

Synthesis, Characterization and Study of Antibacterial and Electrical Conductivity of New Schiff Bases from Thiophene -3-Carboxyaldehyde and Two Different Para Substituted Aniline

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Abstract

Schiff base derived from (3-thiophene carboxyaldehyde with p-amino aniline, and 4-N, N-diethyl aniline) has been prepared and characterized by Infrared and, and C.H.N. elemental analysis. Results of the in vitro antibacterial activity showed that the N-[(thiophene-3-yl) methylidene]-4 (N, N diethylamino) benzene (compound A) was found to be more active than N-[(thiophene-3-yl) methylidene] -4-nitrobenzene (compound B) against most pathogenic bacterial strain for both of Gram-positive bacteria, *Staphylococcus aureus* and Gram-negative bacteria, such as *E. coli* under study. Some of these compounds showed potential antimicrobial activities. In addition, the electrical conductivity of new Schiff bases was characterized; new Schiff bases were showed high electrical conductivity and that increase of ability to prepare anew-organic diodes.

Keywords: Schiff base, antibacterial, electrical conductivity, pathogenic bacteria.

1. Introduction

Compounds of Schiff base produced from aromatic amines with aldehydes by condensation have a enormous range of applications [1-4] in many biological field [5-9], inorganic ligands applications [10-11] and analytical chemistry as chelating compounds for [12]. In the latest years, Schiff bases from thiophene compounds have been getting larger profits in conjugated organic materials due to their attractive electronic properties [13,14]. Many studies on small molecules have been focused on thiophene [15], which has been used as an effective p-type semiconductor for organic field-effect transistors. Schiff bases remained an important area of research due to their simple synthesis, and wide applications. In this, work the synthesis and characterization of the new Schiff bases obtained by reaction of 3-thiophene carboxyaldehyde with 4-nitroaniline and 4-N, N-dimethyl aniline. Because of the development of resistance to the major classes of antibacterial materials are renowned as a serious health worry, the study for new antibacterial materials with different action modes has constantly continue essential. Due to the development of resistance of some types of bacteria to chemical treatments, this is the reason for the constant search for the creation and preparation of new compounds with appropriate behavior and action and is compatible with the development of resistance to these types of bacteria [16].

2. Materials and Instruments

Thiophene-3-carboxaldehyde, 4-N, N-diethyl aniline, and 4-nitroaniline were purchased from Merck and Used without treatment. Other reagents and solvents were of analytical grade and purchased commercially.

FTIR spectra (potassium carbonate discs) were recorded on a JASCO FT/IR 4200 instrument, with a wave number range of 400-4000 cm^{-1} . The electronic spectra were measured in the range of 200 - 1100 nm for 10^{-3} by using DMSO solvent by using UV-Visible spectrophotometer type Shimadzu UV-160A using quartz cell of (1.0 cm) length, Available at Polymer Research Center, Basrah University, Iraq. In addition, the micro elemental analyses were obtained on Exeter Metal Analytical CE 440. Available at Chemistry Department /College of Science/ University of Basrah, Iraq.

2.1. Synthesis of N-[(thiophen-3-yl) methylidene]-4 (N, N diethyl amino) benzene (A).

An ethanolic solution of 3- thiophenecarboxaldehyde (1 mmol, 25 mL) was added to an ethanolic solution of 4-N, N-diethyl aniline (1 mmol, 25 mL) with added 2 drops of glacial acetic acid and the reaction was continued for 4 hours with refluxed on a hotplate at 75°C with stirrer. After cooling, the solution, the product was precipitate, separated, filtered, recrystallization with ethanol, and dried over anhydrous CaCl_2 under vacuum. The obtained product was showed in Figure (1).

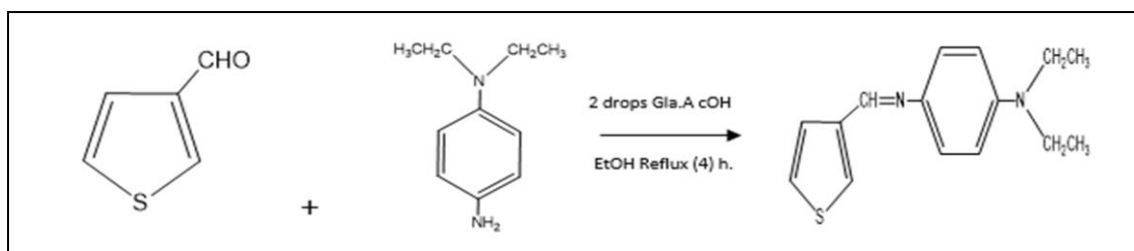


Fig (1): Synthesis of N-[(thiophen-3-yl) methylidene]-4 (N, N diethyl amino) benzene (A).

2.2. Synthesis of N-[(thiophen-3-yl) methylidene]-4-nitrobenzene (B)

An ethanolic solution of 3- thiophenecarboxaldehyde (1 mmol, 25 mL) with added 2 drops of glacial acetic acid was added to an ethanolic solution of 4-nitroaniline (1 mmol, 25 mL) and the reaction was continued refluxed for 4 hours with refluxed on a hotplate at 75°C with stirrer. After cooling, the solution, the precipitate was separated, filtered, recrystallization with ethanol, and dried over anhydrous CaCl_2 under vacuum. Figure (2) showed the chemical reaction of the preparation.

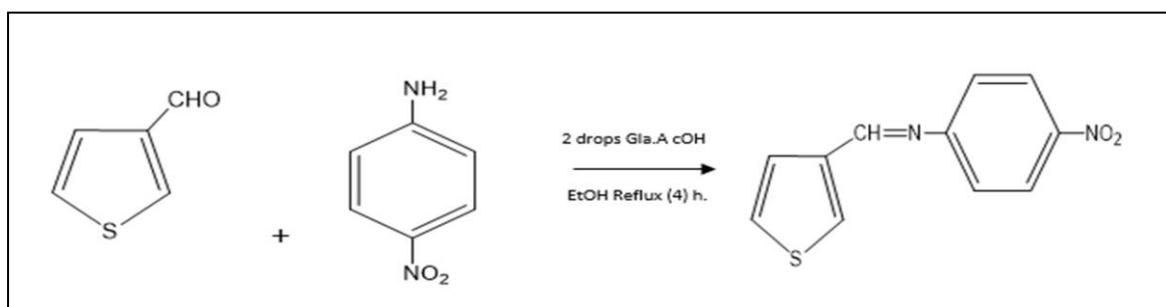


Fig. (2) Synthesis of N-[(thiophen-3-yl) methylidene]-4-nitrobenzene (B).

2.3. Biological activity

To determination of the Antibacterial Activity against anew Schiff bases, six clinical bacterial strains were studied: *Staphylococcus aureus*, *Salmonella typhi*, *Escherichia coli* and *Vibrio parahaemolyticus*.

A bacterial suspension of each isolate was prepared and equalized to 0.5 Mc Far land standard then the solution was spread on the entire surface of Muller Hinton agar by sterilized cotton bud [17], after drying 9 mm, diameter pore was made in the center of each plate by using cork borer with duplicate and control plates. All inoculated Petri dishes incubated at 37°C overnight. Inhibition zone was measured as the diameter of the clearing zone in millimeters [18].

3. Results and Discussions

3.1. Spot Test (Ligands Complex) agents

Spot test technique used to study the ability of prepared ligands to form chelating complexes with various metal, spot-test technique was used by preparation of (1N) of ligands solution in absolute ethanol and (1N) of each metal salt in absolute ethanol, then two drops of ligands solution was added to 2 drops of metal salt solution (1:1) and the changes were noticed and recorded. In addition, Table (1) was showed the results of this technique. We note the ability to formation complex of the metal ions of elements with Schiff bases from the reaction of aniline substituted with donor group of $-N(Et)_2$ especially with Fe^{+3} , Mn^{+2} , Co^{+2} , In^{+3} , and Cu^{+2} .

Schiff base which substituted with electron with drawing group (NO_2) did not complexes with iron ions because of the nitro group is drawing the electronic density and then decrease the electrical negativity of nitrogen of azomethine group by interaction of the double electron of π -system of the ring, that make the group of azomethine weak donor group which make the ability to make complex with metal ion by nitrogen atom and sulfur atom in the Schiff base ligands, but, we think that the Schiff base complex with Pt^{+4} chelate by sulfur atom only of the thiophene-3-carboxaldehyde.

Table (1) show the Ligand complexes

compound	Cd^{+2}	Pt^{+4}	Co^{+2}	In^{+3}	Fe^{+3}	Ga^{+3}	Nd^{+3}	Bi^{+3}	Cr^{+3}
A	-	-	-	+	+	+	-	-	-
B	-	+	-	-	-	-	-	-	-

Compound	CS^{+1}	Ni^{+2}	Mn^{+2}	Pb^{+2}	Cu^{+2}	Sr^{+2}	Ba^{+2}	Mg^{+2}
A	-	-	+	-	+	-	-	-

B	-	-	-	-	-	-	-	-
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3.2. C.H.N. Elemental analysis

The micro elemental analyses were obtained on Exeter Metal Analytical CE

440. available at the Chemistry department /college of science/ University of Basrah, Basrah-Iraq. The elemental analysis data for the prepared Schiff bases are shown in Table (2). The results imply that the experiment % of metals are in a good agreement with the Calculated %, this reflects the expected compositions of the prepared Schiff bases. The melting point is shown in the table (3). Sharp melting point values show the purity of the prepared Schiff bases.

Table (2) C.H.N. Elemental analysis

compounds	theoretical			practical		
	C%	H%	N%	C%	H%	N%
A	69.77	6.977	10.853	69.978	7.254	10.698
B	56.897	3.448	12.069	56.198	3.554	11.997

3.3. FTIR Spectrum

(FTIR) analysis with of each Schiff base was done to affirm the creation of Schiff bases. The FTIR data of the spectra of Schiff bases (A and B) showed a sharp band at 1604-1633 cm^{-1} assigned to the azomethine group ($-\text{C}=\text{N}$), their bands are showed in Table (4), and disappearances of the carbonyl group and amine group, thus clearly gave evidence of condensation between aldehydes and amines. Figures (3 and 4) showed spectrum of (A) and (B). Many other researchers who have synthesis Schiff bases using varieties starting material made similar kinds of observation.

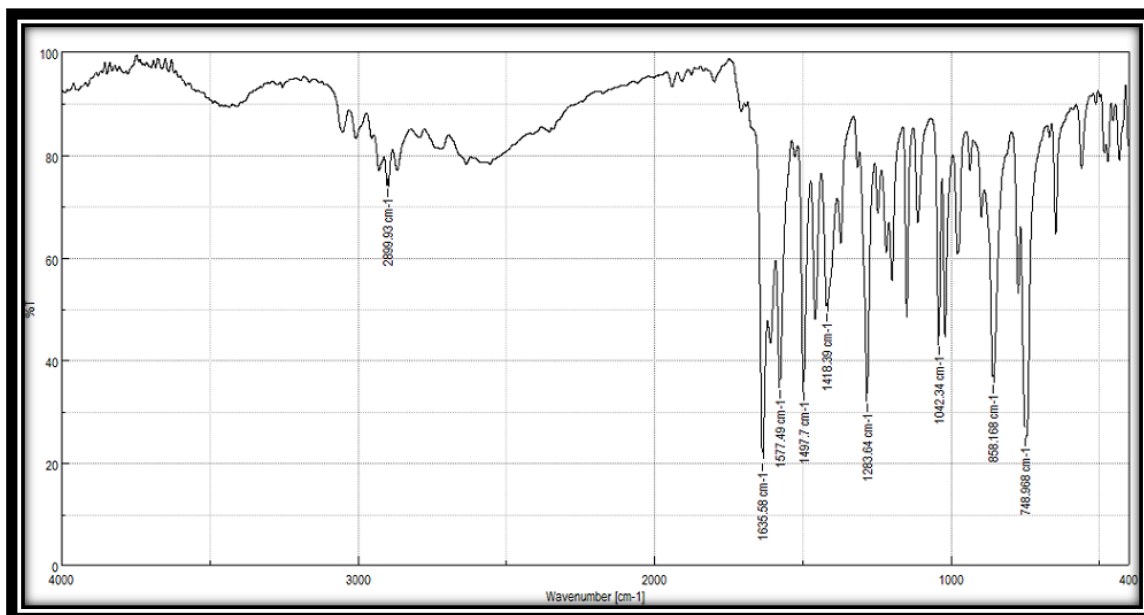


Figure (3) FTIR Spectrum of compound A

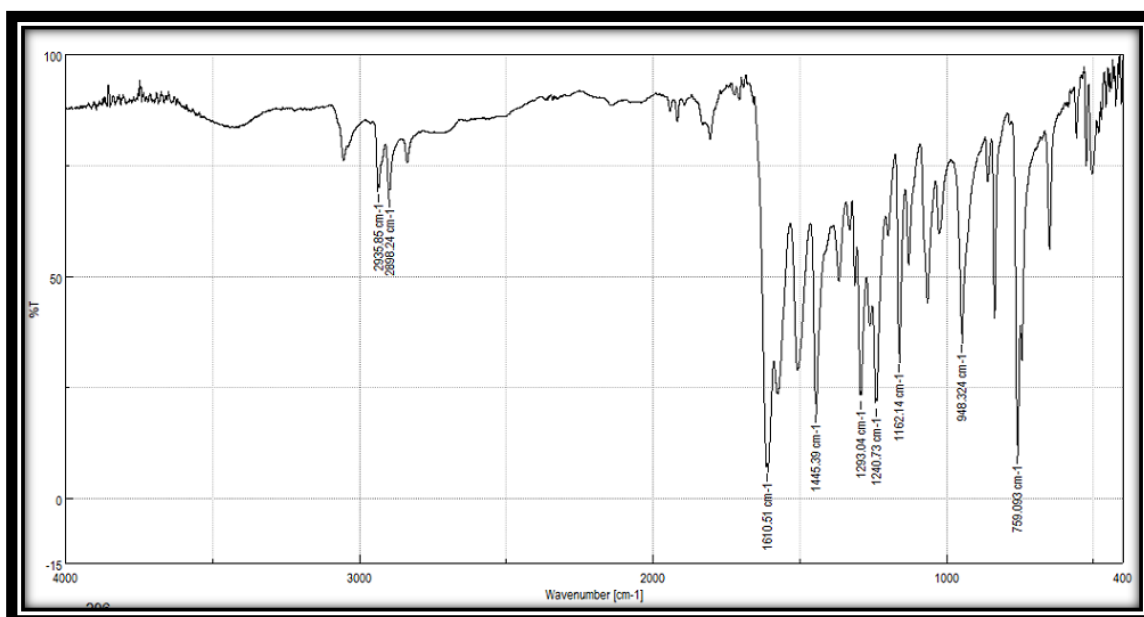


Figure (4) FTIR Spectrum of compound B.

Table (3) Some Physical Properties.

Compounds	Melting point	Physical state
(A)	116-118c ^o	Solid gold crystal
(B)	90-91c ^o	Solid Yellow crystal

3.4. Ultra violet –Visible Spectroscopic study

The (Uv-Vis) spectrum for the compound (A) exhibits high absorption peak at (209 nm) ($\epsilon_{\max}=1720\text{molar}^{-1} \cdot \text{cm}^{-1}$), high absorption peak at (258nm)($\epsilon_{\max}=1700 \text{ molar}^{-1} \cdot \text{cm}^{-1}$) and intense absorption peak at (378nm) ($\epsilon_{\max}= 1600 \text{ 1molar}^{-1} \cdot \text{cm}^{-1}$), which were assigned to ($\pi \rightarrow \pi^*$), ($\pi \rightarrow \pi^*$) and ($n \rightarrow \pi^*$) transition respectively, Table (4) showed the value of UV-Visible spectroscopic of (A) and (B).

Table (4) Absorption bands of Schiff bases in Uv, Visible in an alcoholic solvent and IR vibration bands of imines group.

Compound	(-C=N) Vibration in IR	$\lambda_{\max}/\text{nm} \text{ molar}^{-1} \cdot \text{cm}^{-1}$
A	1635 cm^{-1}	209(1720), 258(1700), 378(1600)
B	1610 cm^{-1}	207(1750), 228(1650), 377(2415)

3.5. Antibacterial activity

In the present study, two compound (A) and (B) were tested against *Salmonella typhi*, *Vibrio parahaemolyticus*, *Escherichia coli* and Gram-positive *Staphylococcus aureus*. The results indicate that the first compound (A) showed a significant and strong effect against most of the pathogenic bacteria under the study while the second compound (B) show less antibacterial activity in comparison with the first compound. This can be seen in Table (5), which shows the diameters of the inhibitory zones and the photographs of inhibition of bacteria was shown in Figure (5).

The reason for these results can be attributed to the electron density on the amine group by the payment of the ethyl aggregates, which are electron aggregates. This electronic density activates the biologic activity of the compound against the bacteria.

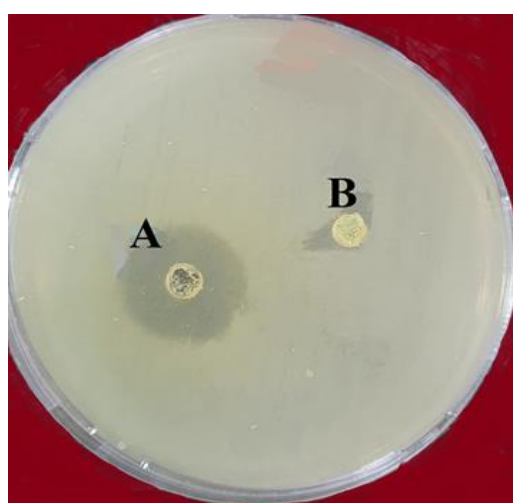
Table (5). Showed the effect of (two Schiff bases A and B) against 4 pathogenic bacterial isolates. The results determined by measuring the inhibition zone diameter millimeter (mm):

Bacteria	Diameters of inhibition zones mm	
	A	B
<i>Staphylococcus aureus</i>	14	8
<i>Vibrio parahaemolyticus</i>	12.5	R
<i>Salmonella typhi</i>	20	7.5

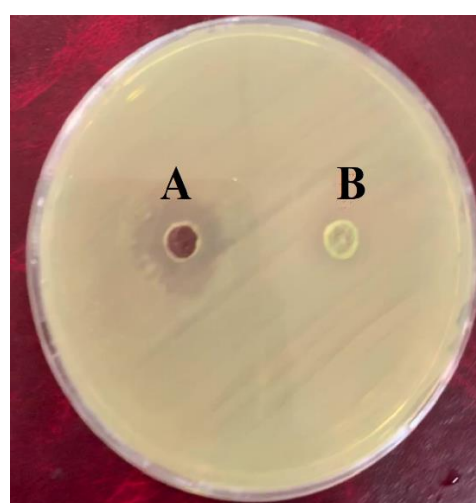
<i>Escherichia coli</i>	16	6
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- Where R means resistance.

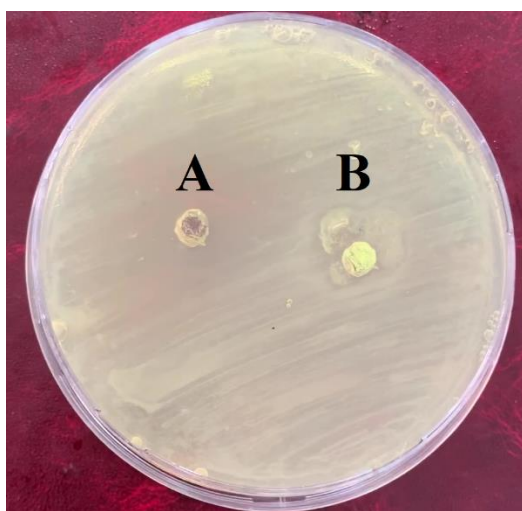
For compound (A), it was observed that the zone of inhibition for *Salmonella typhi* was large than the other bacteria, this indicates the efficacy of the compound to penetrate the bacterial cell wall, such changes may produce to raise in membrane permeability and seepage of intracellular constituents and cause severe damage, in the end causing in cell death. These results were in a good agreement with previously investigation onto antibacterial properties it has been recorded that some chemical compound could cause structural changes when they interact with the outer membrane of bacteria [19]. These results in agree with many researchers who have specified with differences in bacterial susceptibility may be due to structural and compositional differences in the cell membrane of Gram-positive and Gram-negative bacteria [20-22].



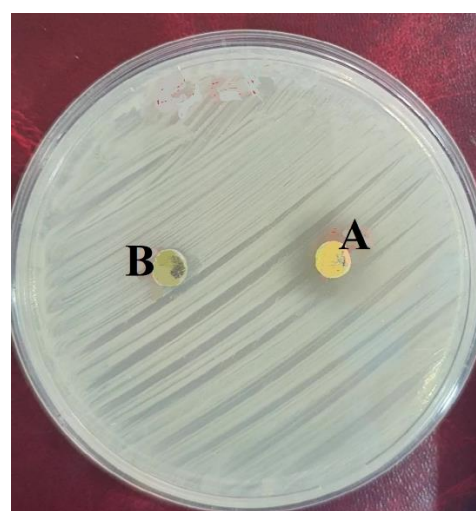
(a)



(b)



(c)



(d)

Figure 5: The effect of (A&B) against bacterial growth. (a) Inhibition zones (mm) of *Escherichia coli*. (b) Inhibition zones (mm) of *Staphylococcus aureus*. (c) Inhibition zones (mm) of *Salmonella typhi*. (d) inhibition zones (mm) of *Vibrio parahaemolyticus*.

3.6. Electrical properties

After the process of cleaning the slides with acetone and the distilled water, the slides were attached to plastic parts by a wire of 0.015mm diameter, and then the electrodes were heated by high purity aluminium. After the evaporation process, the wire was raised. Regular casting for electrical properties study did this process of casting the materials used. For the purposes of measurement, we select two electrodes, each with a membrane on the glass slide and connected to the circuit.

The prepared models were measured within voltages (1-10 volt) and at room temperature. If the current values are observed starting from 6×10^{-8} and settling at 3×10^{-7} for compound (A) and the current values of compound (B) starting from 4×10^{-8} and settling at 5×10^{-7} and that showed in Figure (6) and Figure (7). The voltage is increased by increasing the voltages at 4.5V. We observe a surprising increase in current. At 6.5V, the current stabilizes. This behaviour is due to the connection of Schottky [23, 24]. If it is possible to conclude that, these compounds are diodes (possible as one of the layers of a solar cell)

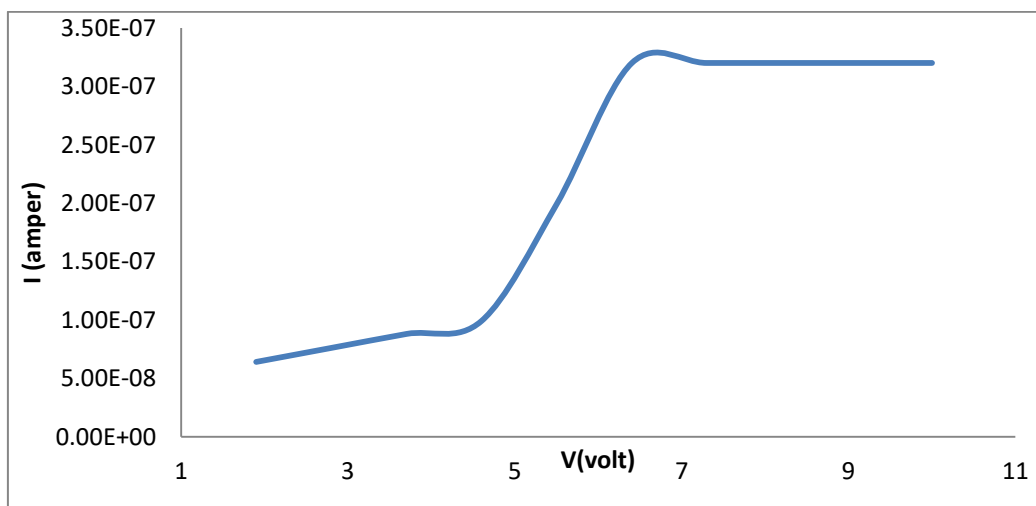


Figure (6) Represents the relationship between the voltages and the current of compound A.

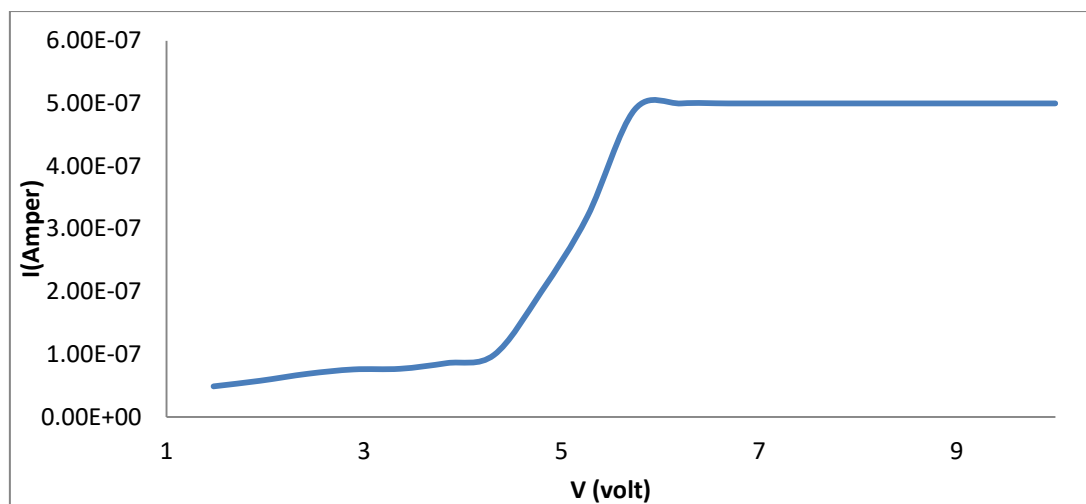


Figure (7) Represents the relationship between the voltages and the current of compound B.

4. Conclusion

Schiff bases of N-[(thiophene-3-yl) methylidene]-4 (N, N diethyl amino) benzene and N-[(thiophene-3-yl) methylidene]-4-nitrobenzene (B) were synthesized and characterized by analytical and spectral techniques, from the study of the ability of compounds (A) and (B) to form complexes, it was observed that compound (B) has a high selectivity towards platinum without any other ions, this behavior is of great importance in the fields of analytical chemistry and element capture. (A) and (B) exhibited significant activity against all the tested bacterial isolates and compound (A) showed a significant and strong effect against most of the pathogenic bacteria under the study while the second compound (B) show less antibacterial activity in comparison with the first compound, the ethyl aggregates, which are electron aggregates. This electronic density activates the biologic activity of the compound against the bacteria. The new Schiff bases have a good electrical conductivity that the current values are observed starting from 6×10^{-8} and settling at 3×10^{-7} for compound A and the current values of compound B starting from 4×10^{-8} and settling at 5×10^{-7} . The behaviour of the tow new Schiff bases was due to the connection of Schottky that, make the possibility of considering that, these compounds are diodes (possible as one of the layers of a solar cell).

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