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Application of biological activity of oxazepine and 2-azetidinone compounds and study of their liquid crystalline behavior

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ABSTRACT

New Schiff bases were prepared in an acidic medium by condensation p-diamino compounds with p-hydroxy benzaldehyde and the compound formed was reacted with alkyl bromide. At finally the oxazepine and 2-azetidinone compound were prepared for azo methyl group. FT-IR and ^{1}H NMR spectrometers have confirmed the structures of such compounds. The liquid crystalline properties were studied using DSC and polarizing optical microscope. By increasing the length of the aliphatic chain, the thermal stability of the smectic liquid crystal increased with a decrease in the thermal stability of the nematic phase, Also, increasing the number of aromatic rings from three to four rings led to an increase in the thermal stability of the liquid crystalline phases due to an increase in the electronic sequence at the center core. In addition to, the oxazepine compounds did not exhibit the liquid crystalline properties, whereas the 2-azetidinone compounds showed only the nematic phase. Also, the antibacterial activities against two different types of bacteria have been studied: 1) *Escherichia coli* Gram (−) ve. 2) *Staphylococcus* Gram (+) ve. compared to the oxazepine and 2-azetidnone compounds and the Schiff bases prepared from them, it was observed that the increase in inhibition is directly proportional to the concentration. In general, the oxazepine compounds gave a high inhibition compared with the azitidine compounds, as (A14) gave the highest inhibition of (3.65 mm) against (*E. Coil*) and (A13) compound the highest inhibition (3.45 mm) against (*Staphylococcus*), Also, the compounds inhibition of (*E. coil*) bacteria were higher than that of (*staphylococcus*).

Selection and peer-review under responsibility of the scientific committee of the International Conference on Advanced Materials Behavior and Characterization.

1. Introduction

Heterocyclic compound are one of very important type of organic molecules as they have been used in different applications [1–15]. Oxazepine is a heterocyclic compound containing five carbon atoms, one oxygen atom, and one nitrogen atom. There are very broad pharmacological applications for heterogeneous organic compounds containing nitrogen in their composition [16], and among them are the oxazepine derivatives. There are three isomers of oxazepine compounds are: (1, 2), (1, 3) and (1, 4). This numbering depends on the location of the oxygen and nitrogen atoms in the seven-membered ring. The increase in the ring size makes it uneven when compared to the benzene ring. As a result, the ring takes the form of a boat in the distribution of vacuum atoms to reduce the tension on the ring to be more stable. Often classified as β-lactam, the 2-azetidinone compounds have been the building blocks for the synthesis of essential biological compounds [17,18]. Because they show a variety of microbiological activity, 2-azetidinone derivatives occupy an important role in medicinal chemistry and this is confirmed by the literature [19,20]. They also act as inhibitors of enzymes and effective in the central nervous system. [21–23]. The carbonyl derivatives of azetidines has been containing carbonyl group at the place (2). These are also known as 2-azetidinones [24]. Therefore, Schiff base and 2-azetidinone have also been synthesized of a diamine and to study the antibacterial activities of the synthesized Schiff base, 2-azetidinone and oxazepine compounds.

Calamitic or rod-like liquid crystals are substances of an elongated shape in which the molecular length is wider than the width. The molecules have effective attracting forces and appear to point in the same direction to help stable a compound structure [25]. The

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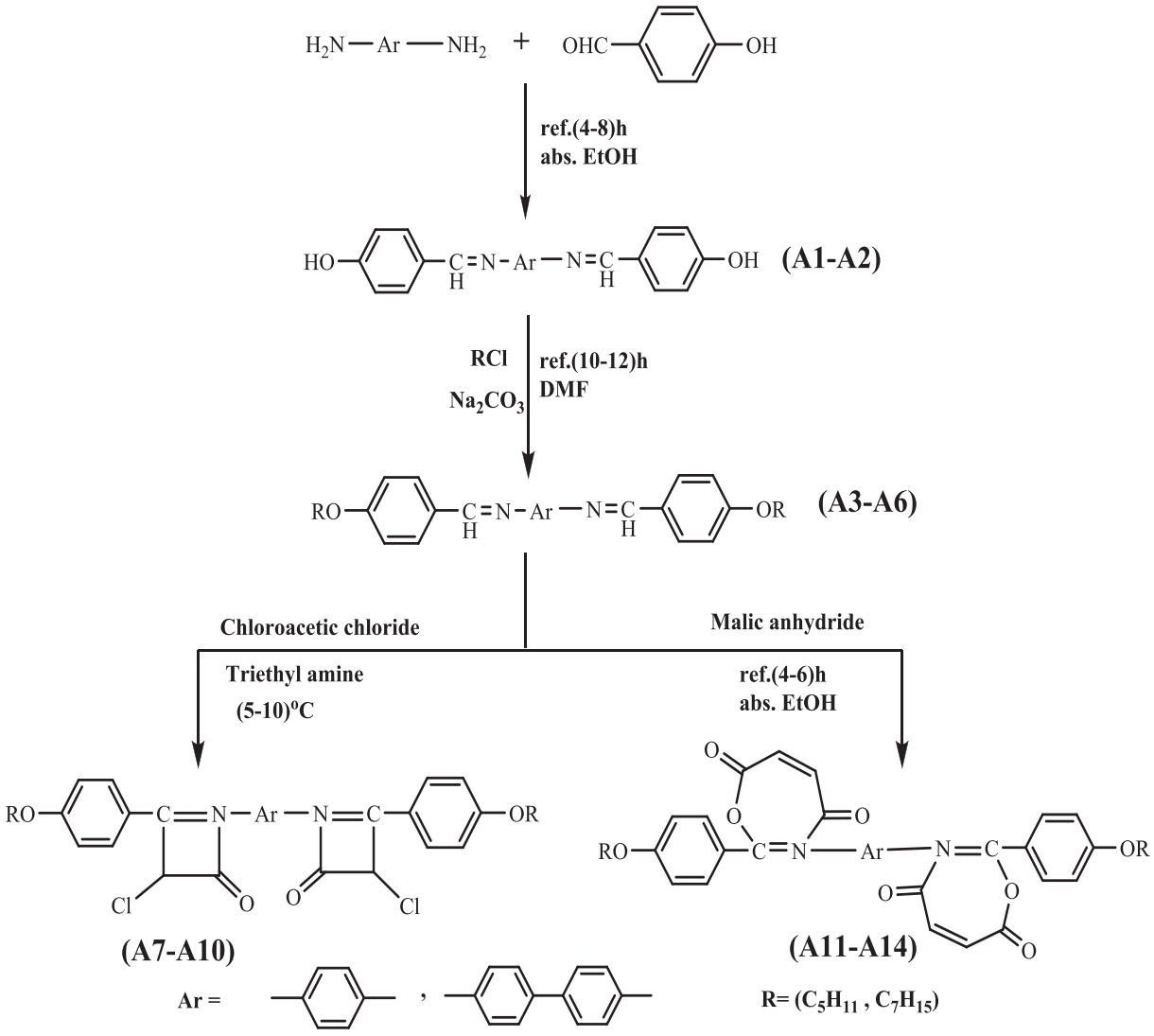


Scheme 1. Synthesis pathway for the compounds A1-A14.

main structure of LC calamite consists of two or more aromatic rings interconnected by linking units, and linked at the ends to the side chain or terminal polar groups. On the other hand, Discotic LC or disc-like molecules have a rigid core surrounded by the calamitic molecules' six side arms [26]. When molecules are oriented in a layer-like manner, a discotic nematic phase can be seen, however when the molecules are stacked into stacks, the columnar Smectic phase can be seen.

2. Experimental

2.1. Instrumentations

Melting point measurement was carried out using the Griffin-Made in Britain-London-Serial NO. 90-01-154 apparatusIR spectra were taken by means of the FT-IR 8400S (KBr) scale of Shimadzu Fourier transform infrared spectrometer (4000–400). Using Evrovector EA 3000A Italia, CHN.O was obtained. ^{1}HNMR pectrum using the Ultra Shield 300 MHZ Bruker 2003 has been registered. The liquid crystalline properties were determined by using the Shimadzu Differential Scanning Microscope Calorimeter (DSC-60) (BEAM ENGINEERS (INDIA)).

The biological activity of all the prepared compounds has been studied, and two types of pathogenic bacteria have been used in this study i) *Escherichia coli* and ii) *Staphylococci*, the study was carried out in the Department of Quality Control - Micro Biology Laboratory/ The State Company for Drugs Industry and Medical Appliances (SDI)- Samarra.

2.2. General procedure

2.2.1. Synthesis of Schiff's base from diamines (A1, A2)

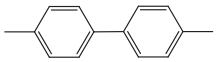
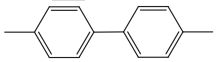
A series of Schiff's bases were prepared as shown in the equation below:

(0.01 mol) of the diamine compound was dissolved in 20 ml absolute ethanol containing few drops of glacial acetic acid as a catalyst. p-hydroxy benzaldehyde (0.02 mol) was added to the reaction mixture. It was refluxed for (4–8) hours (The reaction was monitored by TLC) cooled and then poured into crushed ice the formed precipitate was filtered and recrystallized from ethanol [27] Scheme 1.

2.2.2. Synthesis of ethers (A3-A6)

(0.01 mol) of the Schiff bases prepared in the first chain (I) were dissolved in (20 ml) DMF, afterward added (0.002 mol) of the sodium carbonate and (0.002 mol) of alkyl chloride. The reaction mixture was reflexed over an oil bath with a temperature of

Table 1
The physical properties of the prepared compounds (A7-A10).

Comp. NO.	Ar	R	mp. °C	Colour	Yield %
A7		C_5H_{11}	273–276	Yellow	60
A8			241–242	Orange	51
A9		C_7H_{15}	218–220	orange	55
A10			208–2011	Brown	49

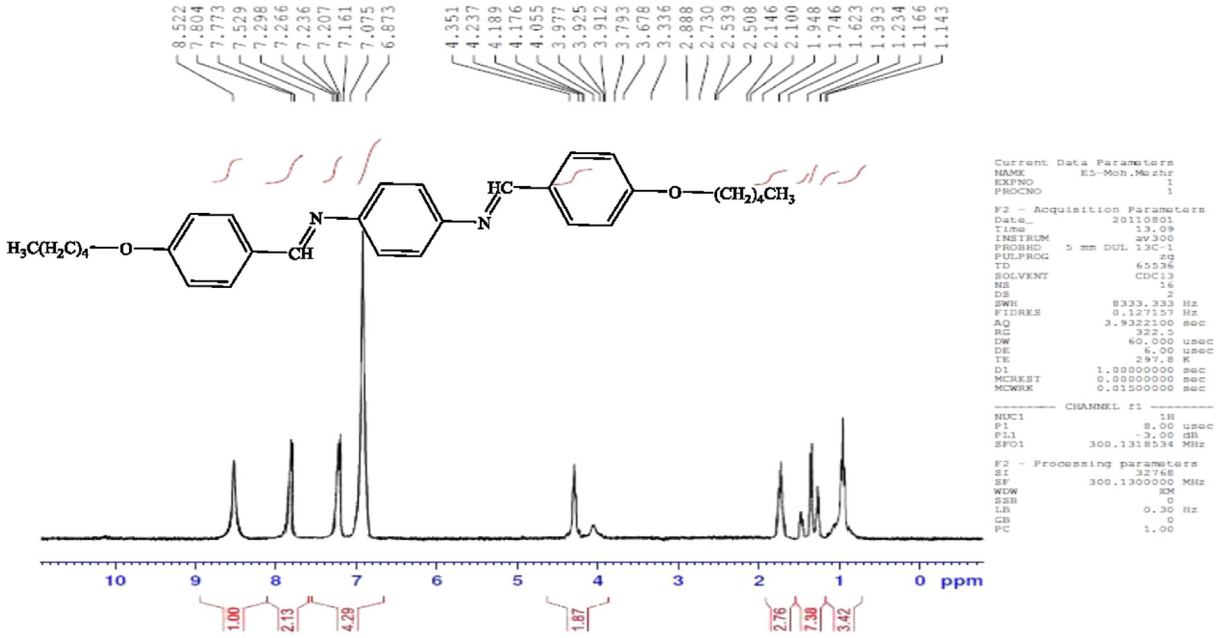


Fig. 1. ^{1}H NMR spectrum of the compound (A5).

(130 °C) for (10–12) hours, (the reaction was monitored by TLC), the solution was cooled and then added to crushed ice, a precipitate was filtered and washed well with cold water and recrystallized from DMF. The physico-chemical data for synthesized Schiff base are given in Table 1.

2.2.3. Synthesis of substituted 2-Azetidinones (A7-A10)

To a mixture of ether compounds II (0.01 mol) in dry dioxane (15 ml), triethylamine (0.025 mol), was added monochloroacetyl chloride (0.025 mol) drop-wise at (0–5) °C. The reaction mixture was stirred for three hours, afterward, the reaction mixture was poured into crushed ice, after which the Precipitated formed was dried and recrystallized from ethanol/water. The reaction was monitored by TLC [28,29].

2.2.4. Preparation of oxazepine compounds (A11-A14)

(0.001 mol) of one of the prepared ether compounds was dissolved in (20 ml) absolute ethanol and then added (0.002 mol 0.092 g) of malic anhydride, the reaction mixture was reflexed over a water bath for (3–4) hours until the reaction was completed. The reaction was monitored by TLC. The precipitate was filtered and recrystallized from DMF.

2.3. Test the bacterial activity of the prepared compounds

Two types of bacteria, Eschershia coli and Staphylococcus, were tested for the biological activity of compounds (A3-A14) using the disk diffusion method. The disks were submerged in a DMSO. Before being placed in bacterial cultures, it was then dried in an incubator. DMSO was the negative power. The plates were incubated for two days at 37 °C. The maximum inhibition zone was observed against each type of test micro-organism and measured for study purposes.

For the purposes of comparison, a standard antibiotic was used (gentamicin sulfate) which is an effective antibiotic against both gram-positive and gram-negative bacteria and has many medicinal uses [30,31].

3. Results & discussion

3.1. Diagnosis of Schiff bases

3.1.1. (4,4′-(([1,1′-biphenyl]-4,4′- diylbis(azaneylylidene))bis (methaneylylidene))diphenol) (A1)

Properties: Yield = 80%, yellow color m.p = (240–242) °C

IR (KBr, cm^{-1}): 3332 cm^{-1} (OH), 3028 cm^{-1} (C – H Aromatic); 1500/1596 cm^{-1} (C = C Aromatic); 1608 cm^{-1} (C = N).

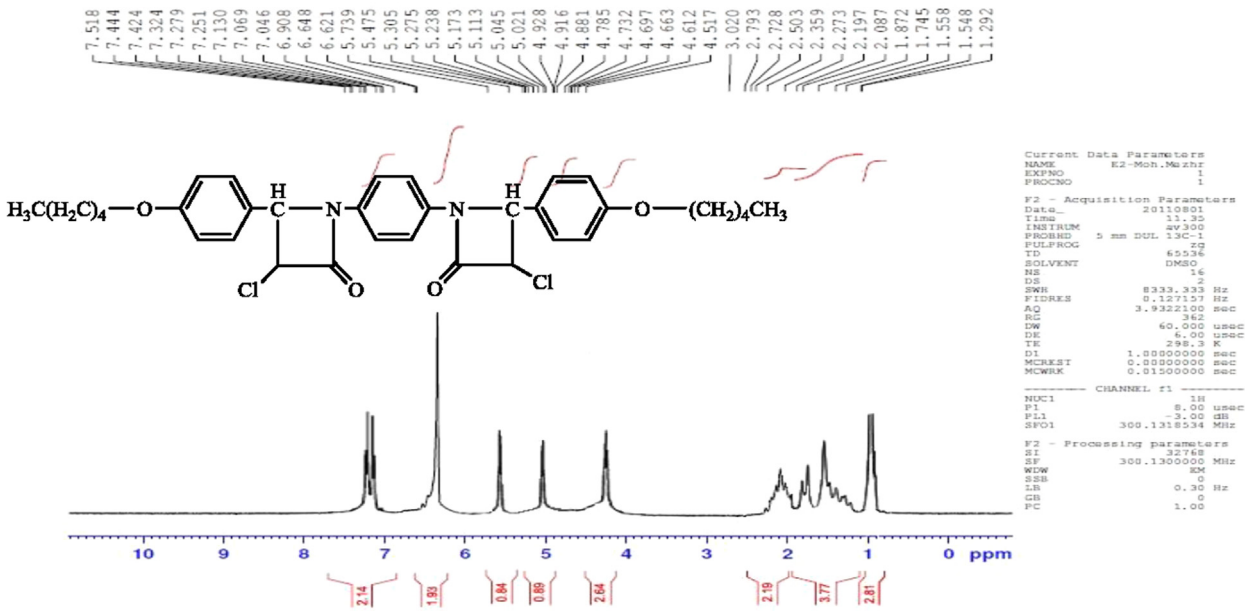


Fig. 2. ^{1}H NMR spectrum of the compound (A8).

3.1.2. 4,4′-((1,4-phenylenebis(azaneylylidene))bis(methaneylylidene)) diphenol (A2)

Properties: Yield = 78%, yellow color m.p = (192–194) °C

IR (KBr, cm^{-1}): 3317 cm^{-1} (OH); 3058 cm^{-1} (C— H Aromatic); 1510/1588 cm^{-1} (C=C Aromatic); 1612 cm^{-1} (C=N).

3.2. Diagnosis of ethers

3.2.1. N,N′-([1,1′-biphenyl]-4,4′-diyl)bis(1-(4-(pentyloxy)phenyl) methanimine) (A3)

Properties: Yield = 62%, orange color m.p = (164–165) °C

IR (KBr, cm^{-1}): 1282 cm^{-1} (R-O-Ar); 3018 cm^{-1} (C—H Aromatic); 2814 cm^{-1} (C—H aliphatic); 1497/1591 cm^{-1} (C=C Aromatic); 1610 cm^{-1} (C=N).

3.2.2. N,N′-([1,1′-biphenyl]-4,4′-diyl)bis(1-(4-(heptyloxy)phenyl) methanimine) (A4)

Properties: Yield = 69%, yellow color m.p = (126–127) °C

IR (KBr, cm^{-1}): 1259 cm^{-1} (R-O-Ar); 3012 cm^{-1} (C—H Aromatic); 2896 cm^{-1} (C—H aliphatic); 1448/1514 cm^{-1} (C=C Aromatic); 1629 cm^{-1} (C=N).

3.2.3. N,N′-(1,4-phenylene)bis(1-(4-(pentyloxy)phenyl)methanimine) (A5)

Properties: Yield = 65%, Yellow color m.p = (175–176) °C

IR (KBr, cm^{-1}): 1228 cm^{-1} (R-O-Ar); 3029 cm^{-1} (C—H Aromatic); 2879 cm^{-1} (C—H aliphatic); 1546/1579 cm^{-1} (C=C Aromatic); 1608 cm^{-1} (C=N).

^{1}H NMR (300 MHz, DMSO-d) δ/ppm: 0.90 (t, 6H, CH_3 Aliphatic proton); 1.23 (m, 8H, CH_2-CH_2 Aliphatic proton); 1.62 (m, 4H, CH_2 Aliphatic proton); 4.23 (t, 4H, CH_2 Aliphatic proton); (6.87–7.80) (m, 12H, Aromatic benzene); 8.52 (s, 2H, CH=N). As shown in Fig. 1.

3.2.4. N,N′-(1,4-phenylene)bis(1-(4-(heptyloxy)phenyl)methanimine) (A6)

Properties: Yield = 71%, Yellow greenish m.p = (124–126) °C

IR (KBr, cm^{-1}): 1240 cm^{-1} (R-O-Ar); 3045 cm^{-1} (C—H Aromatic); 2931 cm^{-1} (C—H aliphatic); 1485/1595 cm^{-1} (C=C Aromatic); 1623 cm^{-1} (C=N).

3.3. Diagnosis of 2-azetidinone compounds

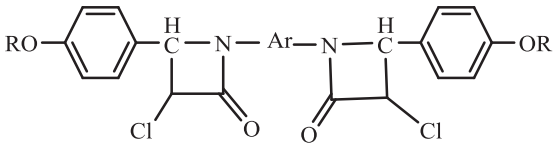
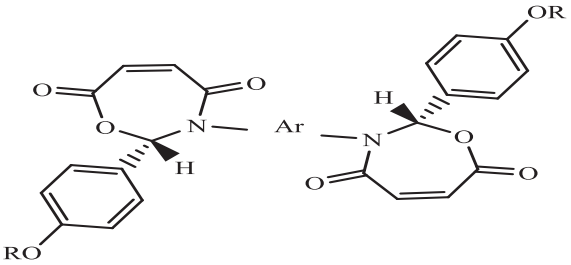


Table 2.
Figs. 4–12

3.4. Diagnosis of Oxazepine Compounds



Tables 3 and 4

3.5. Diagnosis and discussion of liquid crystal phases

Thermal transition values were calculated by taking about 2–3 mg of dry matter and heating it in an inert nitrogen gas atmosphere for liquid crystals and isotropic phases by using DSC Heating with a polarized microscope was diagnosed the liquid crystals

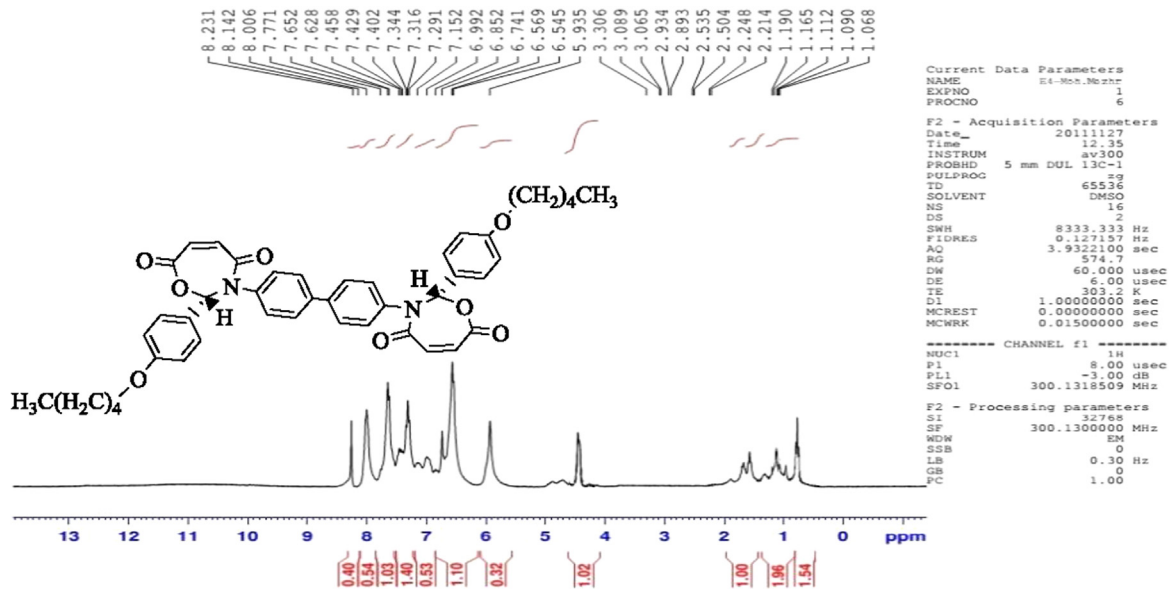
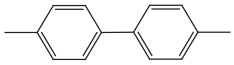
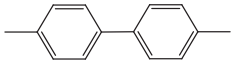


Fig. 3. ^{1}H NMR spectrum of the compound (A11).

Table 2
Results of the FT-IR (cm^{-1}) for the prepared compounds.

Comp. NO.	Ar	R	I.R. – KBr, υ – cm^{-1}			
			C=O	C=C Ar.	C—HAlf.	C—HAr.
A7		C_7H_{15}	1699.17	1542.95 1508.23	2991.80	3083.96
A8			1709.69	1591.47 1492.33	2927.69	3012.08
A9		C_5H_{11}	1693.38	1564.16 1519.80	2889.52	3056.96
A10			1695.13	1566.09 1519.80	2867.95	3058.89

H-NMR of compound 1,1′-(1,4-phenylene)bis(3-chloro-4-(4-(pentyloxy)phenyl)-1l4-azet-2(3H)-one) (A8) was studied and the following results were shown:
H NMR (300 MHz, DMSO-d) δ/ppm: 0.92 (s, 6H, CH_3 Aliphatic proton); 1.55 (m, 8H, CH_2 Aliphatic proton); 2.08 (s, 4H, CH_2 Aliphatic proton); 4.23 (m, 4H, CH_2 Aliphatic proton); 6.64–7.51 (m, 12H, Aromatic benzene); 5.02 (d, 2H, CH-N); 5.73 (d, 2H, CH-Cl). As shown in Fig. 2.

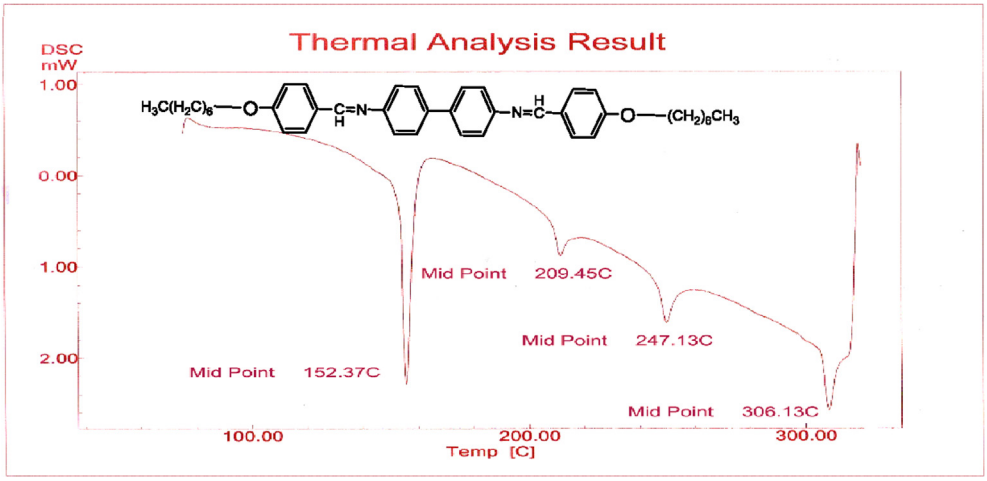


Fig. 4. The DSC of the A4 compound.

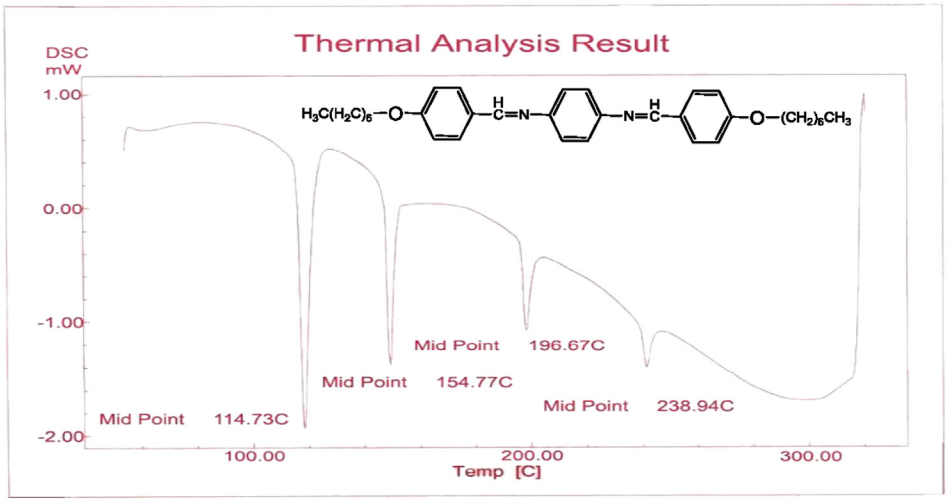


Fig. 5. The DSC of the A6 compound.

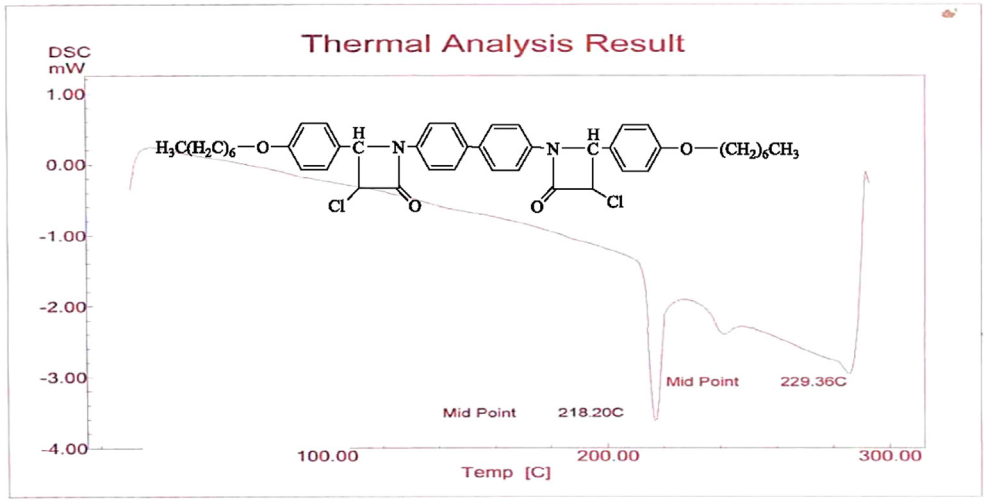


Fig. 6. The DSC of the A9compound.

Fig. 7. S_H of A6 compound.

Fig. 8. S_A of A4 compound.

Fig. 9. S_G of A6 compound.

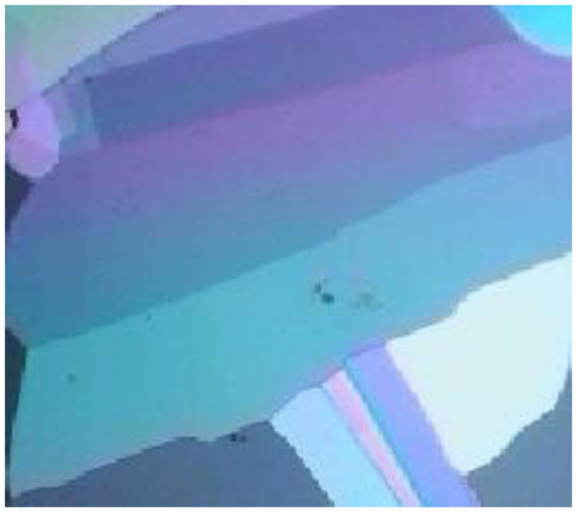


Fig. 10. S_G of A6 compound.

Fig. 11. N of A8 compound.

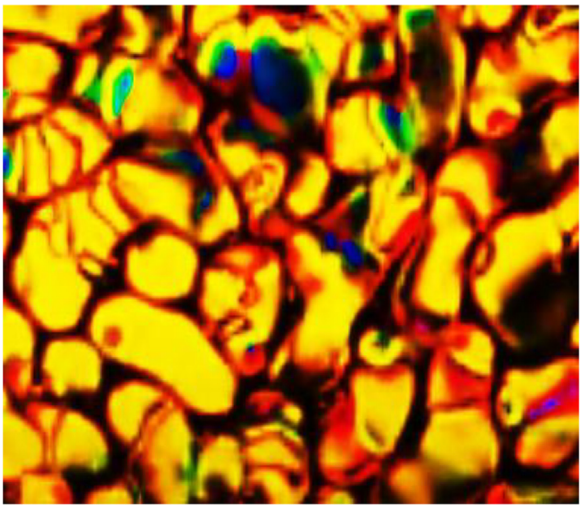


Fig. 12. N of A9 compound.

and it was found that the microscope and DSC values were similar, as seen in Tables 5–7.

Schiff bases showed nematic and smectic phases with thermal stability This is due to the presence of more than one aromatic ring in The chemical structure of those substances, Which provides the molecule with the necessary elongation, in addition to the electronic sequence along the axis of the molecule, which leads to an increase in the variation in the molecular polarization resulting from the terminal attraction forces of the dipole–dipole type, which includes the attraction between alkoxy groups, also, there is a polarization of the azomethene group (C═N).

It has been observed that in oxazepine compounds, the liquid crystal phases do not appear due to the loss of the plane property, which is considered the most important component of the appearance of liquid crystalline properties, Also, the 2-Azetidinone compounds showed the nematic phase only with little thermal stability, the explanation for this is due to the presence of the 2-aztedinone, which weakens the lateral forces and affects the ratio of the length of the molecule to its width.

3.6. Evaluation of biological activity

The biological activity of the prepared compounds was evaluated on two types of bacteria, E. coli. And Staphylococcus, which is negative and positive for gram respectively. By observing the results and comparing the effect of the two types of bacteria on the prepared compounds, it was revealed that there is a varying effect of those compounds on inhibiting the growth of bacteria with different proportions. Gentamycin sulphate was used as a type of antibiotic for comparison in this study because it is considered effective on gram-positive and gram-negative bacteria [17]. The results of inhibition zone in millimeter are shown in Table 8, see Scheme 2 and 3.

4. Conclusion

The liquid crystalline properties were studied using (DSC) and polarizing optical microscope. By increasing the length of the aliphatic chain, the thermal stability of the smectic liquid crystal phases increased with a decrease in the thermal stability of the nematic phase. In addition to, the oxazepine compounds did not exhibit the liquid crystalline properties, whereas the 2-azetidinone compounds showed only the nematic phase. Also, the antibacterial activities against two different types of bacteria

Table 3
The physical properties of the prepared compounds (A11-A14).

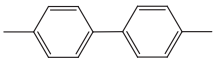
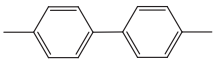
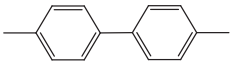
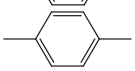
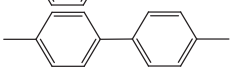
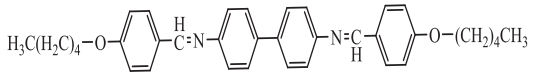
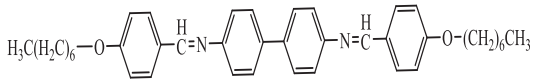
R	Comp. NO.	Ar	mp. °C	Colour	Yield %
C_5H_{11}	**A11**		289–291	Yellow	58
	A12		265–268	Deep orange	51
C_7H_{15}	**A13**		251–253	Orange	53
	A14		237–240	Brown	48

Table 4
Results of the FT-IR (cm^{-1}) for the prepared compounds.

Comp. NO.	Ar	R	I.R. – KBr , υ – cm^{-1}				
			C=O Amide	C=O Ester	C=C Ar.	C—H Alf.	C—H Ar.
A11		C_7H_{15}	1625.24	1697.24	1562.23 1496.66	2987.53	3062.75
A12			1670.24	1710.74	1595.02 1535.23	2993.32	3053.11
A13		C_5H_{11}	1662.52	1685.67	1593.09 1527.52	2937.38	3099.39
A14			1625.88	1695.31	1566.09 1521.73	2866.02	3045.39

H-NMR of compound (2S,2′S)-3,3′-([1,1′-biphenyl]-4,4′-diyl)bis(2-(4-(pentyloxy)phenyl)-2,3-dihydro-1,3-oxazepine-4,7-dione) (A11) was studied and the following results were shown:
H- NMR (300 MHz, DMSO-d) δ/ppm: 0.83 (s, 6H, CH_3 Aliphatic proton); 1.12 (m, 8H, CH_2 Aliphatic proton); 1.75 (s, 4H, CH_2 Aliphatic proton); 4.50 (m, 4H, CH_2 Aliphatic proton); 5.93 (s, 2H, CH oxazepine proton); 6.54 (s, 2H, CH oxazepine proton); 6.64–7.51 (m, 12H, Aromatic benzene); 8.23 (s, 2H, CH-N. As shown in Fig. 3.

Table 5
Liquid crystal phase transitions in DSC and Mic.sc. for ether compounds.

App.	No.	C	S_1	S_A	N	ΔT_{S1}	ΔT_{SA}	ΔT_N	Structure
DSC	A3								
Mic.sc		265	283		390	18		107	
DSC	A4	153	209	247	306	56	38	59	
Mic.sc		158	211	249	309	53	38	60	

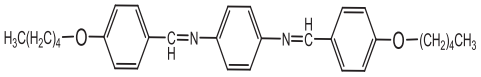
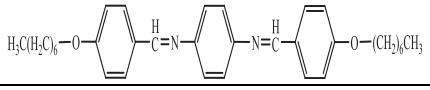
App	No.	C	S_H	S_G	S_C	S_A	N	ΔT_{SH}	ΔT_{SG}	ΔT_{SC}	ΔT_{SA}	ΔT_N	Structure
DSC	A5												
Mic.sc		175				192	271				17	79	
DSC	A6	114	154			196	238	40			42	42	
Mic.sc		125	156	162	169	198	241	7	6	7	29	43	

Table 6
Liquid crystal phase transitions in DSC and Mic.sc. 2-azetidinone compounds.

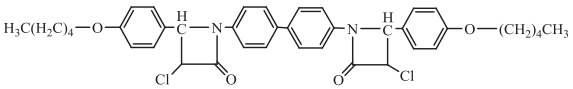
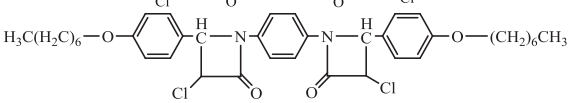
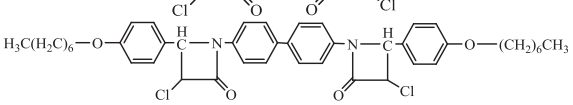
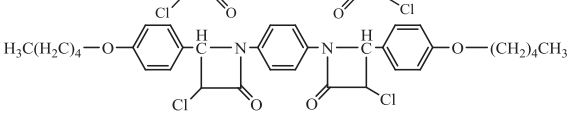
App.	No.	C	N	ΔT_N	Structure
DSC	A7	273			
Mic.sc					
DSC	A8	208			
Mic.sc		206	221	15	
DSC	A9	218	229	11	
Mic.sc		220	232	12	
DSC	A10	241			
Mic.sc					

Table 7
Liquid crystal phase transitions in DSC and Mic.sc. *Oxazepine compounds.*

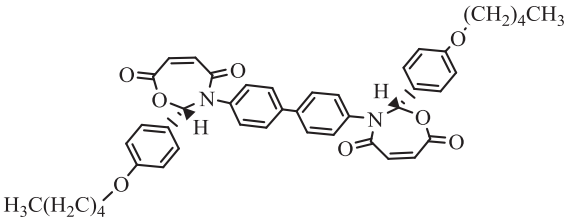
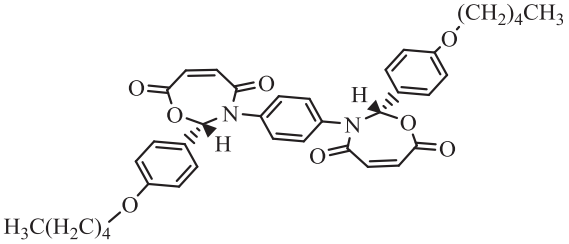
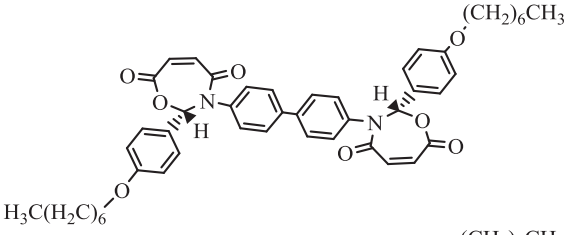
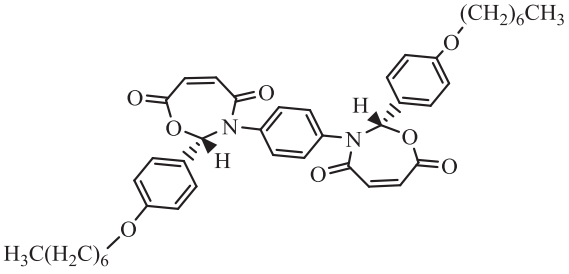
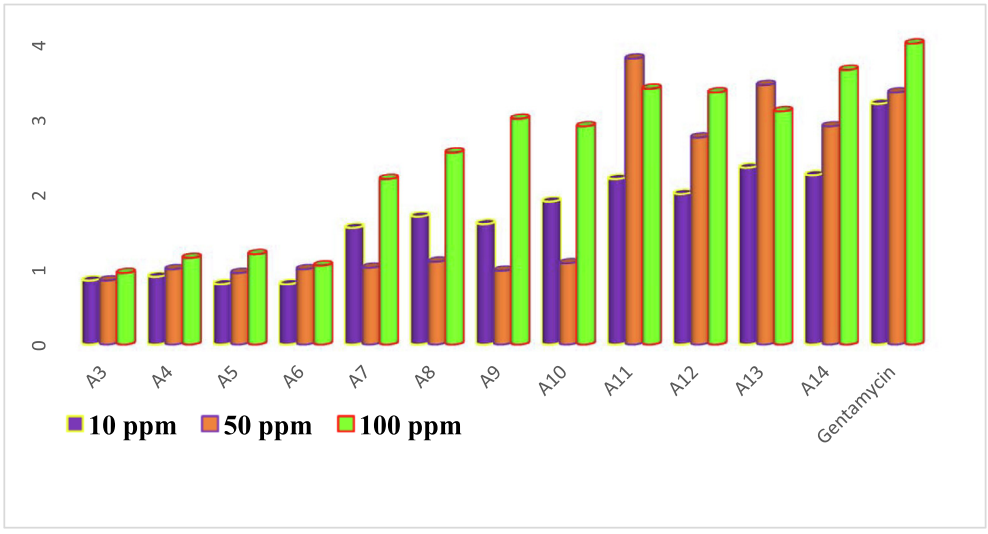
App.	No.	C	Structure
DSC Mic.sc	A11	289	
DSC Mic.sc	A12	265	
DSC Mic.sc	A13	251	
DSC Mic.sc	A14	237	

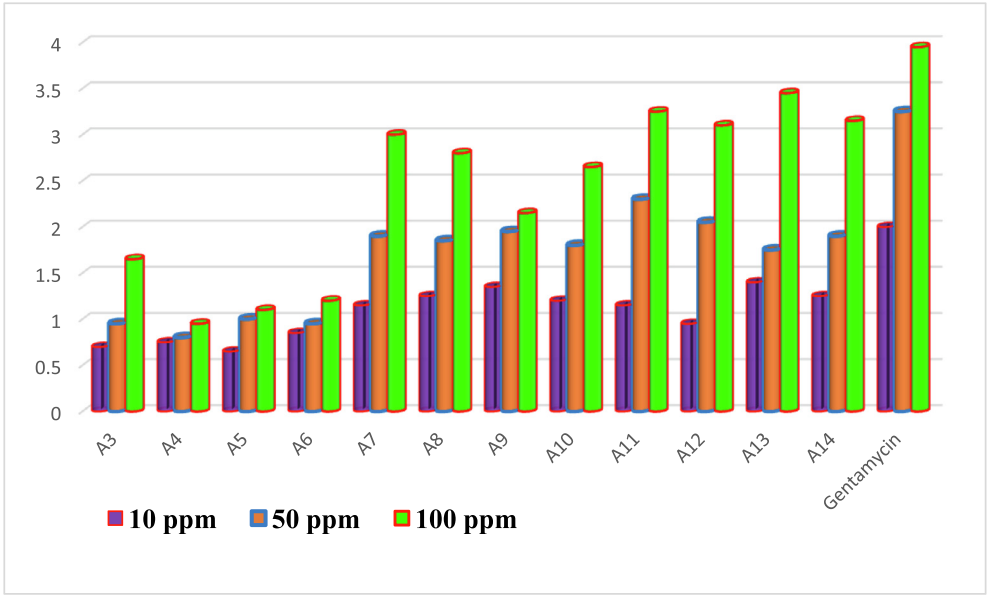
Table 8
Antibacterial activity of the prepared compounds (A3-A14) with control antibiotic.

Comp. No.	*E. Coil* Conc. mg/ml			Staphylococcus Conc. mg/ml		
	10	50	100	10	50	100
A3	0.85	0.85	0.95	0.70	0.95	1.65
A4	0.90	1.00	1.15	0.75	0.80	0.95
A5	0.80	0.95	1.20	0.65	1.00	1.10
A6	0.80	1.00	1.05	0.85	0.95	1.20
A7	1.55	1.02	2.20	1.15	1.90	3.00
A8	1.70	1.10	2.55	1.25	1.85	2.80
A9	1.60	0.98	3.00	1.35	1.95	2.15
A10	1.90	1.08	2.90	1.20	1.80	2.65
A11	2.20	3.80	3.40	1.15	2.30	3.25
A12	2.00	2.75	3.35	0.95	2.05	3.10
A13	2.35	3.45	3.10	1.40	1.75	3.45
A14	2.25	2.90	3.65	1.25	1.90	3.15
Gentamycin	3.20	3.35	4.00	2.00	3.25	3.95
Blank disk	0	0	0	0	0	0

have been studied: 1) Escherichia coli Gram (−) ve. 2) Staphylococcus Gram (+) ve. Compared to the 2-azetidnone compounds and the Schiff bases prepared from them, the oxazepine compounds were found to have the highest inhibition.



Scheme 2. Assessment of inhibitory activity of *E. coli* compounds prepared with a control antibiotic.



Scheme 3. Assessment of inhibitory activity of Staphylococcus compounds prepared with a control antibiotic.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Further Reading

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