10	2
<b>C</b>	10 <sup>-5</sup>	0	0	20	8
<b>K/d</b>		K=30	K=0	K=4	d=6

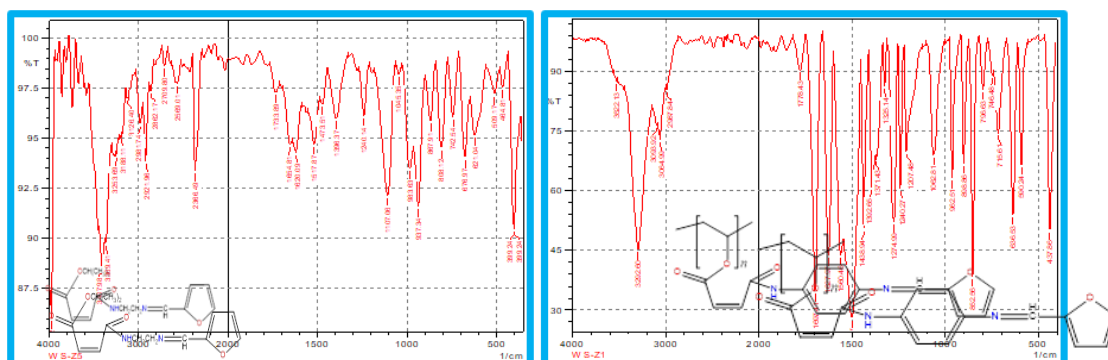


Figure 1: FTIR spectrum of WS.Z1 and WS.Z1

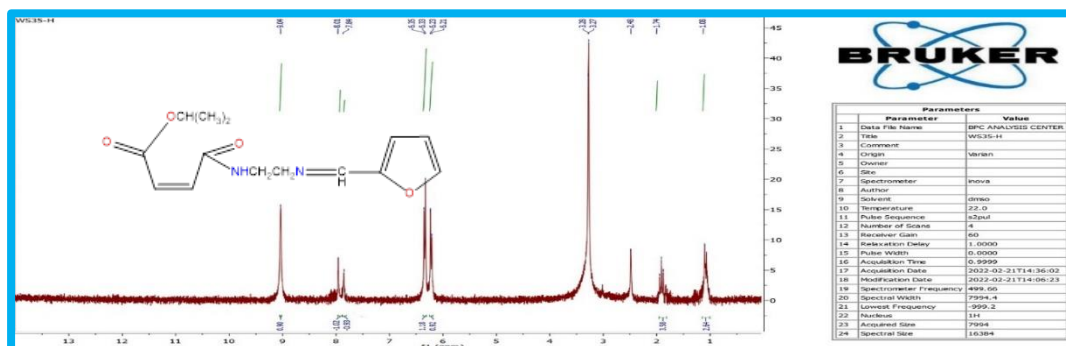
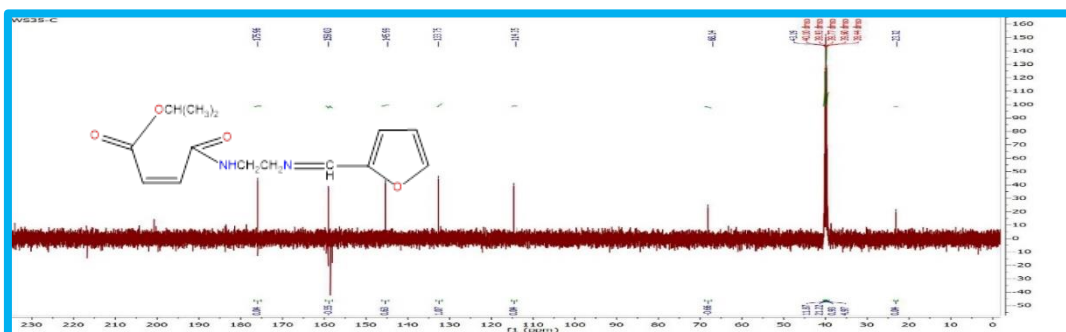
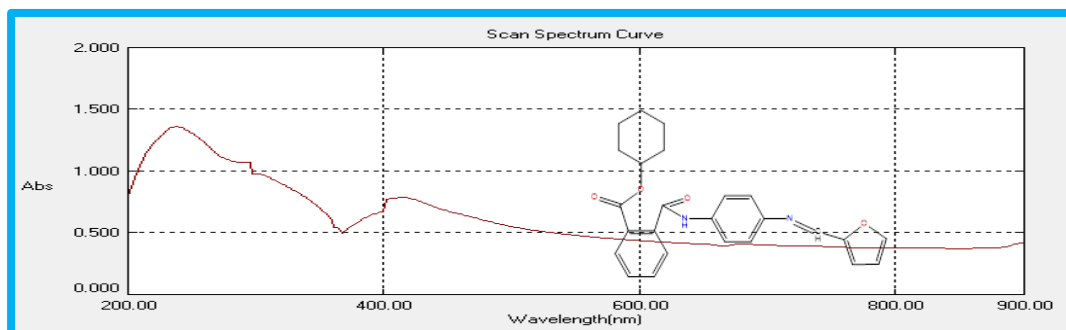
Figure 2:  $^1\text{H}$ -NMR spectrum of WS.Z1Figure 3:  $^{13}\text{C}$ -NMR spectrum of WS.Z1

Figure 4: UV-Visible spectrum of WS.Z2

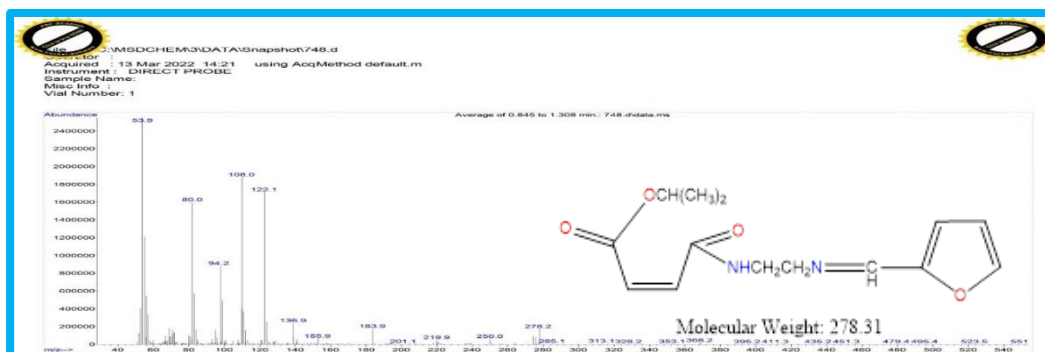


Figure 5: Mass spectrum of WS.Z1

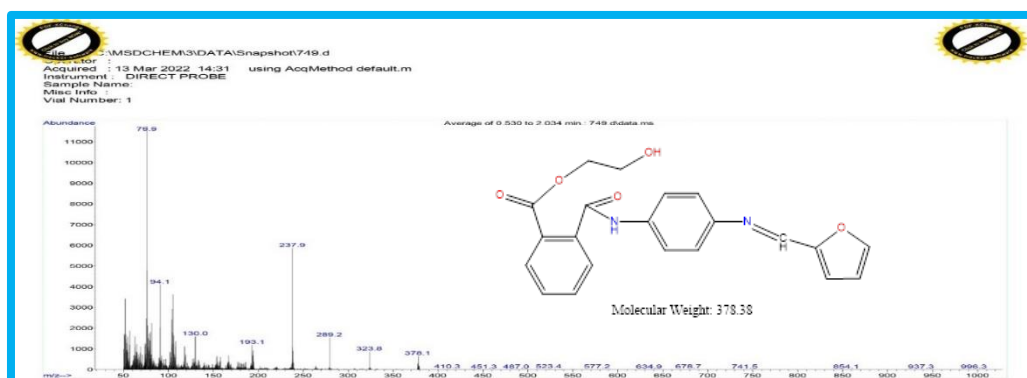


Figure 6: Mass spectrum of WS.Z3

Eager 300 Summarize Results					Eager 300 Summarize Results				
Date : 2002/01/03 at 00:11:53					Date : 2002/01/03 at 00:11:37				
Method Name : Nitrogen/Carbon/Hydrogen/Sulphur					Method Name : Nitrogen/Carbon/Hydrogen/Sulphur				
Method Filename : N C H S system.mth					Method Filename : N C H S system.mth				
Filename	AS Method	Vial			Filename	AS Method	Vial		
WS50					WS35				
# Group	Sample Name	Type Weig.	Pro.F	---	# Group	Sample Name	Type Weig.	Pro.F	---
56 10	WS50	UNK	3.178 6.25	---	55 8	WS35	UNK	2.93 6.25	---
Component name Element %					Component name Element %				
Nitrogen% 7.360085487					Nitrogen% 9.850709922				
Carbon% 66.88191605					Carbon% 60.57764053				
Hydrogen% 4.340785503					Hydrogen% 6.098001957				
Sulphur% 0					Sulphur% 0				
1 Sample(s) in Group No : 10					1 Sample(s) in Group No : 8				
Component Name	Average	Std. Dev.	% Rel. S. D.	Variance	Component Name	Average	Std. Dev.	% Rel. S. D.	Variance
Nitrogen%	7.360085487	0	0.0000	0.0000	Nitrogen%	9.850709922	0	0.0000	0.0000
Carbon%	66.88191605	0	0.0000	0.0000	Carbon%	60.57764053	0	0.0000	0.0000
Hydrogen%	4.340785503	0	0.0000	0.0000	Hydrogen%	6.098001957	0	0.0000	0.0000
Sulphur%	0	0	0.0000	0.0000	Sulphur%	0	0	0.0000	0.0000

Figure 7: C.H.N. of WS.Z1 and WS.Z3

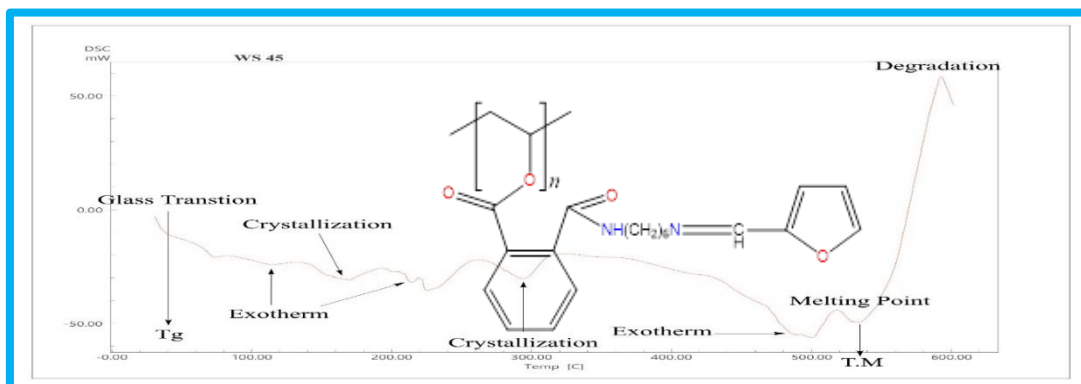


Figure 8: Analysis DSC of WS.Z4

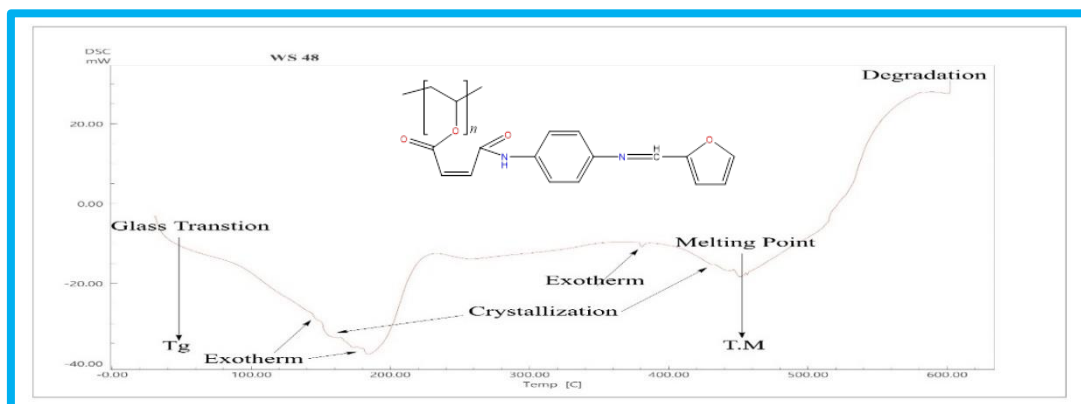


Figure 9: Analysis DSC of WS.Z5

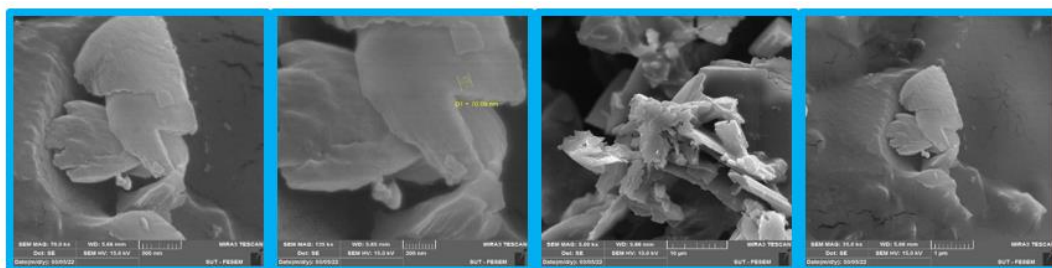


Figure 10: SEM of WS.Z1

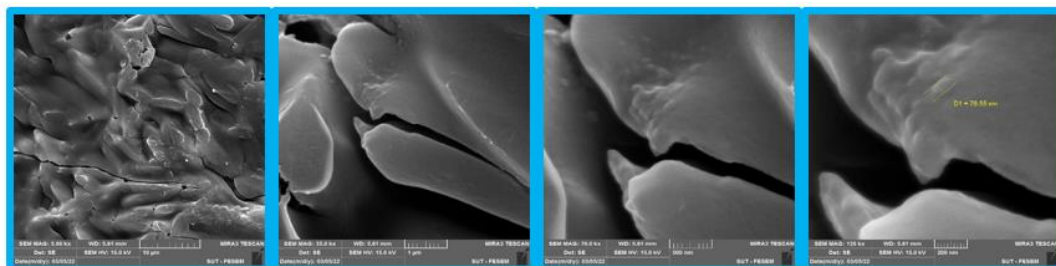


Figure 11: SEM of WS.Z4

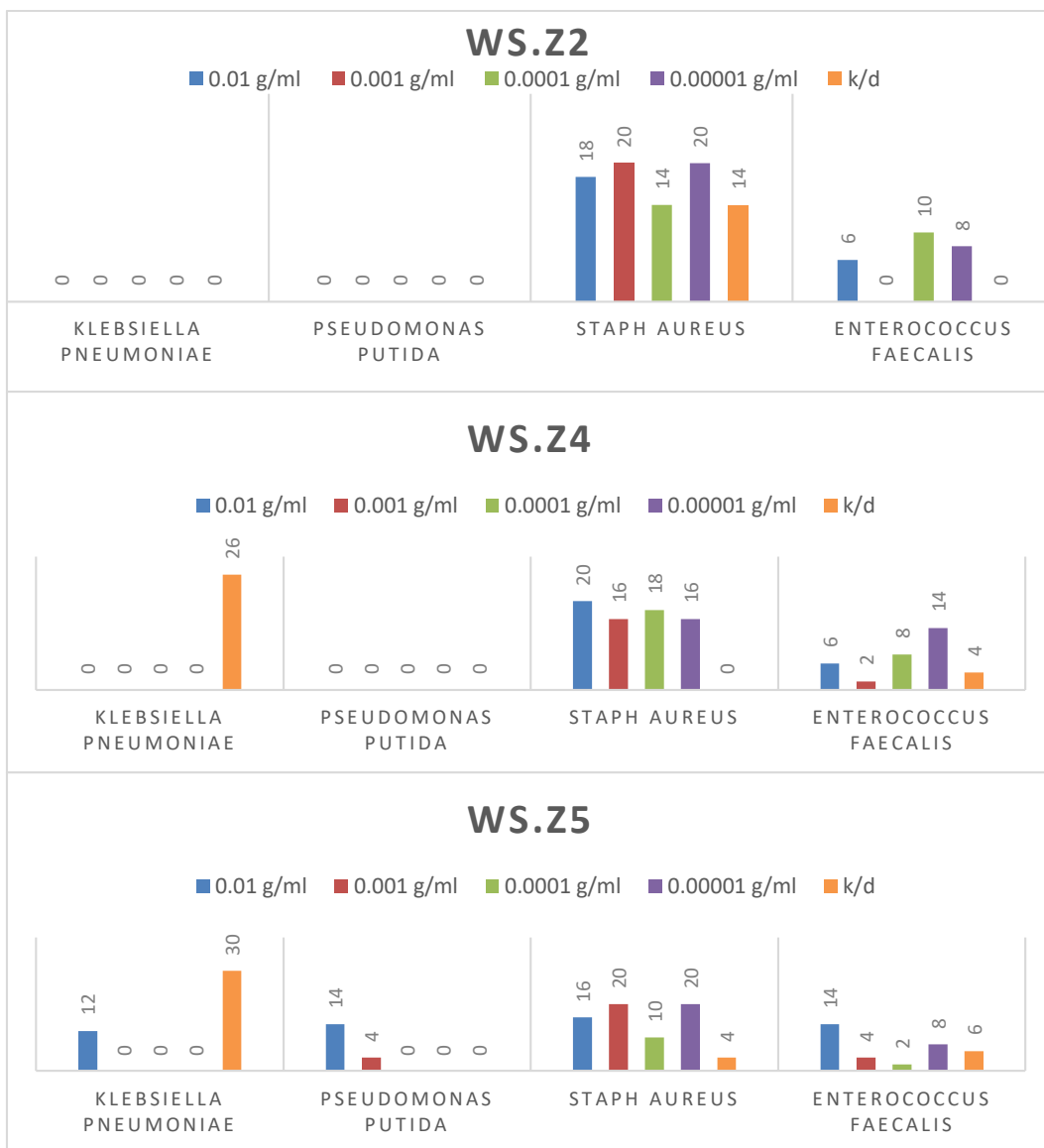


Figure 12: Inhibitory activity of (WS.Z2, WS.Z4, WS.Z5) on the four types of bacteria

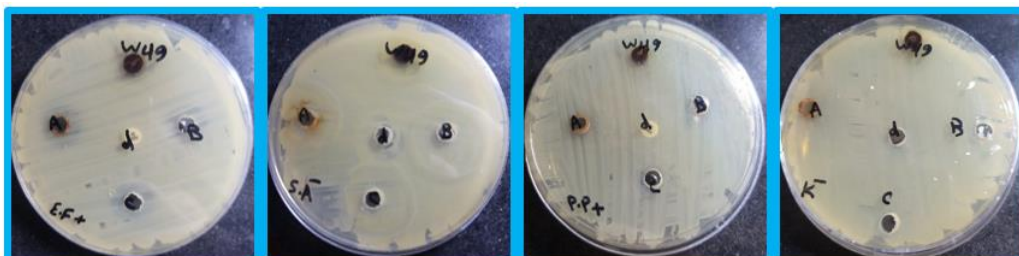


Figure 13: Compound WS.Z2 inhibits the growth of on the four types of bacteria

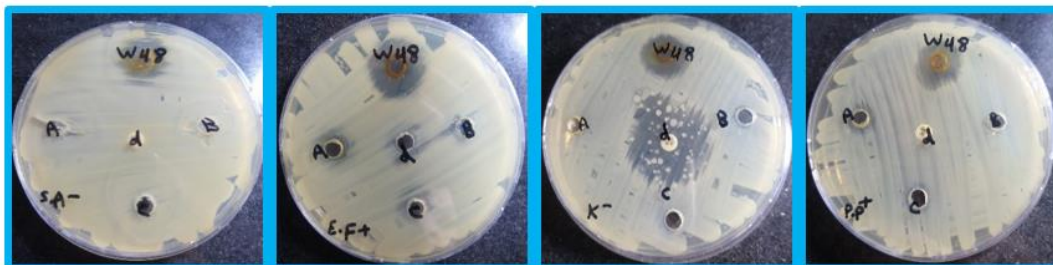


Figure 14: Compound WS.Z5 inhibits the growth of on the four types of bacteria

#### 4. Conclusions

Physical and spectroscopic measurements confirmed the accuracy and validity of the prepared compounds. So the methods used in the preparation were good, successful, and low cost. The values of the microanalysis of the components of the prepared compounds were identical or close to the calculated percentage. The prepared compounds also showed good efficacy against the bacteria used in the study. It was also found that the prepared compounds had good electrical conductivity. Thermal analyzes of the two compounds [WS.Z4 and WS.Z5] revealed high stability at temperatures exceeding (600 °C), and water molecules were lost from the polymeric chains of polyvinyl alcohol (PVA). Linked at a degree (225-150 °C) leads to the formation of quaternary rings of the corresponding epoxide, which was confirmed by the shape of a crystal of epoxide. It was also shown that the effect of laser bombardment by a scanning electron microscope (SEM) of the compounds [WS.Z1 and WS.Z4], compound [WS.Z1] appears at (10  $\mu\text{m}$ ) as sheets of small scattered massifs containing fissures, fissures, and grooves Deep trenches trap gases inside. Simultaneously, the compound [WS.Z4] appears as a hardened piece of plastic (similar to chocolate) with zigzags and scattered holes like volcanic holes and cracks of varying sizes. The surface also contained deep trenches that trap gases inside them.

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