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Synthesis, Characterization and Study of Biological Activity of Some New Schiff Bases

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Abstract

In the present study, Schiff's bases (6a - 6e) and new amids derivatives (6f - 6j) have been synthesized. The glutaroyl chloride (2) has been prepared by the reacting of glutaric acid and thionyl chloride in presence of (DMF). The new compound bis (4-formylphenyl) glutarate (3) has been synthesized from reaction of one mole of glutaroyl chloride and two moles of 4-hydroxybenzaldehyde. Compound 4,4'-(glutaroylbis(oxy))dibenzoic acid (4) was synthesized from one mole of glutaroyl chloride and two moles of 4-hydroxybenzoic acid, while compound bis(4-(chlorocarbonyl)phenyl) glutarate (5) was prepared from 4,4'-(glutaroylbis(oxy))dibenzoic acid and Thionyl chloride . Then Schiff's bases (6a - 6e) that prepared from the reaction of compound no: 3 and derivatives amine in EtOH. Amids (6f - 6j) were synthesized from the reaction of previous acid chloride (5) and derivatives amine in py, and DMF. All the prepared compounds have been characterized as usual by the spectral methods (which including FTIR, ^{1}HNMR (6a, 6d and 6i) besides TLC. Techniques and the melting points were recorded. Then the antibacterial studies were checked against two types of bacterial. We discovered that the compounds 6a, 6d, 6h, and 6i showed high growth against all

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bacteria of *E_ coli* and *staphylococcus aureus* basides *psudomonas aeruginosa*.

Keywords: glutaric acid, Schiff bases, bis(4-formylphenyl) glutarate , bis(4-(chlorocarbonyl)phenyl) glutarate.

Introduction

Heterocyclic compounds are extremely prevalent in both natural and synthetic molecules, and they are crucial in both the pharmacological and synthetic fields [1]. Many compounds including vitamins and amino acids which can be used for drug synthesis all have heterocyclic ring. Schiff bases are molecules with an azomethine group (-C=N-). Moreover, the Biological activity which belongs to them includes anti_bacterial, anti_fungal and anti-cancer properties, they have considerable applications in polymer chemistry and the pharmaceutical industry [2]. Amides hold significant importance as functional groups within the realm of organic chemistry, mostly owing to their involvement in many biological processes such as proteins and peptides[3]. Additionally, their presence may be observed in a wide range of bioactive compounds, further emphasizing their significance. The bioactivity and uses of these molecules are extensive, encompassing several fields such as medicines[4], insecticides[5], polymers, and adhesives. [6].

Experimental

The compounds utilized in this study were procured from commercial suppliers and underwent purification through distillation or recrystallization prior to their application. The points of melting of the substances have been determined which are using an electrothermal (Gallen Kamp) apparatus with open capillary tubes. No corrections were made to the obtained values. The all reactions was conducted as a standard practice, and the assessment of purity was performed with thin-layer chromatography using aluminum plates covered with a substance. The visualization of spots on the plates was achieved by subjecting them to iodine vapors after they had dried. The ^{1}HNMR spectra were obtained utilizing Bruker's 500 FT MHz NMR equipment in Iran. Dimethyl sulfoxide (DMSO) was utilized as the solvent, while tetramethylsilane (TMS) served as the reference for chemical shifts that expressed in parts per million (ppm). The infrared spectra were obtained using a Shimadzu FTIR-8400S spectrophotometer located in Iraq.

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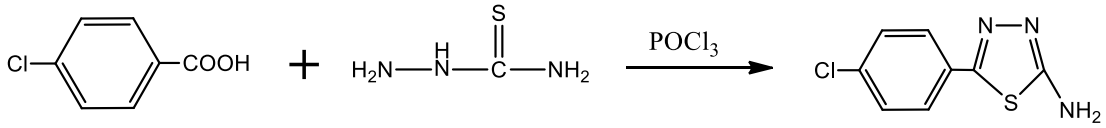
1. Synthesis of 5_(4_chlorophenyl) -1,3,4- thiadiazol-2-amine(1)

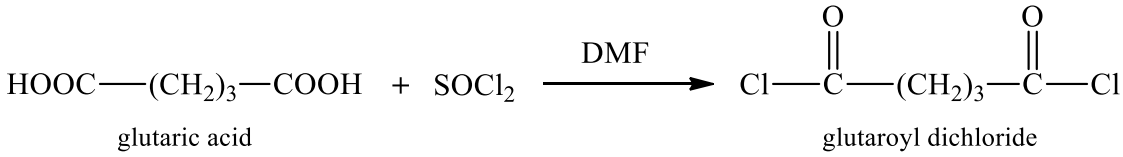
A reaction was conducted using 4-chlorobenzoic acid (3.14 grams, 0.02 moles) and thiosemicarbazide (1.82 grams, 0.02 moles) in phosphorus oxychloride (10 milliliters, 98% purity). The mixture was refluxed for a duration of 3 hours[7]. After cooling, water has been used and added, an additional time the mixture was refluxed (1 hour). Subsequent to the cooling process and subsequent neutralization of the mixture using sodium hydroxide, a solid substance was obtained. This solid was then subjected to filtration, followed by thorough washing with water. Finally, the solid was recrystallized, resulting in the formation of the ultimate product, which possesses a melting point (m.p.). (202-205 ^{0}C),while the yield is (60%).[8]



Equation 1: Synthesis pathway for the compounds (1).

2. Synthesis of acid chloride (2).

(glutaric acid) (5 g, 0.0378 mol), was mixed with (12 ml, 0.0378 mol) of thionyl chloride with a very less drops of (DMF) and refluxed for 2 hours. Coold and the relevant solvent was evaporated keept dry [9].



Equation 2: Synthesis pathway for compound (2).

3. Synthesis of bis(4-formylphenyl) glutarate (3):-

In an ice bath, 4-hydroxybenzaldehyde (3.036 g, 0.022 mol) has dissolved or fluxed in THF (15 ml) with a few drops of (DMF), and glutaroyl dichloride (2 g 0.011 mol) has added. The mixture was stirred continuously at 30 ºC for 24 hours. After that, the mix has put into acidic iced water to remove the pyridine residue before being washed with distilled water [10].

4. Synthesis of ester derivatives (4):-

In dry pyridine (15 ml) with a few drops of (DMF), ***p***-hydroxybenzoic acid (0.002 mol) was dissolved. This solution then received glutaroyl dichloride (0.001 mol) in an ice bath. Then mix was swirled continuously for 24 hours in the room of temperature. To eliminate the remaining pyridine, the reaction liquid was dumped into acidic iced water once the reaction was complete, and it was then washed with distilled water [11].

5. Synthesis of acid chloride (5).

A mixture of (1.17 g, 0.003 mol) of (4,4'-(glutaroylbis(oxy))dibenzoic acid, (12 ml , 0.003 mol) of thionyl chloride, and a few drops of (DMF) were added then the mix has refluxed into two hours. The pertinent solvent evaporated while being kept dry, and it was cooled [9].

6. Synthesis of Schiff bases derivatives (6a – 6e).

The aniline derivative was dissolved in 5 mL of absolute ethanol and then combined with bis(4-formylphenyl) glutarate (EG1) (0.1 g, 0.00029 mol) dissolved in 15 mL of absolute ethanol. Subsequently, the combination underwent reflux for a duration of 4 hours. Following the reaction's completion and the mixture was subjected to cooling. The resulting solid was subsequently purified by a series of steps. Firstly, it was assessed using thin-layer chromatography (TLC) using ethanol and benzene (50: 50) as the solvent. Subsequently, the solid was filtered and rinsed with distilled water. Finally, recrystallization was carried out using absolute ethanol. [12**,**13].

7. Synthesis of Acetamides Derivatives (6f-6j)

In dry pyridine (15 ml), an aniline derivative (0.002 mol) was dissolved with a few drops of (DMF), and 0.001 mol of bis(4-(chlorocarbonyl)phenyl) glutarate was added to this solution in in the bath of ice. The mixture was stirred continuously for 24 hours at room temperature. To

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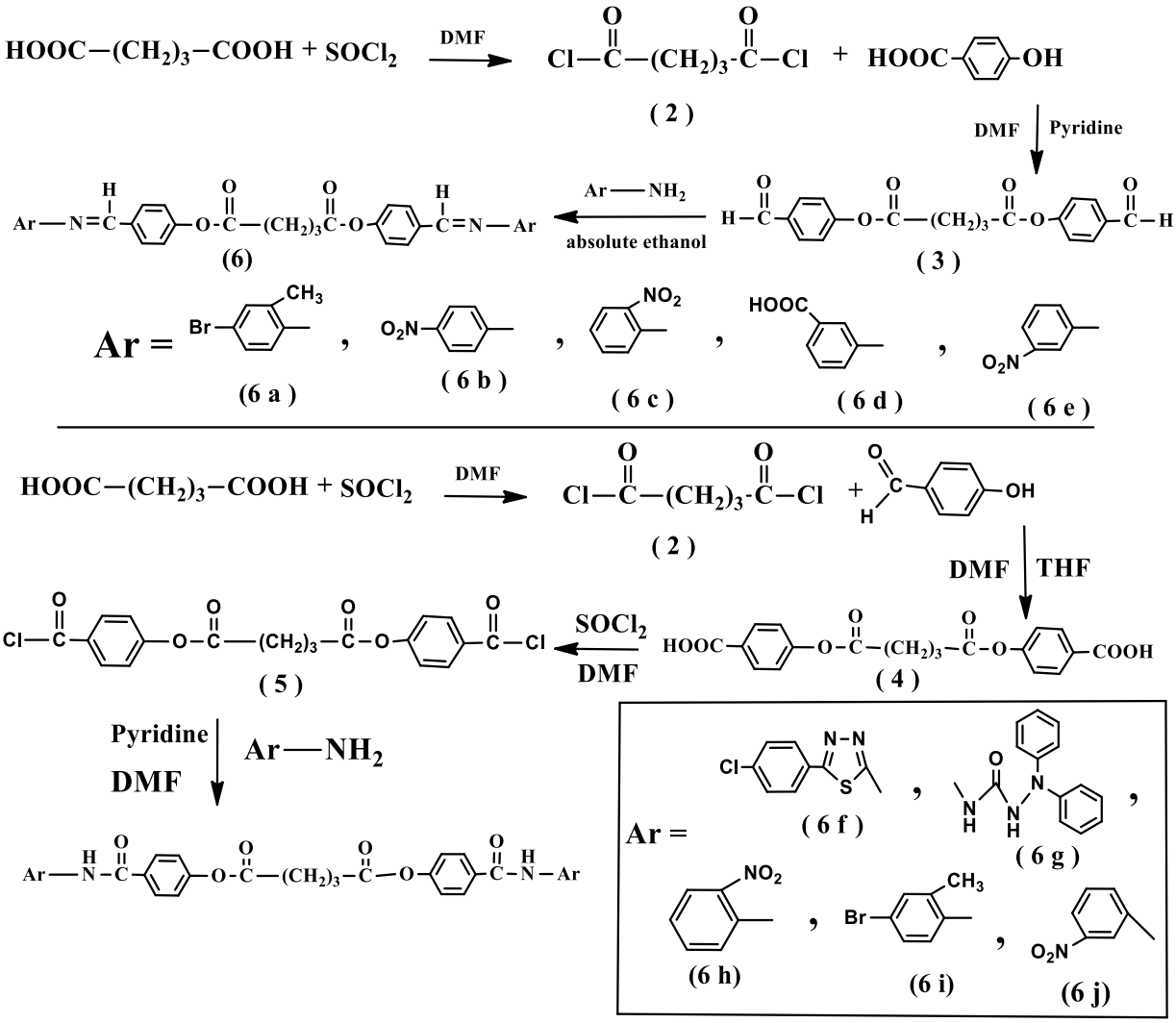
eliminate the remaining pyridine, the mixture's reaction was put into acidic iced water once the reaction was completed. It was then washed with distilled water [11].



Scheme 1: Synthesis pathway for the compound (2 – 6j)

Results and Discussion

Table 1 displays the characteristics of physical properties of Schiff base and newly synthesized amide derivatives. The compounds exhibit a high degree of stability when exposed to dry air and demonstrate solubility in a wide range of organic solvents. The pathways of synthesis is leading to the desired molecules are outlined in Scheme 1.The

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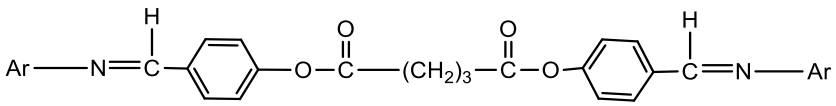
determination of the chemical contents of these structure were established by the utilization the points of melting along with data of spectra.

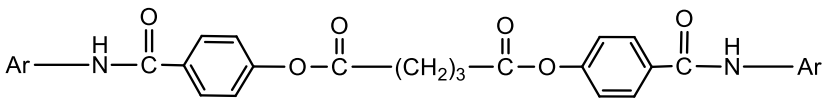
Table 1: The properties of physical and synthesized compounds.

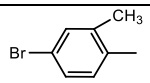
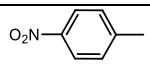
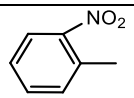
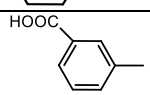
Comp: no.	Nomenclature	Formula Molecular M.Wt g/mol	Colour	M.P C^0	Yield %	Rf
2-	bis(4-formylphenyl) glutarate	$C_{19}H_{16}O_6$ 340.33	Brawn	91 – 93	50	0.77
3-	4,4'-(glutaroylbis(oxy))dibenzoic acid	$C_{19}H_{16}O_8$ 372.33	Dark Red	>250	60	0.82

Compound structure (6a – 6e)



Compound structure (6f – 6j)



Comp. No.	Ar	Nomenclature	Molecular Formula M.Wt g/mol	Colour	M.P C^0	Yield %	Rf
6a -		bis(4-(((4-bromo-2-methylphenyl)imino)methyl)phenyl) glutarate	$C_{33}H_{28}Br_2N_2O_4$ 676	Black	140 – 145	60	0.87
6b-		bis(4-(((4-nitrophenyl)imino)methyl)phenyl) glutarate	$C_{31}H_{24}N_4O_8$ 580	Yellow	> 250	58	0.90
6c-		bis(4-(((2-nitrophenyl)imino)methyl)phenyl) glutarate	$C_{31}H_{24}N_4O_8$ 580	Brown	77 – 85	60	0.57
6d-		3,3'-((((glutaroylbis(oxy))-bis(4,1-phenylene))bis-(methanylylidene))bis(azanylylidene))dibenzoic acid	$C_{33}H_{26}N_2O_8$ 578	Dim Gray	81 – 120	69	0.90

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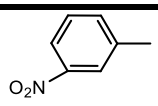
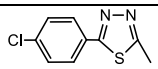
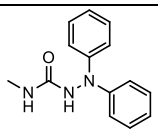
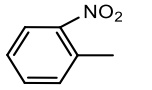
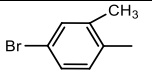
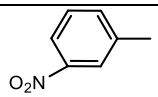
6e-		**bis(4-(((3-nitrophenyl)imino)methyl)phenyl) glutarate**	**$C_{31}H_{24}N_4O_8$ 580**	**Deep green**	**73-93**	**73**	**0.88**
6f-		**bis(4)(5)(4) chlorophenyl)(1,3,4) thiadiazol-2-yl)carbamoyl)phenyl) glutarate**	**$C_{35}H_{24}Cl_2N_6O_6S_2$ 759**	**Black**	**133 – 145**	**52**	**0.86**
6g-		**bis(4-(2-(2,2-diphenylhydrazinecarbonyl)hydrazinecarbonyl)phenyl) glutarate**	**$C_{45}H_{41}N_8O_8$ 821**	**Dark red**	**100 – 115**	**57**	**0.93**
6h-		**bis(4-((2-nitrophenyl)carbamoyl)phenyl) glutarate**	**$C_{31}H_{24}N_4O_{10}$ 612**	**Black**	**190 –214**	**50**	**0.6**
6i-		**bis(4-((4-bromo-2-methylphenyl)carbamoyl)phenyl) glutarate**	**$C_{33}H_{28}Br_2N_2O_6$ 708**	**Brown**	**160 – 180**	**60**	**0.8**
6j-		**bis(4-((3-nitrophenyl)carbamoyl)phenyl) glutarate**	**$C_{31}H_{24}N_4O_{10}$ 612**	**Brown**	**165 - 190**	**47**	**0.7**

1. FT-IR Spectra

All the substances which belong to the infrared spectra included in this study were obtained by using solid state that indicates in the disk of KBr method. Data of FTIR spectra prepared the compounds which belong to table no : 2. The presence of Schiff base compounds (6a –6e) was confirmed by analyzing their infrared spectra, which revealed the characteristic stretching band corresponding to the azomethine [CH=N] functional group from (1674 to 1612) cm^{-1} [14]. This band in region shows the disappear of FTIR absorption band in region (3200-3400) cm^{-1} corresponding to NH_2 group and at (1710) cm^{-1} for (C=O) group of bis(4-formylphenyl) glutarate (3) [15]. While Amids derivatives (5f-5j) confirmed by the appearance of FTIR absorption band in the region(1600-1670) cm^{-1} according to (-C=O-) amid group[16] and appearance of FTIR in the region absorption band (3200-3400) cm^{-1} due to (–N–H) , with the disappear of FTIR in the region of absorption band à (3200-3400) cm^{-1} corresponding to (NH_2) group and (550 – 800) cm^{-1} for (C–Cl) group of acid chlorides [17].

2. ^{1}HNMR spectra:

^{1}HNMR is related to the base of Schiff which derivatives (6a)shows the appearance of the signals at δ (1.1-2.81) ppm, δ (6.95-8.53) ppm, δ(9.80) ppm that belongs to $CH_2CH_2CH_2$ protons,

aromatic protons, N=C-H protons, respectively[18]. Then Schiff base derivative (6d) showed the appearance of the signals at δ(0.9-2.97)ppm, (6.80-8.52) ppm,δ (9.80) ppm, δ(10.12) ppm that belongs to $CH_2CH_2CH_2$ protons and protons of aromatic then (N=C-H) protons, COOH protons, respectively[19], While amids derivatives (6i) confirmed by the appearance of the signals at δ(1.10-3.16) ppm, δ(6.87-8.03) ppm, δ(8.58) ppm that belongs to $CH_2CH_2CH_2$ protons, protons of aromatics , (H-N-C=O) protons, respectively[20].

3. Antibacterial activity:

This study examines the impact of some synthesized chemicals on bacterial growth, specifically *The Eschershia coli, Psudomonas aeruginosa, Staphylococcus* aureus. [21]:

The activity of antibacterial of the synthesized compounds were investigated then the findings indicated that several compounds exhibited notable antibacterial efficacy. The findings are presented in number 3 table.

Table 2: Major bands of FTIR absorption (cm^{-1}) of the compounds of synthesis

Comp. no.	IR(KBr),cm^{-1}							
	V =C-H Aromatic	V -C-H Aliphatic	VC =N	C=C V Ar.	C=O V	V C-N	C-H V Out of plane	Others
2	3066	2943 2890	-	1485 1600	1732 1685	-	744 833	C-H (aldehyde) V (2825)
3	3078	2943 2843	-	1508 1604	1751 1691	-	763 860	---------
6a	3039	2974 2858	1627	1504 1581	1755	1261	775 867	Br_2 V (659)
6b	3078	2979 2866	1631	1504 1597	1751	1303	752 837	NO_2 V asy(1515) Vsy(1396)
6c	3074	2958 2862	1623	1500 1597	1759	1280	725 837	NO_2 V asy(1510) Vsy(1346)
6d	3066	2926 2875	1625	1500 1573	1700	1270	733 845	OH V (3350)

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6e	3071	2944 2868	1627	1489 1519	1740	1261	732 867	NO_2 V asy(1588) Vsy(1350)
6f	3012	2916 2812	-	1508 1593	1728 1693	1257	759 887	N-H V (3375) C-Cl V (825)
6g	3059	2966 2854	-	1496 1597	1728 1651	1242	725 848	N-H V (3259)
6h	3074	2931 2873	-	1500 1600	1735 1627	1259	756 860	N-H V (3325) NO_2 V asy(1588) Vsy(1342)
6i	3020	2970 2885	-	1489 1600	1732 1639	1257	752 867	N-H V (3294) C-Br V (813)
6j	3074	2943 2873	-	1504 1600	1735 1627	1257	736 883	N-H V (3320) NO_2 V asy(1527) Vsy(1342)

.**Table 3:** In *vitro* antibacterial activity substituted Schiff bases, 6a, d and acetamide 6 h, i .

Comp. No.	Sample No. (In image)	*Staphylococci Aurues*	*Eschershia Coli*	*Psudomonas Aeruginosa*
6 a	48.H	++	++	+
6 d	59.H	++	++	++
6 h	36.O	++	++	++
6 I	37.H	++	++	+

Symbols of the key

When the activity is high= +++ (the zone of inhibition > 20 mm). The activity is moderate = ++ (the zone of inhibition 11-20 mm). The activity is slight = + (the zone of inhibition 5-10 mm). Totally inactive= – (the zone of inhibition< 5 mm).

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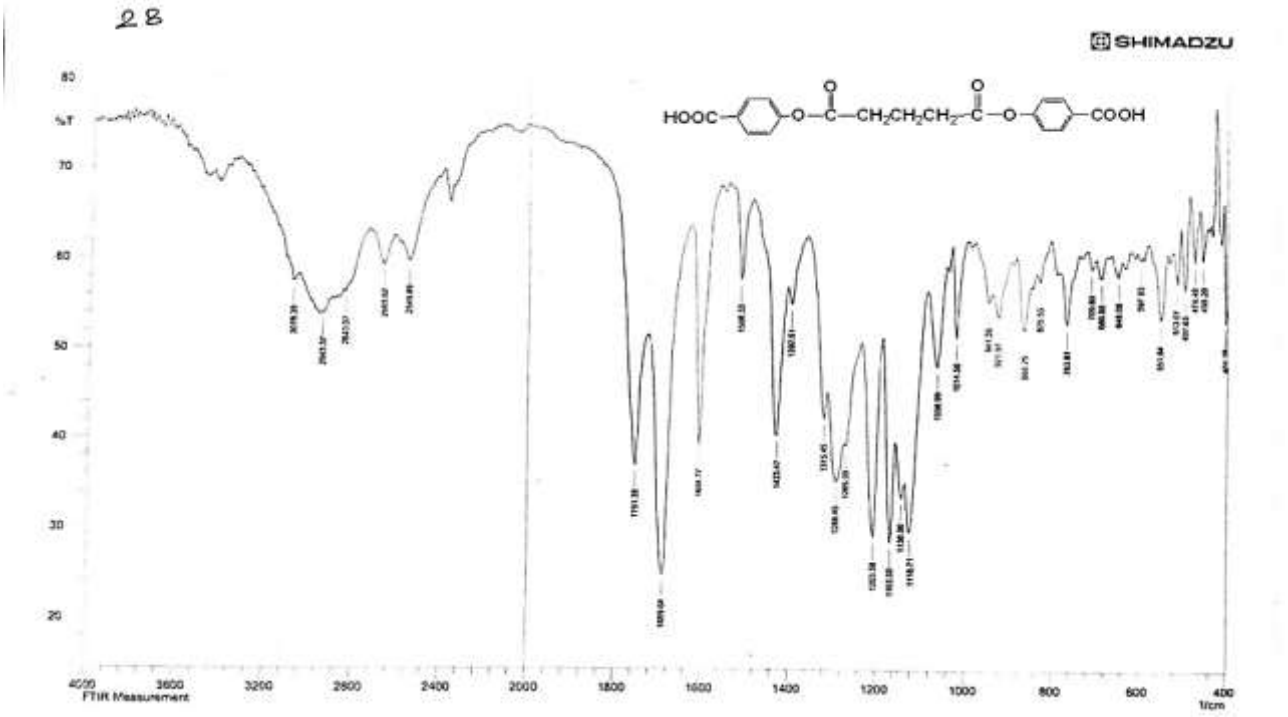

Figure 1: FTIR spectrum for compound (4)

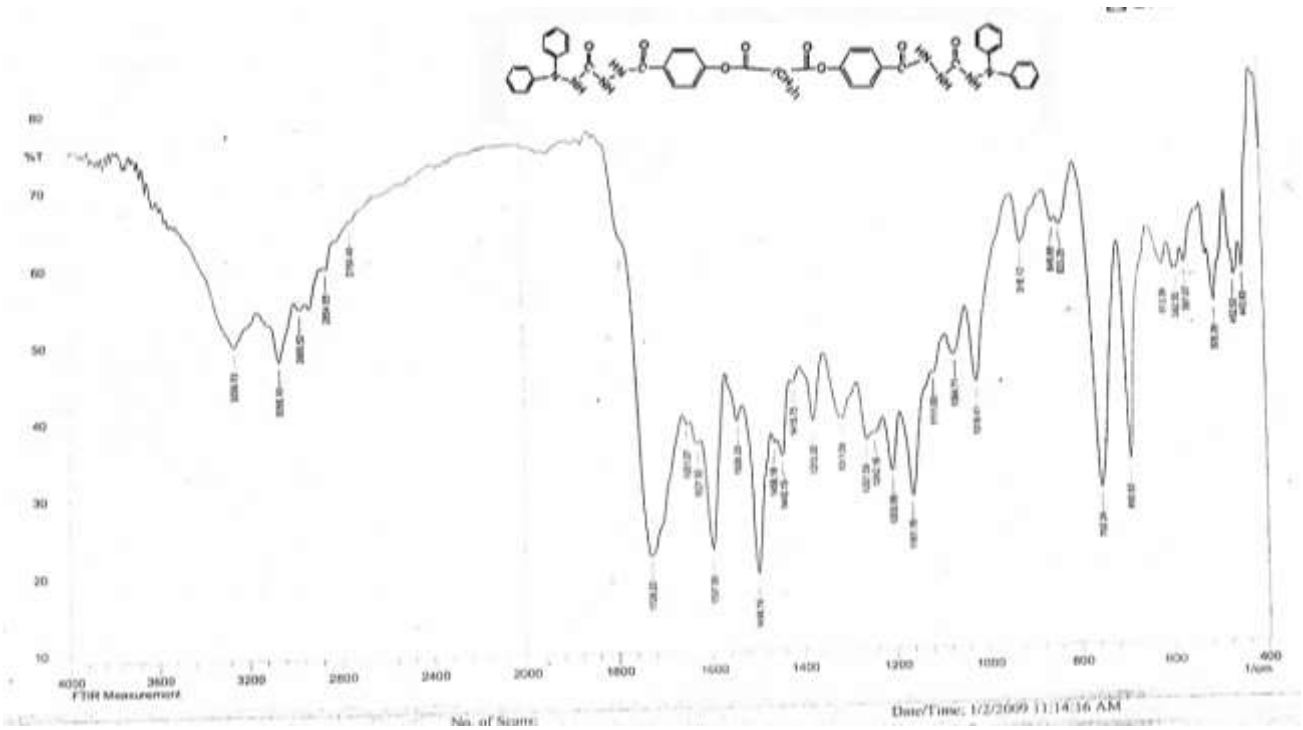

Figure 2: FTIR spectrum for compound (6 g)

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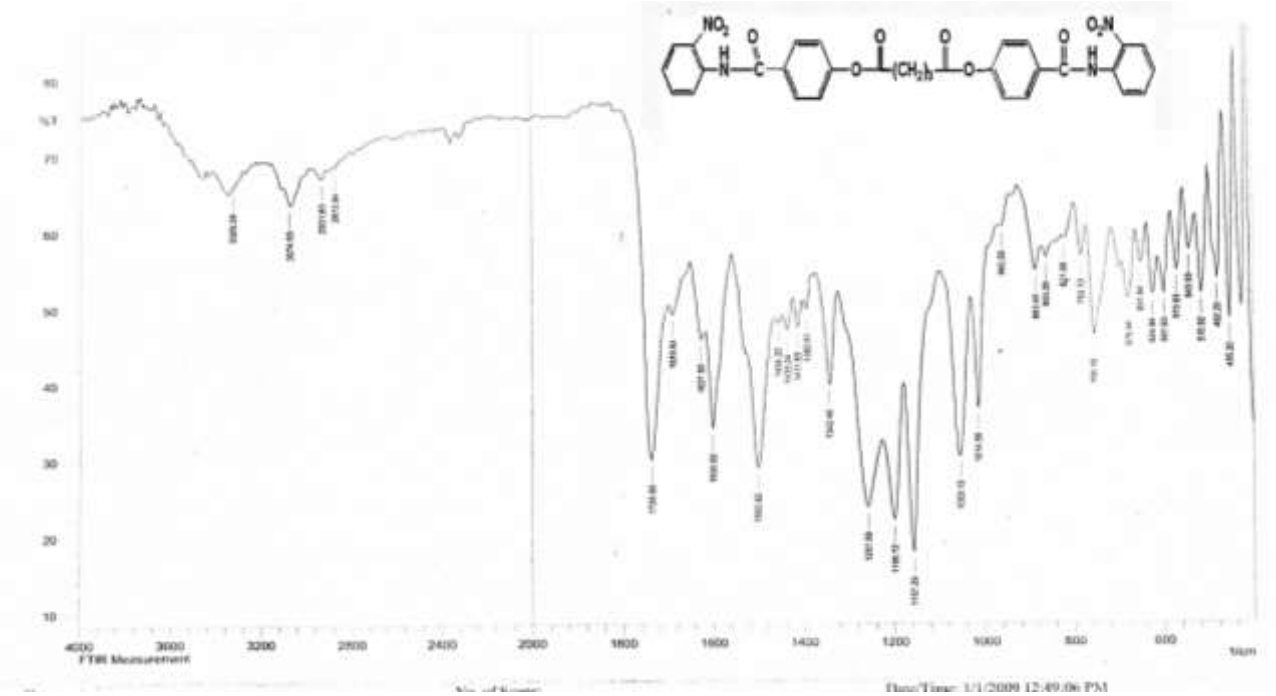

Figure 3: FTIR spectrum for compound (6 h)

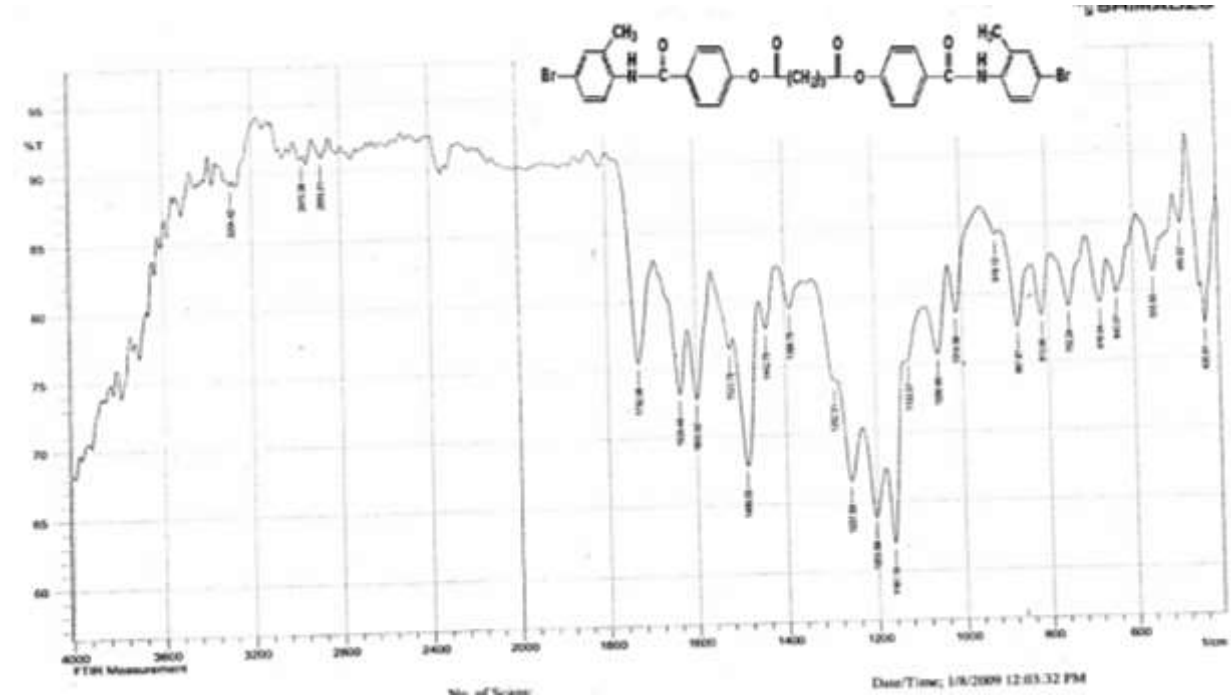

Figure 4: FTIR spectrum for compound (6 i)

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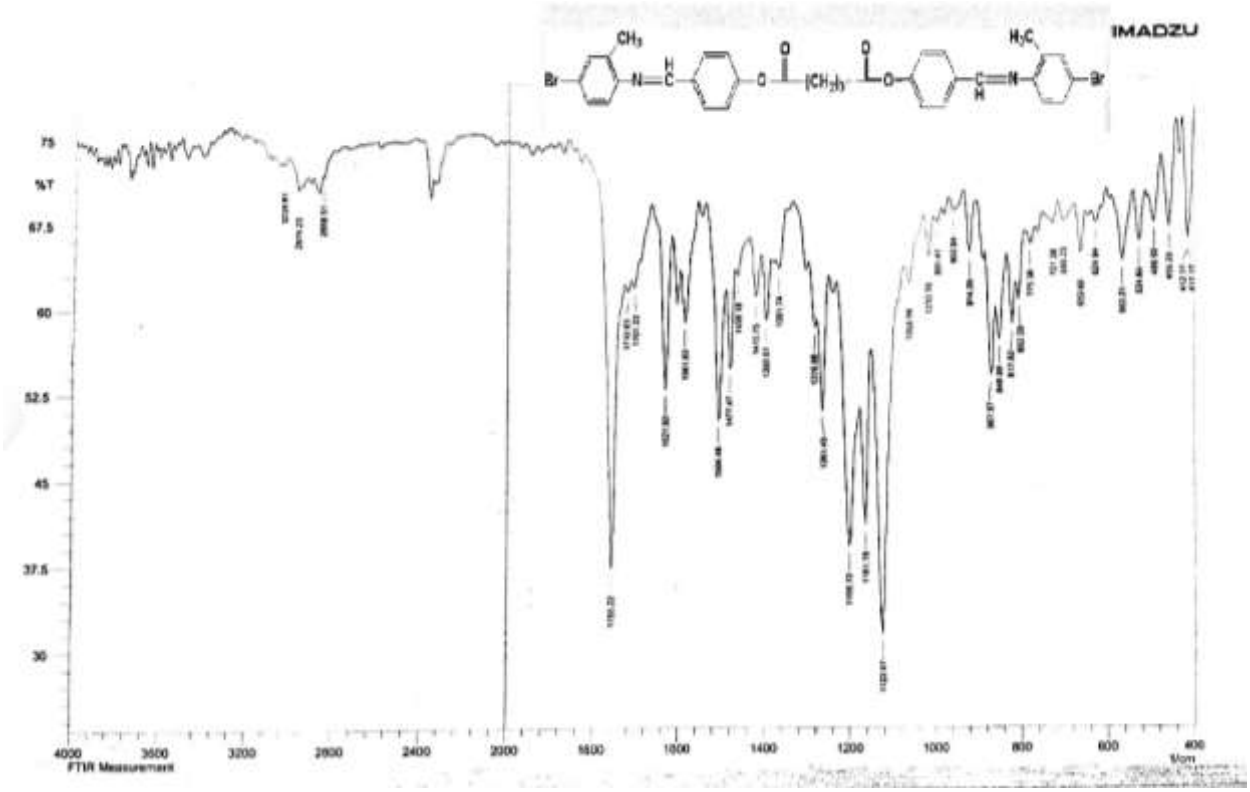

Figure 5: FTIR Spectrum for compound (6 a)

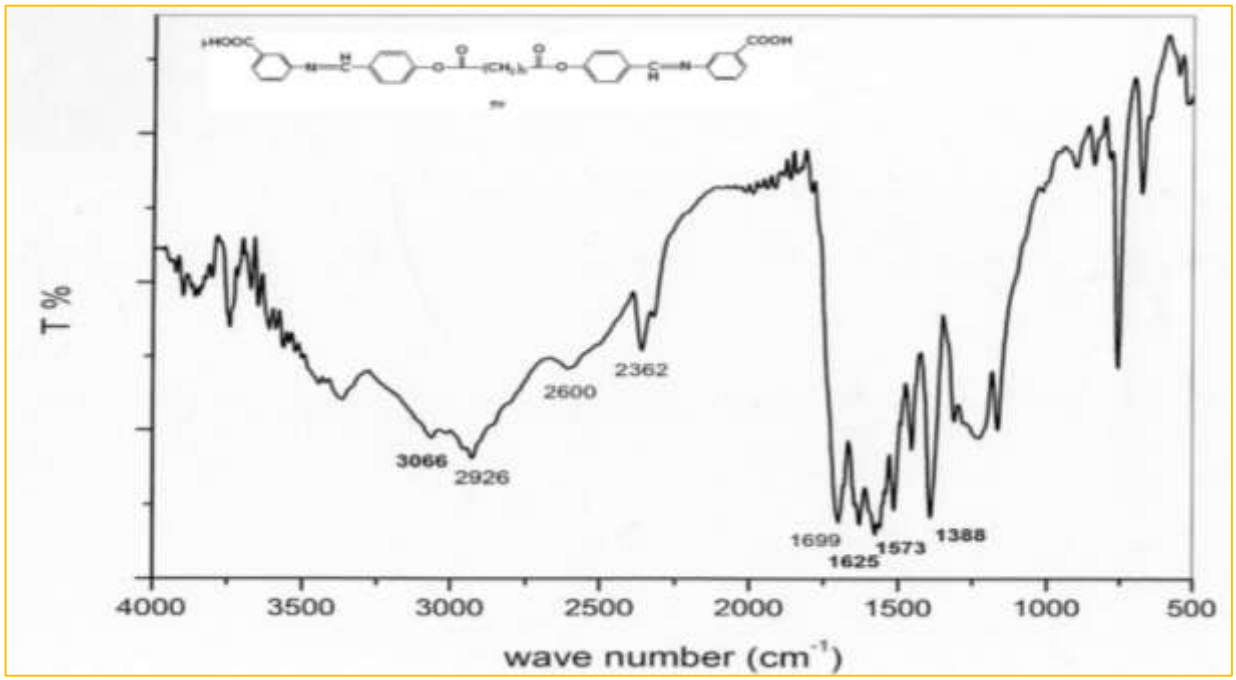

Figure 6: FTIR Spectrum for compound (6 d)

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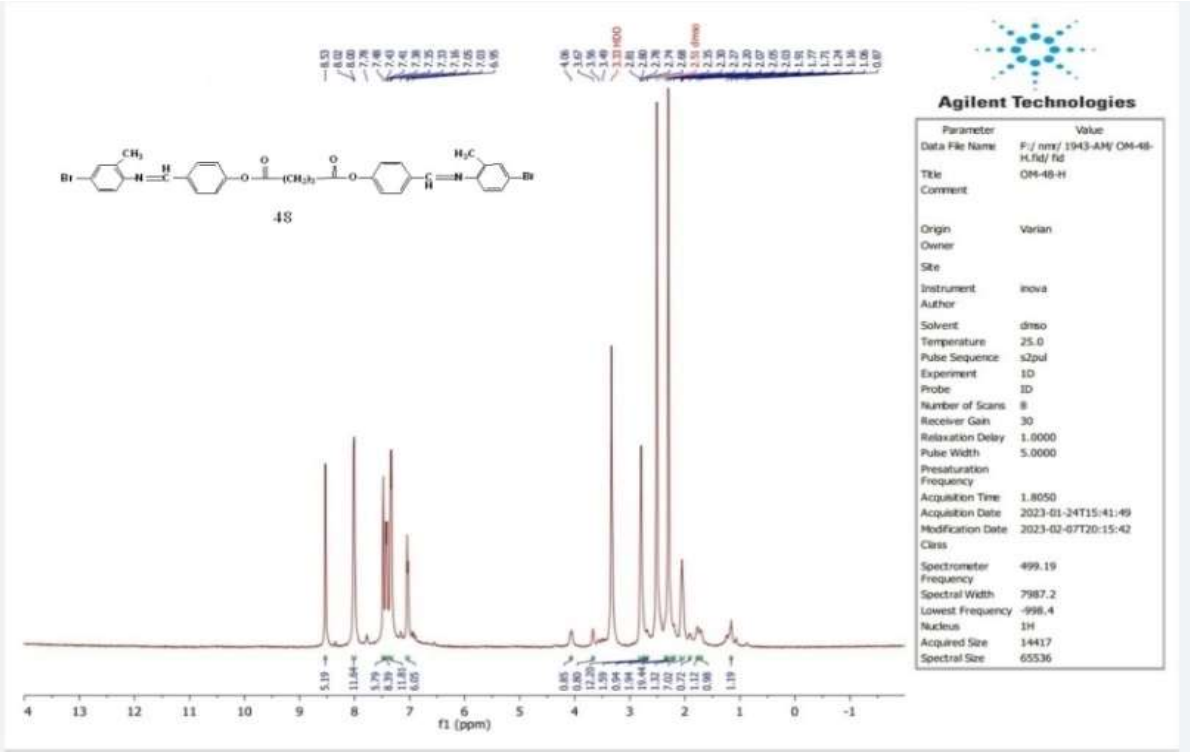

Figure 7: [1]HNMR Spectrum for compound (6 a)

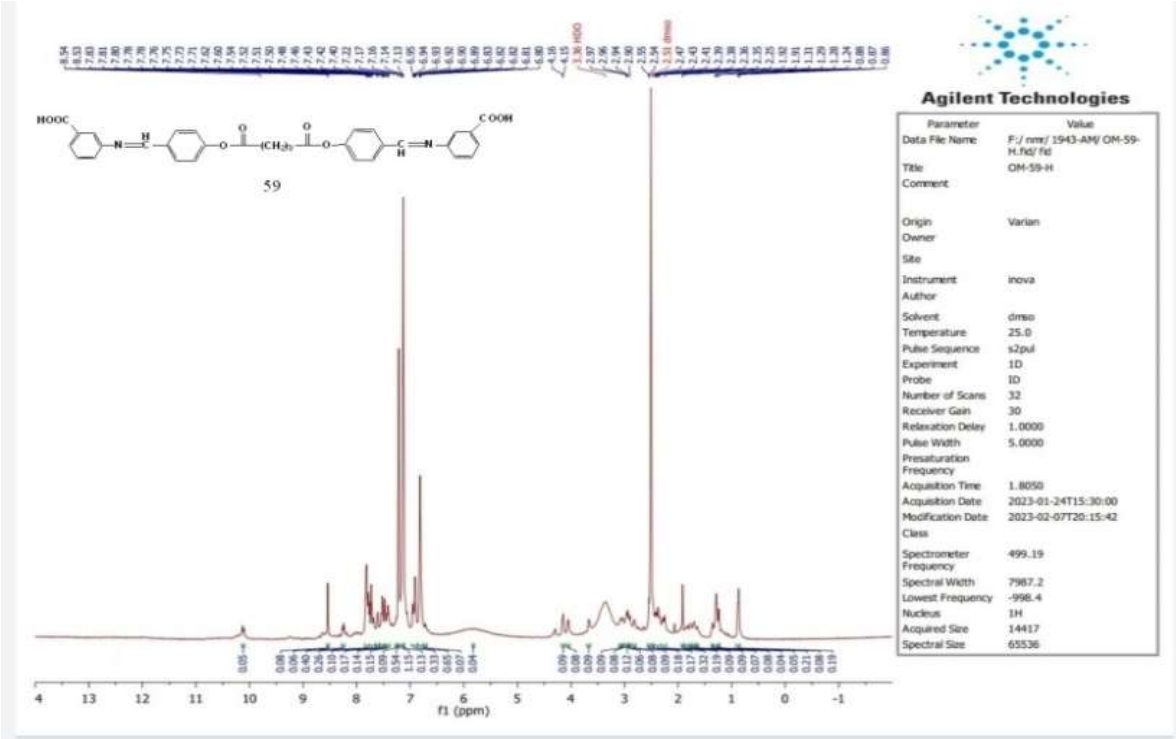

Figure 8: [1]HNMR Spectrum for compound (6 d)

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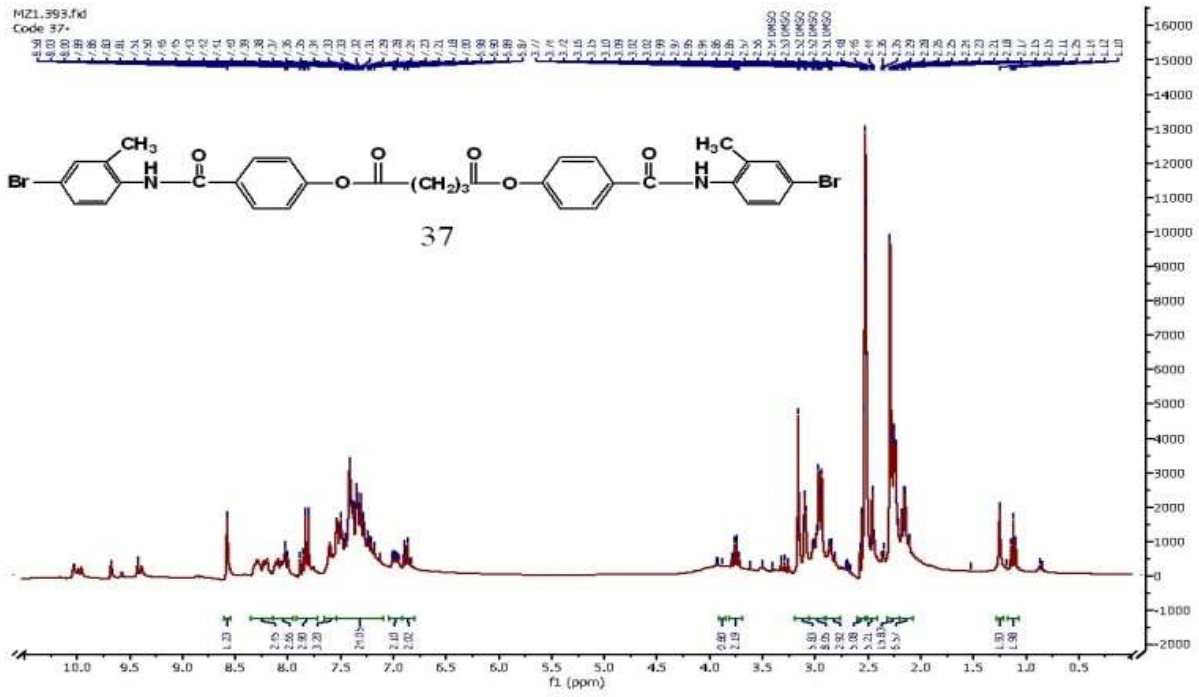

Figure 9: [1]HNMR Spectrum for compound (6 i)

Biological activity

The antimicrobial activity of the compounds 6a, 6d, 6h and 6I in a 10% DMSO solution has been assessed against three of strains of bacteria such as: *E.coli*, *psudomonas aeruginosa* then the *staphylococcus aureus*. The experimental part has been conducted by employing (nutrient agar plates) [22].All the plates were incubated at a temperature of 37 degrees Celsius for a duration of 24 hours [23,24]. The findings of the study indicate that each chemical exhibits distinct biological action on the aforementioned bacteria, with the exception of compound (6 a), which has no discernible biological activity towards *E. coli* and compound (6 d) has no activity toward *staphylococcus aurous* and compound (6 a) and (6 i) have no activity toward *Psudomonas aeruginosa*. Table (3).

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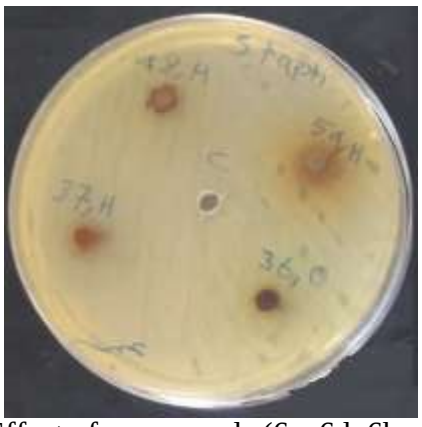

Figure 10: Effect of compounds (6a, 6d, 6hand 6i) on *staph*….

Figure 11: Effect of compounds (6a, 6d, 6hand 6i) on *E.coli*….

Figure 12: Effect of compounds (6a, 6d, 6hand 6i) on pseud

Conclusion

Present research work involves synthesis of Schiff bases derivatives to explore their antibacterial activity. We discovered that the compounds 6a, 6d, 6h, and 6i showed moderate growth against all bacteria of *E_ coli* and *staphylococcus aureus* basides *psudomonas aeruginosa*. Hence, it is conculuded that there is ample scope for further study in developing these as good lead compounds for the treatment of bacterial strain.

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