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FULL PAPER

Synthesis, characterization, and studying of (thermal, spectral and physical) properties of new Schiff base monomers and liquid crystal compounds from Ampicillin

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This work presents syntheses of six new Schiff base compounds from Ampicillin, p-hydroxyl Benzaldehyde reacted with (pentanol, hexanol, and heptanol) to produce (4-pentloxy, 4-hexaloxy, and 4- hepteloxy) Benzaldehyde, and compounds (p-hydroxy, p- chloro, p-nitro) Benzaldehyde used to produce Schiff base compounds. The prepared compounds have been identified using (FT-IR and H1-NMR) techniques. The microscopic phases were determined using a polarized light microscope (POM), and the thermal properties of the transition temperatures for these compounds were measured using a differential scanning calorimeter (DSC). All compounds' melting points, colors, and yields were detected. Then the liquid crystalline behavior was studied, which showed that the compounds gave liquid crystalline properties and determined whether the compounds gave the nematic phase or the smectic phase.

KEYWORDS

Schiff base; monomers; liquid crystal compounds; Ampicillin; thermal; spectral and physical properties.

Introduction

Ampicillin is an essential compound of cyclic organic compounds containing heterogeneous rings, as well as including in its composition two nitrogen atoms and one sulfur atom. These rings have medicinal chemical properties and can enter several essential reactions, including Schiff reactions because they contain the primary amine group's functional group [1]. The compounds resulting from these reactions were liquid crystals. These organic compounds pass through a state intermediate between the solid and liquid forms, i.e., between the regular and random arrangements [2]. Nitrogen-containing compounds have a medical and pharma logical importance [3]. They were effective as anti-cancer [4] and anti-bacterial [5,6]. Liquid crystals have been classified according to how they arise into two main categories: **lyotropic** liquid crystals, which arise from adding specific volumes of a polar solvent such as water or alcohol to calculated amounts of amphiphilic organic compounds at room temperature or higher, and **thermotropic** liquid crystals [7].

This class of liquid crystals arises from melting solid organic compounds with standard general molecular features. They are long, relatively hard, and rod or lamella shaped, so they are thermally activated mesophases. Since this research focuses on the study of thermo-tropic liquid crystal compounds, the discussion in the following paragraphs will focus on this type of liquid crystal. The distinction between aero tropic liquid crystals and thermotropic liquid crystals is not always sharp as it has been

found that many compounds exhibit erotic and Thermo tropic properties called Ionomeric liquid crystals [8].

Preparations

-Preparation of 4- alkoxybanzaldehayd [9,10,11,12]

(0,025) mol of 4-hydroxybenzaldehyde was dissolved in 10 mL of ethanol, and (0,033) mol KOH solution was added to it slowly in a sublimation flask, 0.025 mol of the appropriate alkane bromide was added to it and left for 1 hour, and the product was extracted By petroleum ether. The solvent was evaporated, and the product was obtained as a yellow liquid [9].

Alkane bromide of Preparation-

Alkane bromide is prepared from 48% HBr acid with 16.5 mL of concentrated sulfuric acid in batches in a reaction flask placed in an ice bath, then 0.5 moles of suitable alcohol is added, and the second batch of sulfuric acid 12.5 mL, then the mixture is refluxed and heated for (5–10) hours until the bromide layer separates from the acid solution layer, then cools down and is transferred to the distillation apparatus until the descent of the oily drops of bromide ends, washed sequentially with water, hydrochloric acid and sodium carbonate solution, then with water, then dried with anhydrous calcium chloride [10,11].

Synthesis of Schiff basis compounds

Synthesis of Schiff's base compounds from Ampicillin by taking an equal number of moles of Ampicillin and various aldehyde compounds, dissolved in 50 mL of absolute ethanol and drops of glacial acetic acid for 5 hours at 120 °C, after which the precipitate is filtered and recrystallized [11,12], as shown in Figure 1.

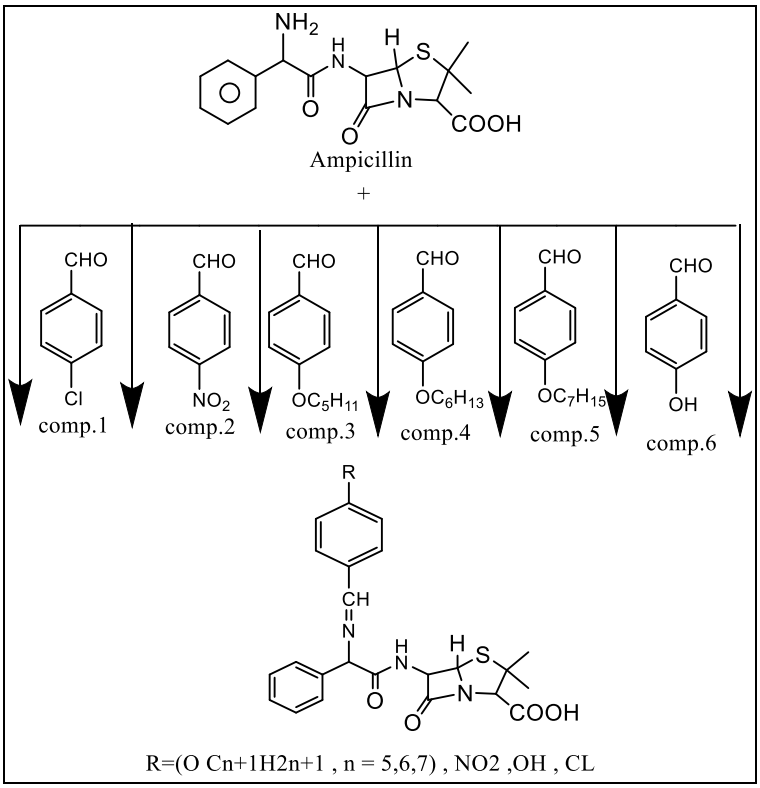


FIGURE 1 Interaction diagram

Results and discussion

Monomers are small molecules containing at least two functional groups (bifunctional monomers) used to connect with other monomers to form the chain of polymer [13,14]. All the prepared compounds contain a carboxylic acid group and other fictional groups depending on the structure. For example, Compound 6 includes hydroxyl groups, Compounds containing hydroxyl and carboxyl functional groups are the basic monomers for synthesizing polyesters [15]. Chlorine is an excellent leaving group, and when it connects to the benzene ring, such as compound 1, it can transform to hydroxyl by hydrolysis reaction with heating and pressure [16]. Compounds 3, 4, and 5 are aromatic ethers that can convert to phenols (hydroxyl group) by reaction with boron tribromide, then hydrolysis with an excess of water [17]. Compounds containing Nitro groups, like compound 2, can reduce to amino groups influential in the synthesis of polyamides [18].

FT.IR-data(cm^{-1}) of compounds

In the first compound, we notice the presence of the peak of the Azomethine group at **1593** cm^{-1} and the disappearance of the pseudo-group in the aldehyde(C=O). The appearance of the acidic functional group (C=O) of the ampicillin compound, the second compound we note the clear and sharp peak of the Azomethine group at **(1523)** cm^{-1} and the acid (OH) group at (**3394**) cm^{-1}, compounds 3,4, 5 We find the group of Azomethine from (**1512 _ 1514**) cm^{-1} and the appearance of the ether group(CO) at (**1020–1053**) cm^{-1}, in addition to the presence of a(COC) group at **(1300 – 1452)** cm^{-1}, as for compound 6 we find the presence of an(OH) group of aldehyde in addition to the acid (COOH) group of the amine compound that appeared at (**3200-3290**)cm^{-1} wide. [19,20] as in Figures 2-9.

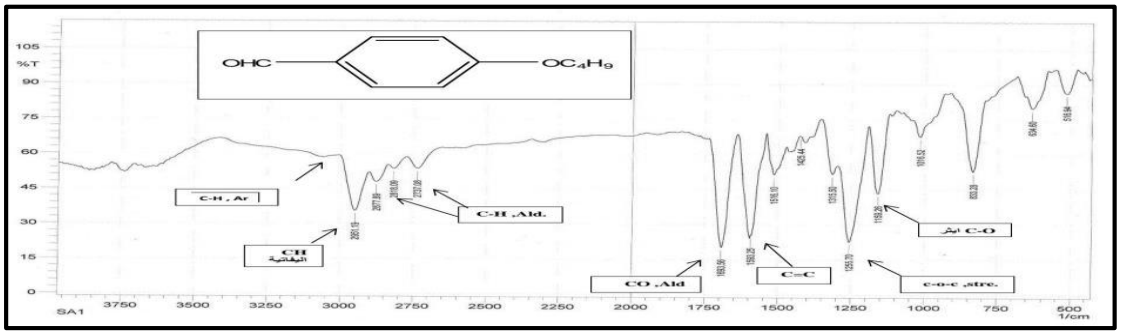


FIGURE 2 4-alkokxy benzaldyhed

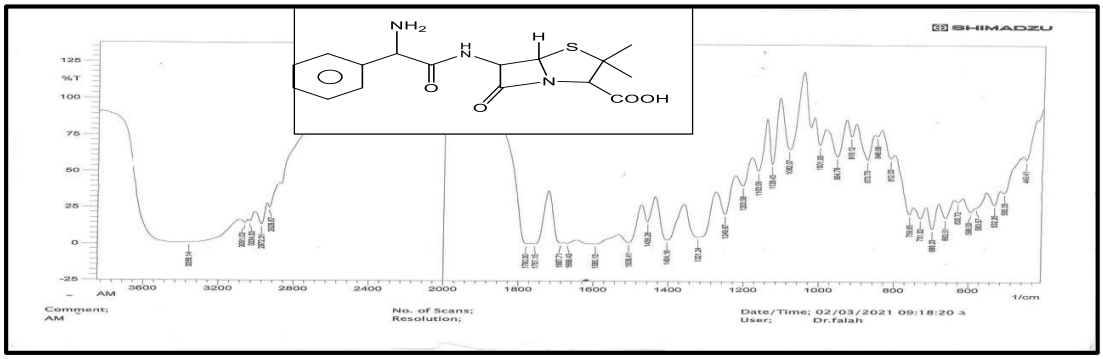


FIGURE 3 Ampicillin

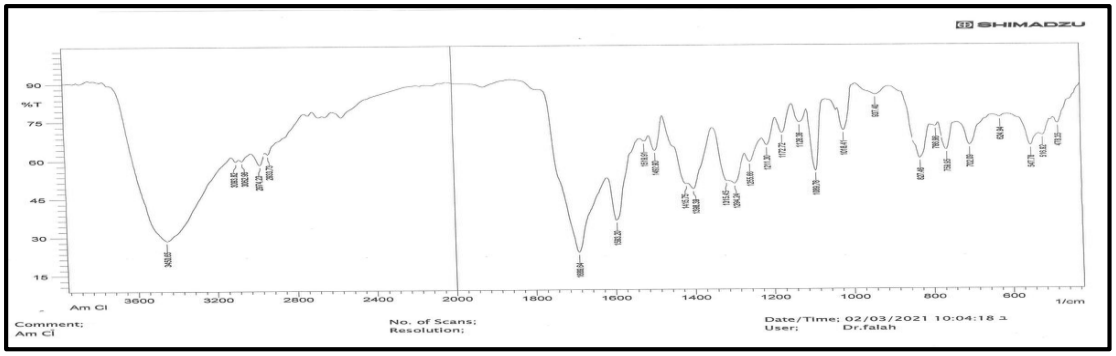


FIGURE 4 Compound 1

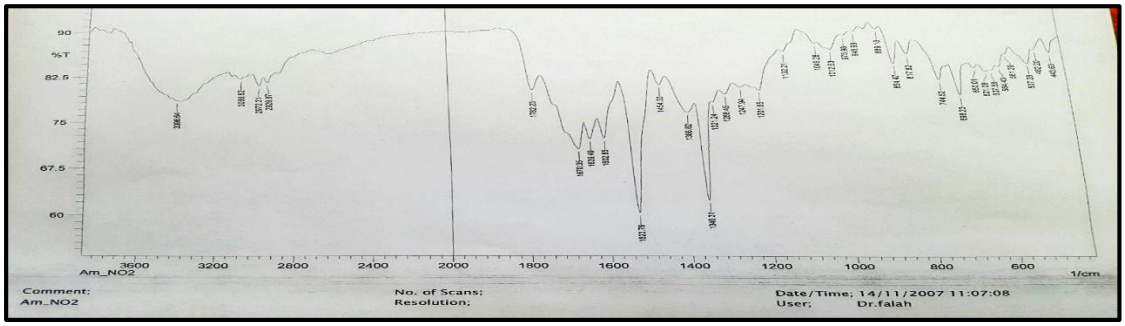


FIGURE 5 Compound 2

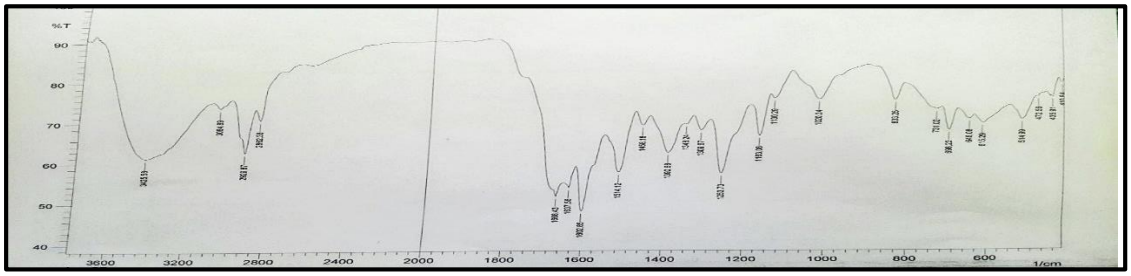


FIGURE 6 Compound 3

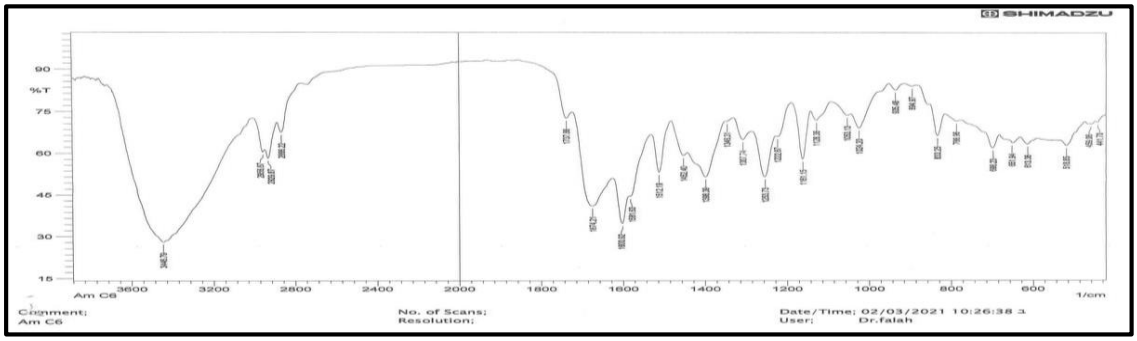


FIGURE 7 Compound 4

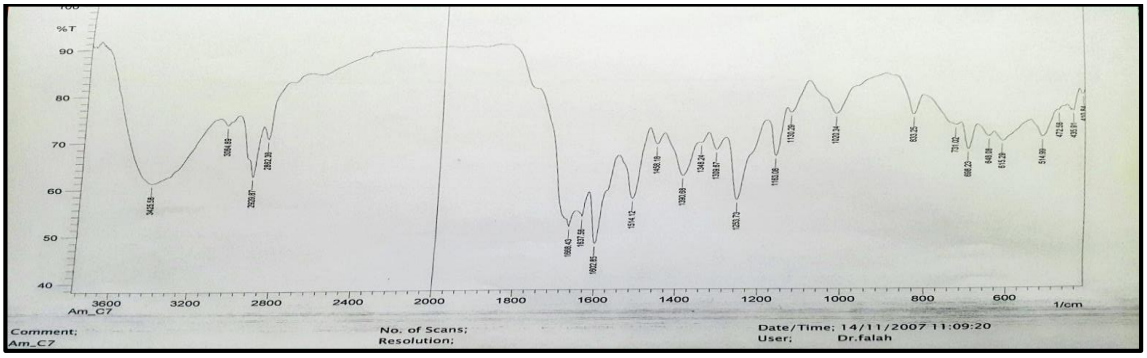


FIGURE 8 Compound 5

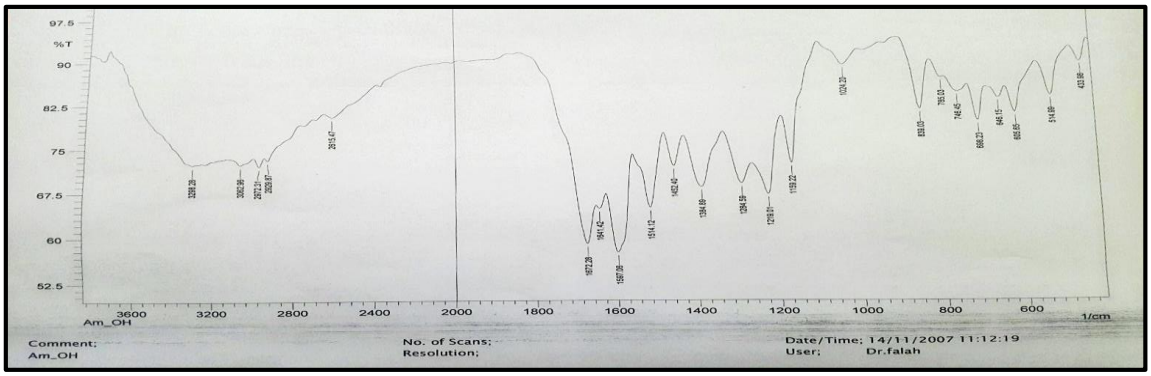
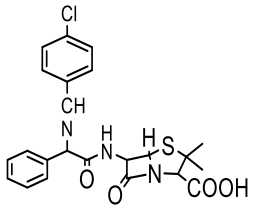
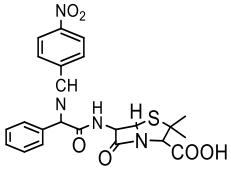
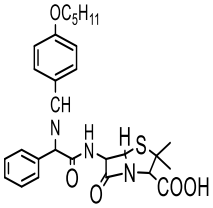


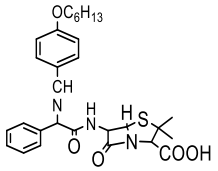
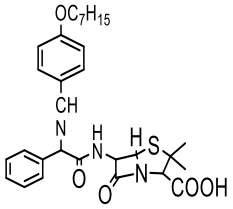
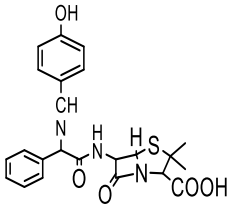
FIGURE 9 Compound 6

Studying the physical properties

The physical properties of the sixth prepared compounds were investigated by measuring the melting point, detecting the colors, and calculating the yield, as shown in Table 1.

TABLE 1 the physical information of the prepared compounds

NO .of compound	MP. C$_o$	Color	Yield	Chemical structure
1	114	Brown	80%	
2	110	Brown	85%	
3	114	Yellow	72%	

4	119	Yellow	64%	
5	109	Yellow	68%	
6	115	Yellow	76%	

H1-NMR Spectrum

It showed a signal at (7.811) that returns (CH=N) a proton from the Imine group in the compounds, as well as a signal at (12,238) that returns to the proton of the carboxylic acid group (COOH) of amoxicillin, as shown in the Table 2. [21,22,23].

The polarized optical microscope depictions are shown in Table 3.

TABLE 2 H1NMR-data (δ ppm, DMSO) of some compounds

Compound No	(HNMR)only important groups 1
1	(12.238) proton of (OH) hydroxyl of carboxylic acid,(8.579)proton of (C=O-NH), (CH2, (CH3) proton group at (0.776-0.719),(CH=N) proton of imine group (7.811), Ar-H proton of phenyl ring (6.778-7.766).
2	(12.238) proton of (OH) hydroxyl of carboxylic acid,(8.50)proton of (C=O-NH), (CH2,) proton group at (0.776-0.719),(CH=N) proton of imine group (7.814), Ar-H proton of phenyl ring (7.814),(6.59) proton of phenol group.

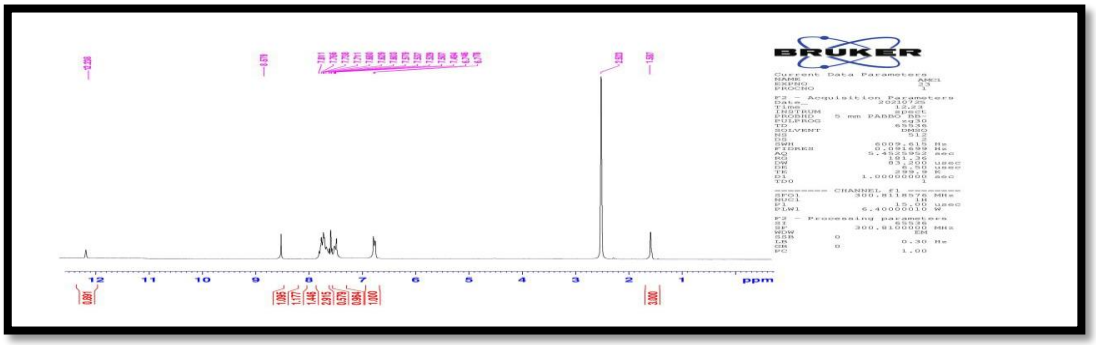


FIGURE 10 Compound 1

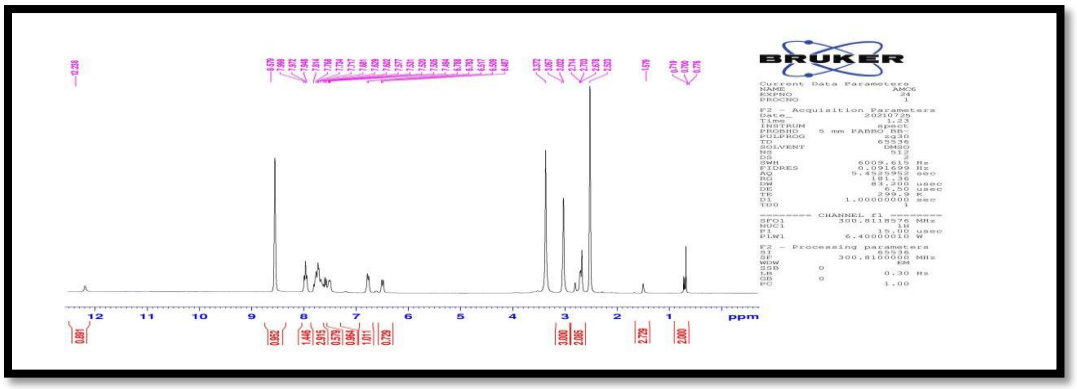
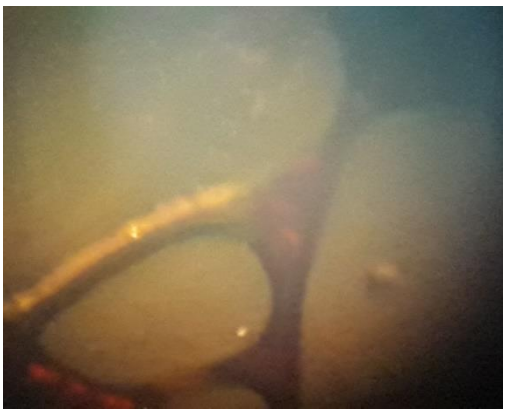
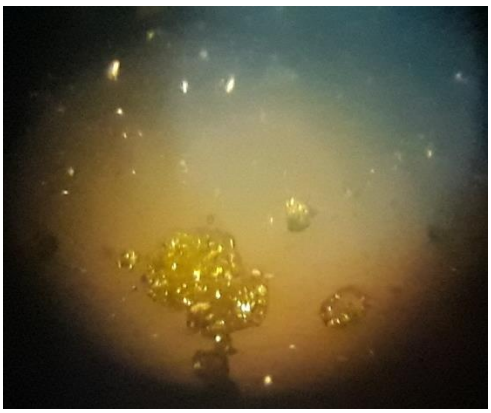


FIGURE 11 Compound 2

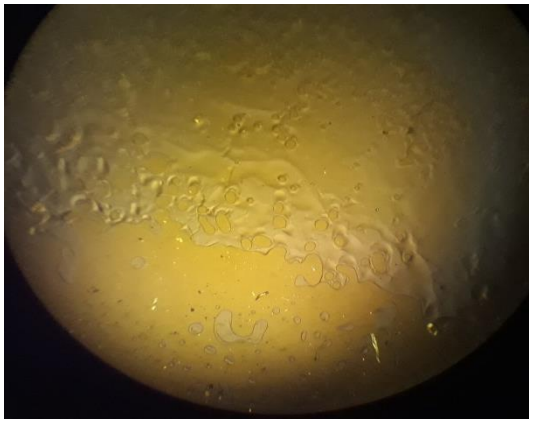
TABLE 3 Polarized light microscope of the prepared compounds by interpretation of microscope images based on [24,25]



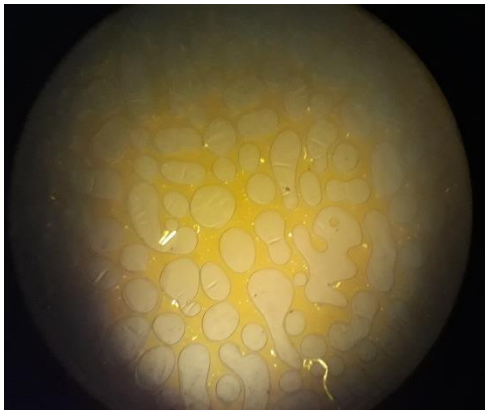
Compound 1 nematic phase at 112 °C by cooling



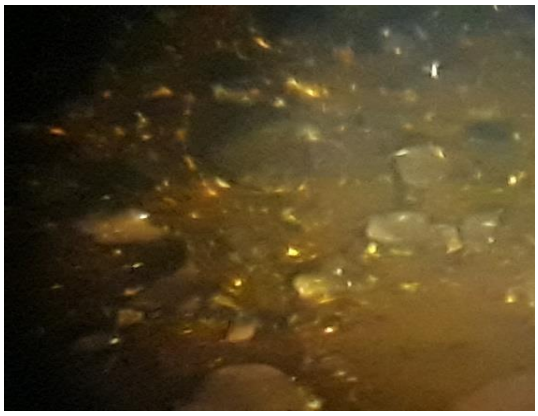
Compound 2 nematic phase at 116 °C by heat



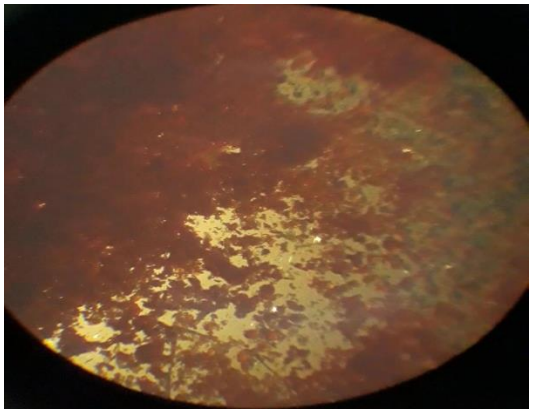
Compound 3 Smectic phase at 114 °C by heat



Compound 4 Smectic phase at 105 °C by heat



Compound 5 Smectic phase at 106 °C by Heat



Compound 6 nematic phase at 108 °C by cooling

Characterization of liquid crystalline compounds through differential scanning Calorimetry (DSC) measurements [26,27,28,29]

The first compound showed the presence of a nematic phase at 110 °C by heating, another by cooling at 108 °C, and a smectic phase at approximately 96 °C.

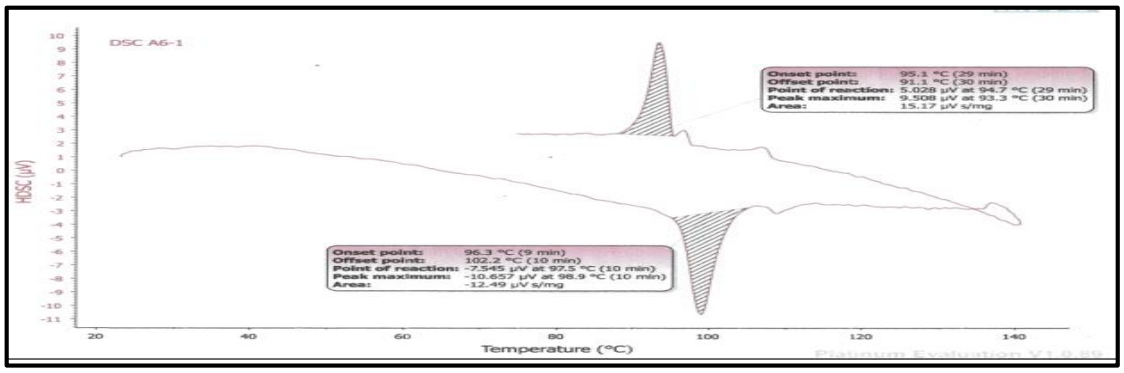


FIGURE 12 Compound 1

In the second compound, a phase appears at 110 °C by heating, and two phases appear by heating at 108 and 88.9 °C, which both were nematic phases.

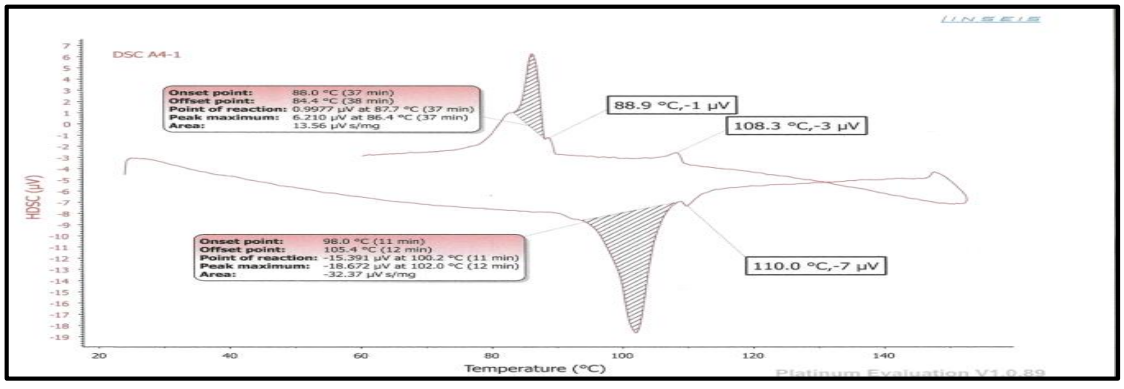


FIGURE 13 Compound 2

In the case of the Third compound, there were two phases, the first one was a smectic phase by heating at 109 °C, and the second one was a nematic phase by cooling at 107.5 °C.

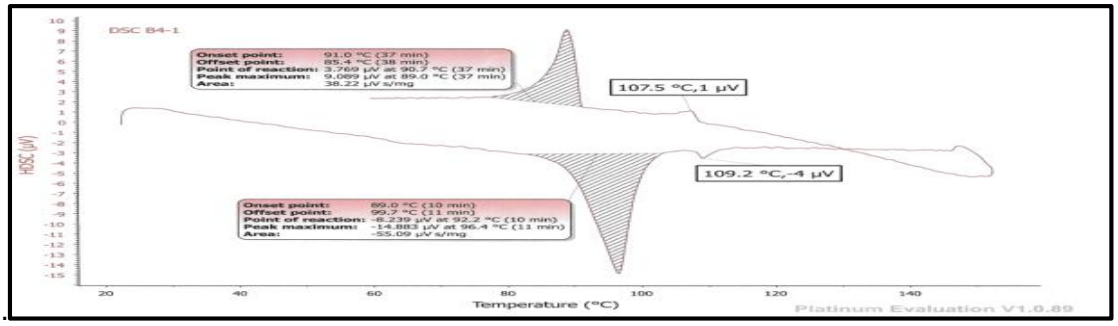


FIGURE 14 Compound 3

The phases of the fourth compound only appeared in the case of cooling at (104.9 and 98.3) °C. The first is smectic unknown, and the other is smectic C.

The appearance of a smectic phase in the fifth compound by heating at 107.9 and the last smectic phase by cooling at 106.3 °C.

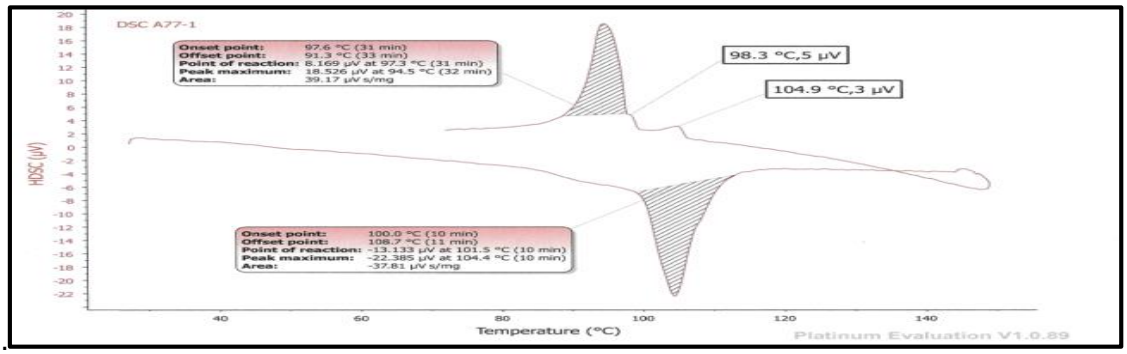


FIGURE 15 Compound 4

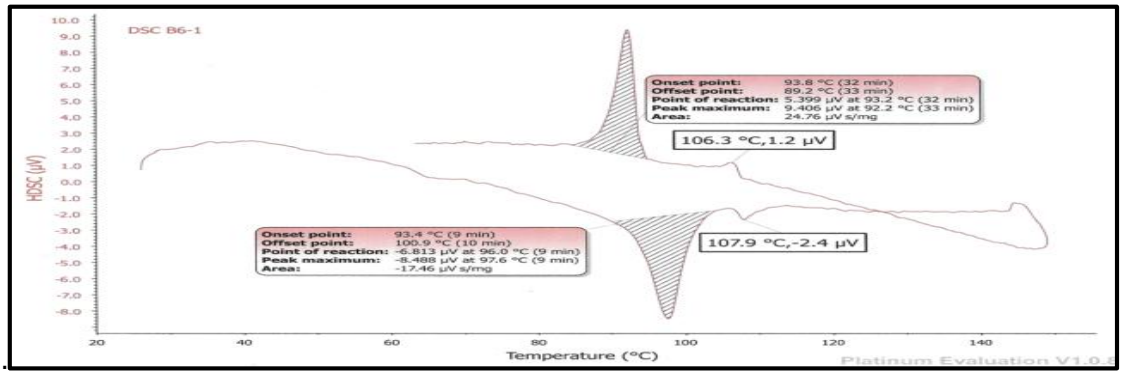


FIGURE 16 Compound 5

In the latter compound, most of the phases appeared cooling, as two nematic phases appeared at (92.2 and 94.8) °C, respectively, and the appearance of a smectic phase at 85.7 °C was due to the smectic phase.

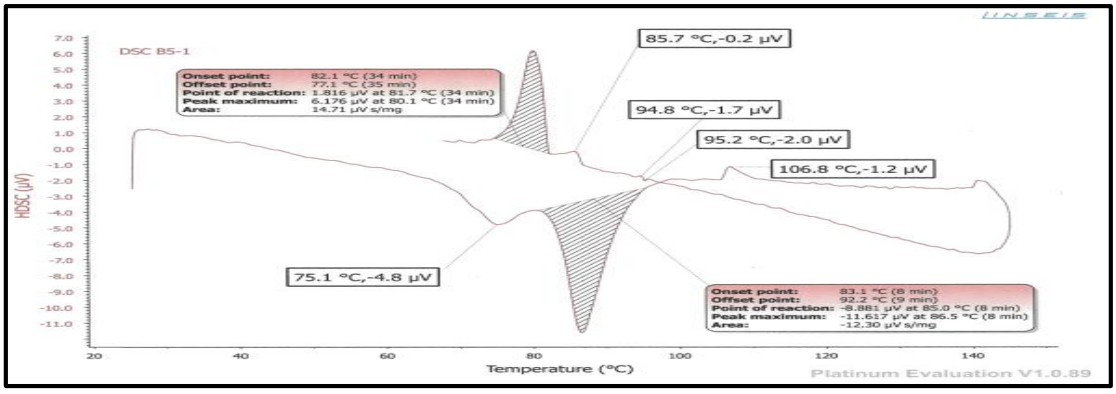


FIGURE 17 Compound 6

4- Use of electrothermal phenomenon and determination of semiconducting properties of Schiff base compounds Liquids and their polymers to enter the mesogenic field and electronic semiconductors.

Conclusions and future work

Six novel Schiff base compounds were synthesized by reacting Ampicillin with p-hydroxyl Benzaldehyde, p-chloro Benzaldehyde, and p-nitro Benzaldehyde to create 4-pentloxy, 4- hexaloxy, and 4-hepteloxy Benzaldehyde, respectively. Chemical analysis (in the form of FT-IR and H1-NMR) has confirmed the compounds' identities. The thermal properties of transition temperatures for these compounds were evaluated using a differential scanning calorimeter, and the microscopic phases were determined using a polarized light microscope (POM) (DSC). We were able to determine the melting points, hues, and yields of all compounds. Subsequent research into liquid crystalline behavior confirmed the compounds' ability to form liquid crystalline phases and helped establish whether the compounds produced nematic or smectic phases.

Through the results obtained, in addition to what was found from previous studies, it is possible to address some suggestion

1-Preparation of mesogenic polymers from bases with partial or asymmetrical compounds for each series prepared in this study.

2-Preparation of symmetric or asymmetric Schiff bases derived from Benzaldehyde by substitution of groups. Others are different at different positions concerning the Schiff base or an additional increase in the length of the molecule and study their crystal properties.

3-Preparation of mesogenic polymers by linking different monomers from the prepared chains, studying their crystal properties, and using the mixing method to find the critical or minimum point (Eutectic).

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Conflict of Interest

There are no conflicts of interests.

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