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Preparation and characterization of new compounds for schiff base 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 Schiff bases from heterocyclic compounds using furfural by the traditional method (sublimation), where mono- and diamine compounds were used. The compounds were diagnosed using FT-IR, ¹H-NMR, ¹³C-NMR, UV-Vis Mass, and SEM to determine the shape of the molecule and whether it is nanoscale. The biological activity that showed moderate to high inhibitory ratios were also evaluated, and measured the molar electrical conductivity of some of them was.

Keywords---furfural, Schiff's bases, amic acids, pseudomonas putida, klebsiella pneumoniae, staphylococcus aureus, enterococcus.

Introduction

Furfural (a heterocyclic chemical) and its derivatives have been widely employed in industrial and applied areas (nematicides, fungicides, and insecticides), polymers, food and beverage taste enhancers, resins decolorizing agents, and transportation fuels. Aviation fuel stocks, gas line additives, lubricants, and pharmaceuticals are examples of this [1]. Its tensile strength and a high degree of transmission to glass make it suitable for the manufacture of compressed glass fibres exceeding its power (349 MPa), corrosion resistance, and as a catalyst in cellulosic kinin compounds, but it isn't strong when used as an adhesive; therefore, it is strengthened by adding it to epoxy resins, maleic anhydride, and ethylenediamine to improve its mechanics [2]. Furfural has been connected to several mono- and diamine compounds to generate Schiff bases, formed by simple

condensation of the azomethane group ($C=N$). Combining ketones or aldehydes with primary amines is among the most well-known and important organic chemical substances in organic production [3,4]. Amic acids are distinguished by the diversity of their compounds and the variety of application methods; this is due to their numerous advantages, whether in organic preparations for imides or isoimides or in polymer science, and it has also been used in pharmacology and the pharmaceutical industry [5]. Epilepsy [6,7] and arthritis [8] have been treated. Bacilli and nematodes are anti-tuberculosis [9]. It was also used in medical research [10]. Because of its great destruction capacity, it was also utilized as an insecticide and fungicide [11, 12]. And putting out fires and keeping flames from spreading [13]. Esters are organic molecules ($R-CO-OR$) carboxylic acid derivatives that may be saturated, unsaturated, or aromatic aliphatic. Because esters include a carbonyl group, they are polar and have a molecular weight similar to aldehydes and ketones; as a result, their boiling point is lower [14]. Many businesses employ esters because they are simple to make, whether cyclic or noncyclic. Furthermore, they are bases that protect the hydroxyl and carbonyl groups. Their production results in a category of natural products that are more abundant than others, such as sugars and peptides [15]. It may also be found in pharmaceuticals like Quadron, an anti-tumour treatment that contains methyl taxol, which is used to treat ovarian cancer [16] and breast cancer [17].

Experimental

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

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.

Preparation of derivatives of Schiff's bases [18]

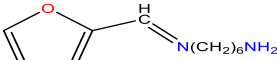
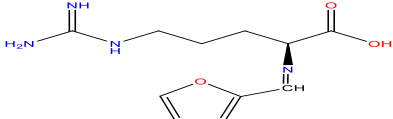
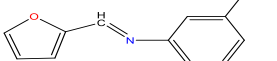
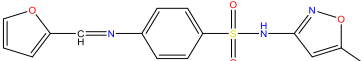
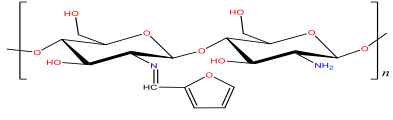
In a circular flask (250 ml) (0.01 mol) of amines dissolved in a specific solvent (according to the type of amine mentioned in the table below), the same number of moles of furfural disbanded in the same class have added. The solvent (for each amine) through a drop-by-drop separating funnel did not form a precipitate, where it was sublimated in a water bath at a temperature of (100°C) for a period of (3 hours), after which the mixture was placed in ice for some time. (30 minutes), the precipitate was filtered, and the filtrate was evaporated.

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

Comp. No.	Alcohol	Yield%	M.P $^\circ\text{C}$	Color	Recr. Solvent
WS.X1	Isopropyl	80	125-127	Dark Brown	Benzene
WS.X2	Cyclohexanol	77	Gum	Black	Acetic acid
WS.X3	Ethylene glycol	73	125-127	Dark Brown	Methanol

WS.X4	PVA	76	205-207	Off White	Ethanol
WS.X5	PVA	87	158-160	Light Yellow	Water

Table (2): Prepared Compounds

Comp. No.	Structure	Name
WS.X1		(Z)-6-((furan-2-ylmethylene)amino)hexan-1-amine
WS.X2		(S)-2-((furan-2-ylmethylene)amino)-5-guanidinopentanoic acid
WS.X3		(E)-3-((furan-2-ylmethylene)amino)benzoic acid
WS.X4		4-((furan-2-ylmethylene)amino)-N-(5-methylisoxazol-3-yl)benzenesulfonamide
WS.X5		(2R,3R,4R,5S,6R)-3-amino-5-(((2S,3R,4R,5S,6R)-3-(((E)-furan-2-ylmethylene)amino)-4-hydroxy-6-(hydroxymethyl)-5-methoxytetrahydro-2H-pyran-2-yl)oxy)-6-(hydroxymethyl)-2-methoxytetrahydro-2H-pyran-4-ol

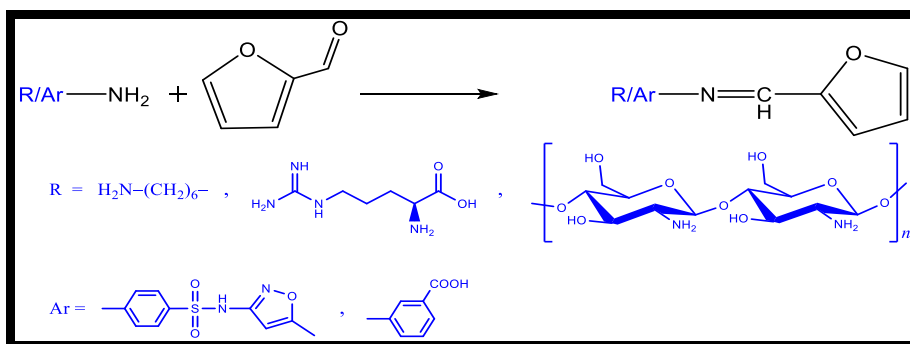
Biological efficacy assessment [19]

When testing the biological activity of certain prepared compounds, four types of pathogenic bacterial isolates were used: two Gram-negative (Gr-Ve) [*Pseudomonas putida* (P-P-), *Klebsiella Pneumoniae* (K-)], and two Gram-positive (Gr +Ve) [*Staphylococcus Aureus* (S.A+), *Enterococcus Faecalis* (E.F+)]. These bacteria were collected from the Department of Life Sciences laboratory at the College of Education for Pure Sciences. Mueller-Hinton agar was used as the culture medium. Mueller-Hinton Agar is a transparent nutrient medium with a yellow colour that is very useful in testing the sensitivity of microorganisms to antibiotics. It contains an animal infusion extracted from starch and casein, and it has a high ability to grow most microorganisms and microbes. It is widely used in measuring the biological effectiveness of antibiotics and chemicals with medical uses (MIC). The agar well diffusion technique (18) was used to examine the efficiency of the produced compounds on bacteria. Chemical solutions of the compounds were made [WS.X2, WS.X4, WS.X5.] a solvent (DMSO) and four concentrations of each chemical (10^{-5} , 10^{-4} , 10^{-3} , and 10^{-2} g/ml). The culture medium Müller-Hinton Agar was sterilized in a pressure oven (Autoclave) before being placed on Petri plates and allowed to harden. Then each dish was drilled with four holes and put in the incubator for 24 hours (24 hours). The findings were read at 37 degrees Celsius to indicate the sensitivity of the compounds utilized, which is determined by measuring the damping diameter surrounding

the dishes' visible holes. The size of the inhibition diameter for the typical antibiotics used is compared.

Results and Discussion

Schiff bases were prepared from the reaction of furfural with amines in a ratio of (1:1 mol), as shown in the following equation:



Spectroscopic interpretation (IR, ¹H-NMR, ¹³C-NMR, UV, Mass)

When studying the infrared spectrum of the compound [WS.X1], a binary absorption band of the (NH₂) group appeared at (3338 cm⁻¹) asymmetrical and (3205 cm⁻¹) symmetrically, a stretching beam of the aromatic (C-H) group appeared at (3032 cm⁻¹), a stretching beam of the (C=N) group appeared at (1578 cm⁻¹). A stretching band appeared at (1274,1234 cm⁻¹) belonging to the (C-O) group of the furan ring. The infrared spectrum of the compound [WS.X5] also showed a binary absorption beam of the (NH₂) group at (3384 cm⁻¹) asymmetrical and (3307 cm⁻¹) symmetrical. A stretching band appeared at (3076 cm⁻¹) belonging to the (C-H) aromatic group, a stretching band appeared at (1612 cm⁻¹) belonging to a group (C=N), and a stretching band appeared at (1258 cm⁻¹) belonging to (C-O) group of furan ring [20].

Table (3): Values of FT-IR spectrum in cm^{-1} units for compounds [WS.X1-WS.X5]

Comp. No.	IR (KBr) cm ⁻¹				
	vNH ₂ ass	vC-H alkyl	vC=C aromatic	vC-N	Others
	vNH ₂ sym	σNH ₂	vC=C aromatic	vC-O Furan Ring	
	vC-H aromatic	vC=N	σC-H alkyl		
WS-X1	3338	2928,2854	1562	1319	
	3205	1647	1481,1460	1274,1234	
	3032	1578	1394		
WS.X2	3571,3539	2985	1560	1313	vC=O carboxylic: 1704
	3417	1643	1497	1286,1271	
	3076	1627	1407		

					vC-O carboxylic: 1219
WS.X3	/	/	1542	1389	vC=O carboxylic: 1706 vC-O carboxylic: 1228
	/	/	1487,1457	1269	
	3087	1600	/		
WS.X4	3487	/	1581	1333	
	3378	1665	1465	1271	
	3087	1628	/		
WS.X5	3384	/	1560	1375	vOH alcohol: 3458-3028 vC-O alcohol: 1242
	3307	1652	1477,1431	1258	
	3076	1612	/		

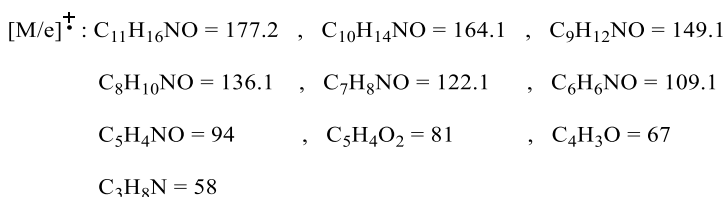
By studying the proton nuclear magnetic resonance spectrum ($^1\text{H-NMR}$) of the compound [WS.X1], we note that there is a signal at (ppm 8.11) that belongs to the group ($\text{HC} = \text{N}$), as we note Signals appear at (ppm 7.77, 6.85, 6.57) belong to the group ($\text{HC}=\text{CH}$) of the furan ring. The signals appear at (ppm 2.53 - 1.29) belong to groups (CH_2) aliphatic, and the signal appearing at (ppm 1.54) belongs to the (NH_2) group [21].

And the carbon nuclear magnetic resonance ($^{13}\text{C-NMR}$) spectrum of the same compound showed a signal at (ppm 159.03) that belongs to the group ($\text{HC} = \text{N}$). Signals at (ppm 150.01, 145.50, 114.18, 112.28) belong to the carbon groups in the furan ring, while the signals at (ppm 61.10, 30.89, 27.01) belong to the aliphatic (CH_2) groups [22].

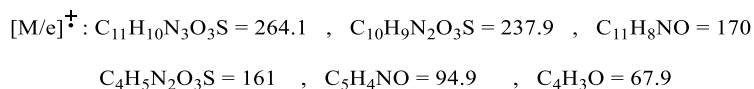
The proton nuclear magnetic resonance spectrum ($^1\text{H-NMR}$) of the compound [WS.X4] also showed a signal at (8.42 ppm) belonging to the group ($\text{HC}=\text{N}$). Signals at (ppm 7.84, 6.73, 6.56) belong to the ($\text{HC}=\text{CH}$) group of the furan ring, and the signals at (ppm 7.83, 7.38) belong to the aromatic system of the benzene ring, and the signal at (ppm 11.42) belongs to the (NH) group [23].

When performing the UV spectroscopy of the Schiff bases, we observed the appearance of an absorption band ($\pi \rightarrow \pi^*$) with a small wavelength and greater intensity; Because there are pathologically shifted ($\text{C}=\text{C}$) bonds of the derivatives of Schiff bases within the range (238 nm). Also, an absorption band ($n \rightarrow \pi^*$) with a large wavelength and less intensity appeared because of unshared electron pairs on the oxygen and nitrogen atoms and the bathochromic displacement of the presence of oxo and cascade groups within the range (402 nm) [24].

By mass spectrometry, it was found that the molecular weight of the compound [WS.X1] is (194.2), and the calculated molecular weight of the compound itself is (194.28) [25].



The mass spectrometry also found that the molecular weight of the compound [WS.X4] is (331.1), and the calculated molecular weight of the compound itself is (331.35).



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 solution increases, while the degree of conductivity in the complex that does not ionize is low [26].

Table (4): The molar electrical conductivity values of the compounds [WS.X1, WS.X4] in a solvent (DMSO) at a concentration (10^{-3}) molar

Test Comp.	Conductivity	T/°C
WS.X1	4.9	20.9
WS.X4	8.8	21.8

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.X1, WS.X4] [24], as shown in the table below:

Table (5): Elemental analysis statement (C.H.N) for compounds [WS.X1, WS.X4]

Comp.	Found & Cal.	Carbon%	Hydrogen%	Nitrogen%
WS.X1	Found	67.85816956	9.379836082	14.84932041
	Calculate	68.003757	9.337616	14.419086
WS.X4	Found	53.66306686	4.222303867	12.44227409
	Calculate	54.371661	3.954103	12.681485

Scanning Electron Microscopy (SEM) Analysis

The effect of laser bombardment by scanning electron microscopy (SEM) of the two compounds [WS.X1, WS.X4], the compound [WS.X1] appears at (10 μ m) as a cut-off from the rocks are interspersed with deep gaps and surfaces with cracks and fissures on the rocky massif with cavities and trenches that trap gases inside.

At the same distance, the compound [WS.X4] appears in the form of rock masses with voids that trap gases within them. At (1 μm) and (500 nm) [WS.X1] appear like an iceberg with hollows, grooves and trenches containing small blocks of ice (or cotton-like) interspersed with channels: deep and voids. The compound [WS.X4] appears in the exact two dimensions in the form of tiny rock fragments containing deep trenches scattered over a large rock mass with cracks, fissures and deep grooves. At (200 nm), [WS.X1] appears as a porous cotton streak scattered on a mountain with deep trenches with the appearance of particle dimensions. At the same distance, the compound [WS.X4] appears in the form of scattered pieces of rock with the presence of deep trenches and the appearance of nanoparticles that prove that the molecule possesses different characteristics from one before it. As if it is connected to electric current.

Biological activity

Through the biological activity of some of the prepared compounds, it was found that most of these compounds have antibacterial activity in comparison with some pre-manufactured antibiotics for these types of bacteria, such as Ampicillin, Cefixime and Ceftriaxone. which are highly effective antibacterial that is 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.X1, WS.X4] 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 [27]. The results shown in Table (6) below indicate that most prepared compounds can inhibit bacteria through different concentrations (10⁻⁵, 10⁻⁴, 10⁻³, 10⁻²). g/ml, as the diameter of the damping ranges between (0-20 mm). Figures (30 - 23) show that the compound [WS.X1] increased the inhibition values against all types of bacteria; This is because the compound is aliphatic, and despite the decrease in the basicity, there was an increase in the inhibition values; This is due to the presence of (CH₂) groups of electrons. For compound [WS.X4], the highest braking rate was obtained; This appears to be due to atoms of heterogeneous groups such as (N, S, O) despite the low basicity [28].

Table (6): The inhibitory activity of the compounds [WS.X1, WS.X4] on positive and negative bacteria (inhibition diameter measured in mm)

Comp.	C (g/ml)	K –	P.P –	S.A +	E.F +
WS.X1	10 ⁻²	0	6	18	8
A	10 ⁻³	0	2	14	6
B	10 ⁻⁴	0	2	18	2
C	10 ⁻⁵	0	2	16	2
K/d		d=0	K=26	K=0	K=0
WS.X4	10 ⁻²	36	36	60	36
A	10 ⁻³	20	2	30	26
B	10 ⁻⁴	0	0	24	14



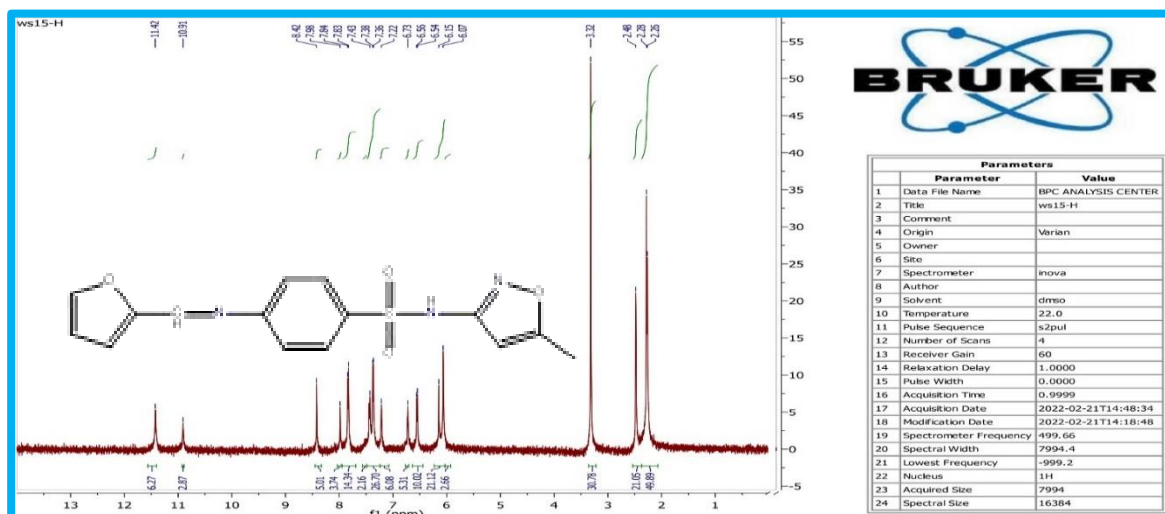
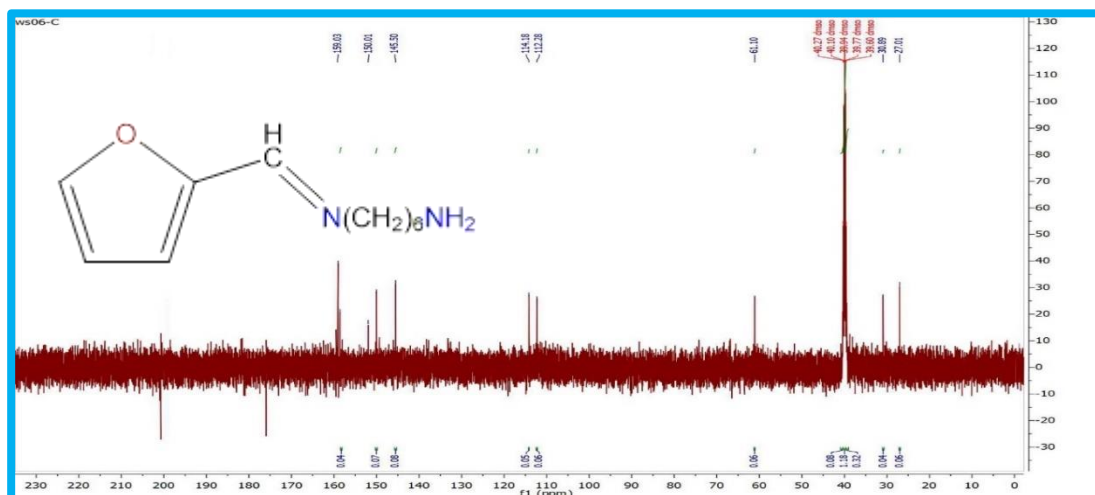
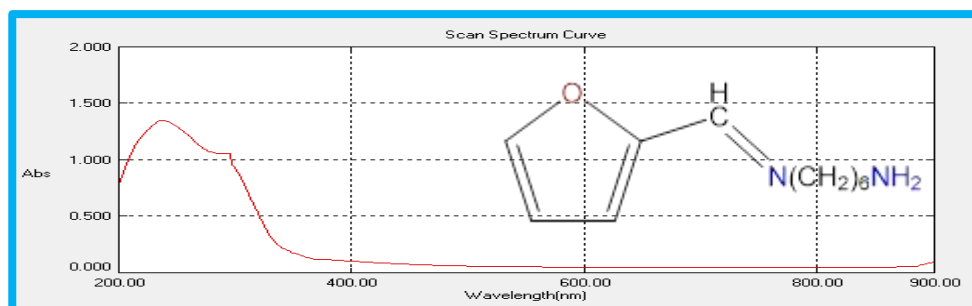
Figure 3: ¹H-NMR spectrum of WS.X4Figure 4: ¹³C-NMR spectrum of WS.X1

Figure 5: UV-Visible spectrum of WS.X1

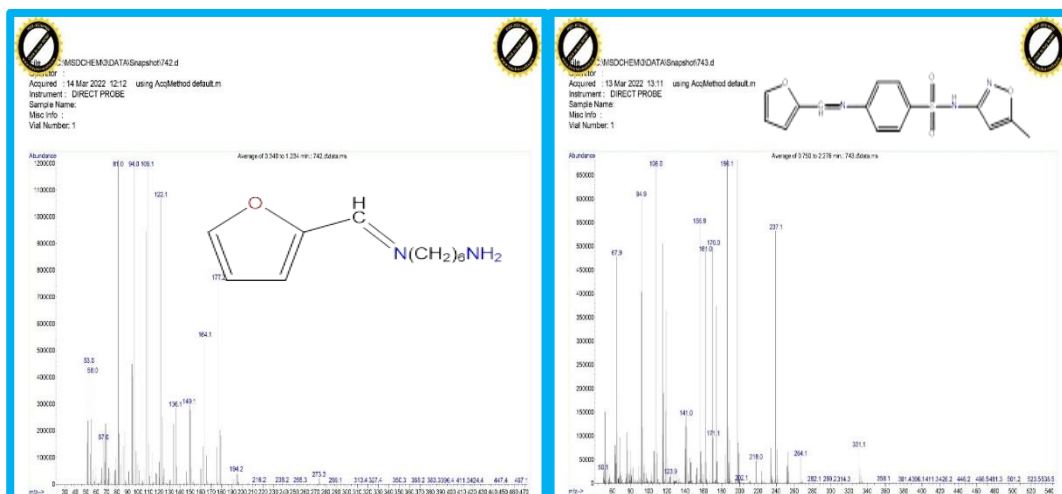


Figure 6: Mass spectrum of WS.X1 and WS.X4

Eager 300 Summarize Results							Eager 300 Summarize Results						
Date : 2002/01/03 at 00:09:07							Date : 2002/01/03 at 00:09:38						
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		
WS06							WS15						
#	Group	Sample Name	Type	Wt.	Pro.F	---	#	Group	Sample Name	Type	Wt.	Pro.F	---
49	2	WS06	UNK	3.333	6.25	---	50	3	WS15	UNK	3.162	6.25	---
Component name		Element %					Component name		Element %				
Nitrogen%		14.84932041					Nitrogen%		12.44227409				
Carbon%		67.85816956					Carbon%		53.66306686				
Hydrogen%		9.379836082					Hydrogen%		4.222383867				
Sulphur%		0					Sulphur%		0				
1 Sample(s) in Group No : 2							1 Sample(s) in Group No : 3						
Component Name		Average		Std. Dev.		Variance	Component Name		Average		Std. Dev.		Variance
Nitrogen%		14.84932041		0		0.0000	Nitrogen%		12.44227409		0		0.0000
Carbon%		67.85816956		0		0.0000	Carbon%		53.66306686		0		0.0000
Hydrogen%		9.379836082		0		0.0000	Hydrogen%		4.222383867		0		0.0000
Sulphur%		0		0		0.0000	Sulphur%		0		0		0.0000

Figure 7: C.H.N. of WS.X1 and WS.X4

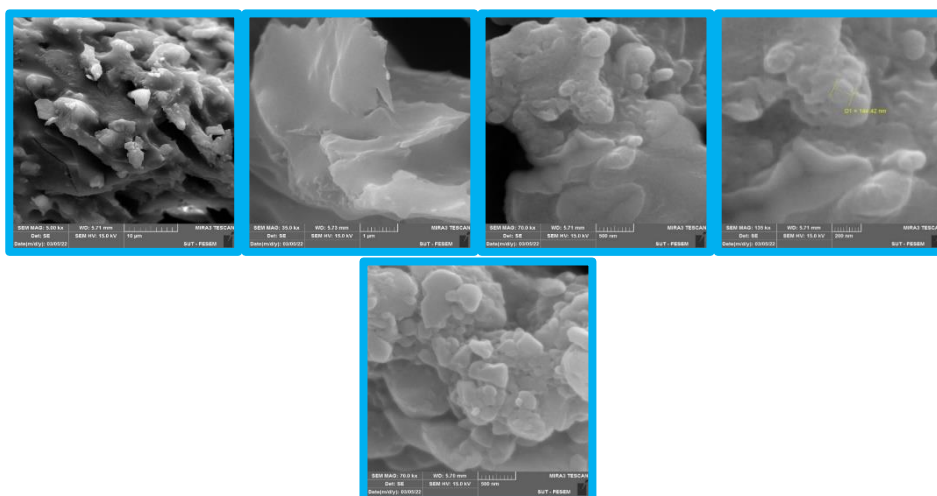


Figure 8: SEM of WS.X1

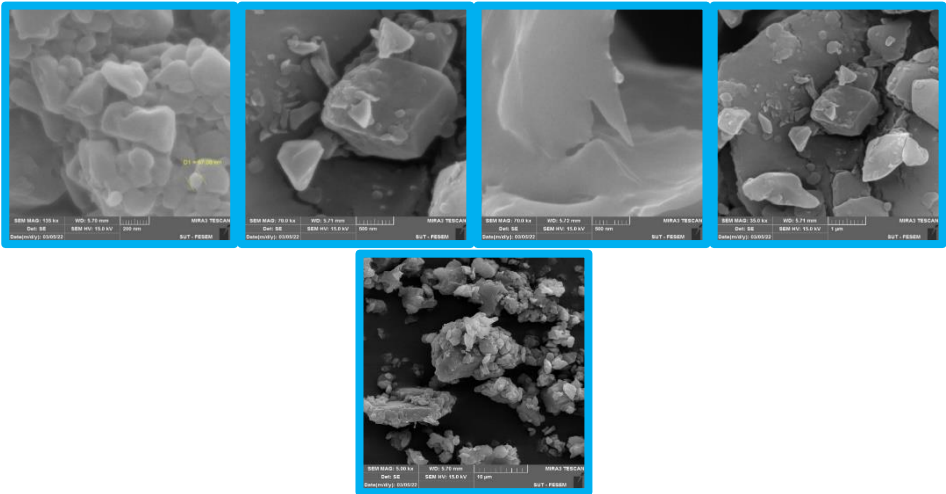


Figure 9: SEM of WS.X4

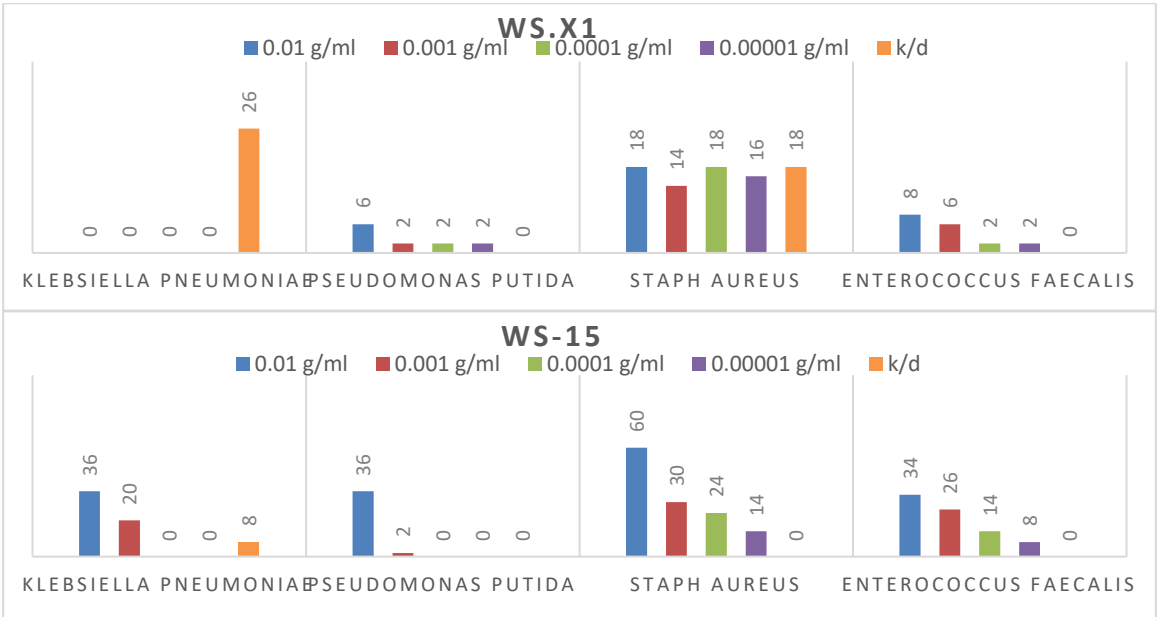


Figure 10: Inhibitory activity of (WS.X1, WS.X4) on the four types of bacteria

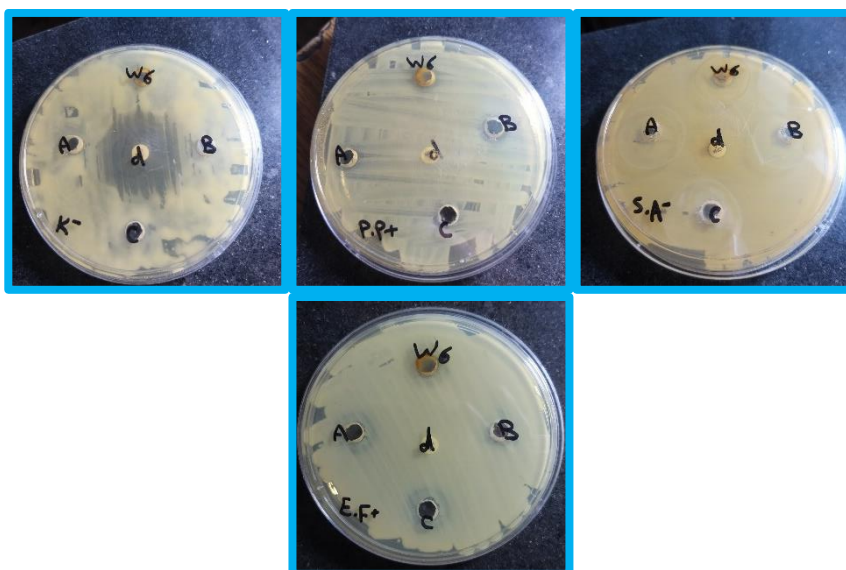


Figure 13: Compound WS.X1 inhibits the growth of on the four types of bacteria

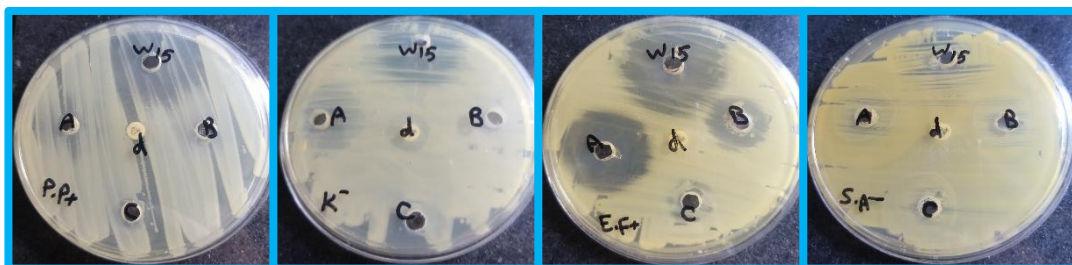


Figure 11: Compound WS.X4 inhibits the growth of on the four types of bacteria

Conclusions

Physical and spectroscopic measurements validated the produced compounds' correctness and validity. As a result, the preparation procedures were effective, efficient, and low-cost. The microanalysis results of the components of the produced compounds were exactly or almost equivalent to the estimated proportion. The synthesized chemicals were also effective against the microorganisms employed in the experiment. The synthesized compounds were also found to have high electrical conductivity. The impact of laser bombardment on the compounds [WS.Z1, WS.Z4] was also shown using a scanning electron microscope (SEM). Compound [WS.Z1] appears at (10 m) as sheets of tiny distributed massifs with fissures and grooves. Gases are trapped within deep holes. WS.Z4 looks like a hardened piece of plastic with zigzags and dispersed holes that resemble volcanic craters and fractures of various sizes. There were also deep holes on the surface that trapped gases.

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