

How to Cite:

Hussain, N. B., & Karam, N. H. (2022). Heterocyclic compounds-based liquid crystals: Synthesis and mesomorphic properties. *International Journal of Health Sciences*, 6(S10), 609–619. <https://doi.org/10.53730/ijhs.v6nS10.13557>

Heterocyclic compounds-based liquid crystals: Synthesis and mesomorphic properties

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Abstract---A series of liquid crystals comprising a heterocyclic dihydro pyrrole and 1,2,3-triazole rings [VII]-[X] were synthesized by many steps starting from a reaction of 3,3'-dimethyl-[1,1'-biphenyl]-4,4'-diamine with chloroacetyl chloride in a mixture of solutions DMF and TEA to synthesise the compounds [I], then the compounds [I] reacted with malononitrile in 1,4-dioxane and TEA solutions to produce compounds [II], then the first step is repeated with compound [II] where it reacted with chloroacetyl chloride in mixture of DMF and TEA to give compound [III], this compound reacted with sodium azide in the presence of sodium chloride and DMF as solvent to produce the compound [IV], which reacted with acrylic acid by a 1.3 dipolar reaction in solvent THF to give compound [V], this compound which reacted with thionyl chloride to gave carbonyl chlorides compound [VI], finally the last compound reacted with a variety of amines to product compounds [VII]a-d. The structures of the synthesized compounds were actually determined by Fourier-transform infrared spectroscopy FT-IR and Proton nuclear magnetic resonance ¹H-NMR, The properties of liquid crystalline were characterized by hot stage Polarising Optical Microscope POM. The synthesized molecules exhibited enantiotropic liquid crystal phases, and the compounds [VII]a-d exhibited an enantiotropic nematic phase only.

Keywords---liquid crystal, mesomorphic, heterocyclics, pyrrole.

Introduction

Liquid crystals are fascinating materials with intermediate properties between solid and liquid materials. As it is well known that molecular shape has a dominant influence on the existence of the liquid crystalline state, over many decades, an enormous number of liquid-crystalline materials containing heterocyclic units have been synthesized. This research field has grown even more in the past few years because of modifications in synthetic methodologies (Al-Humaidi, Alissa, Katariya, Abu Al-Ola, Hagar, & Khalil, 2021). Heterocycles are of significant importance as core units in thermotropic liquid crystals because of their ability to impart lateral or longitudinal dipoles combined with modification of molecular forms and properties, phase structure, and geometry of prepared compounds (Ghosh & Lehmann, 2013). Also, the incorporation of heteroatoms can result in large changes in the corresponding liquid-crystalline phases and/or in the physical properties of the observed phases, because the heteroatoms commonly introduced (N, O, and S) are chemically classified as more polarisable than a carbon atom (Gallardo, Bortoluzzi, & De Oliveira Santos, 2008).

So the presence of heteroatoms (N, O, and S) can significantly change the polarity, polarisability, and sometimes the geometric form of the molecule, thus affecting the type of mesophase, the phase transition temperatures, dielectric and other properties of the mesogens (Ong, Ha, Yeap, & Lin, 2018). Some of the heterocyclic rings used as a core system in the liquid crystalline molecules include pyrrole (Irfan, Sumra, Zhang, Song, Liu, & Zeng, 2021), imidazoles (Guo, Yu, Xiong, & Yang, 2020), benzimidazoles (Du, et al., 2020), isoxazoles (Hernandez, Burchell-Reyes, Braga, Lopez, & Forgione, 2022), pyrazoles (Dash & Karim, 2021), thiazoles (Santos, et al., 2020), 1,2,3- and 1,2,4- triazoles (Tomi , Al-Daraji , Al-Qaisi , Al-Heetimi , & Al-Qaisi, 2016), 1,2,4- and 1,3,4-oxadiazoles (Fouad , Ness , Wang , Ruth , Britton, & Twieg, 2019), (Fouad , Khairuddean, & Twieg, 2021).

More of the five-membered heterocycles, such as pyrroles and 1,2,3-triazoles and their derivatives are the popular heterocycles in liquid crystalline, where they have received more attention than other heterocycles in studies of bent-core liquid crystalline systems, because this heterocycle can be conveniently and efficiently obtained, in addition, the heterocycles themselves have demonstrated wide utility in materials chemistry as dyes, corrosion inhibitors, photo stabilisers, and photographic materials (Mandal, et al., 2021). Many methods in the literature for the preparation of 1,2,3- triazole compounds include the cyclization of azido acetamide compounds by 1,3-dipolar cycloaddition reaction (Maiuolo, et al., 2019), (Wang, Sprunt, & Twieg, 2018). A large number of 1,2,3-triazoles synthesized via 1,3-dipolar cycloadditions organic azides and acrylic acid with azides.

In general, the cycloaddition reaction when the acrylic acid has electron withdrawing was faster, but on azides, the effect of these groups have the opposite, also the bulky substituents hinder the reaction, but the important thing is the selectivity (REZKI & AOUD, 2017), (Shaabani, Mofakham, Mousavi , Mousavi , & Pedarpour , 2017). This paper discusses the synthesis and study of liquid crystalline behaviour containing dihydro pyrrole and 1,2,3-triazole

heterocyclic rings, which which helped to understand the effect of substitution of the terminal groups on mesomorphism. And we synthesized these compounds to study the effect of the lateral, and terminal groups, and central linkages on mesomorphism and thermal stability of these compounds.

Experimental

Materials

The materials were taken from Aldrich, Fluka, and Merck Co.

Techniques

FT-IR spectra recorded by SHIMADZU (IR Affinity - 1) FT-IR spectrometer in the wave number range 4000 - 600 cm^{-1} . $^1\text{H-NMR}$ spectra have measured by the company, broker, at 500 MHz and were reported in ppm (δ), the compounds were dissolved in $\text{DMSO} - d_6$ solution with the TMS as the internal standard. The melting point measurement by Gallen Kamp apparatus. Also, liquid crystalline properties were investigated by using the POM model Leica DM2500 M.

Synthesis

All the new compounds were synthesized by using the conventional methodologies in the references (Ibraheem & Karam, 2018), (Karam, Jber , & Al-Dujaili, 2019), (Karam & Ibraheem, 2018) according to the synthetic route outlined in Scheme (1).

Synthesis of **$\text{N,N}'$ -(3,3'-dimethyl-[1,1'-biphenyl]-4,4'-diyl)bis(2-chloroacetamide) [I]**

A mixture of 3,3'-dimethyl-[1,1'-biphenyl]-4,4'-diamine (0.212 gm, 0.001mol), was mixed with triethylamine (TEA) (18mL) and chloroacetylchloride (0.226 gm, 0.002 mmol) in DMF (15 ml), and stirred in an ice bath for 3 hours, then cooling the mixture by adding to ice water, then filtration dried and crystallized from ethanol. The compound [I], has a brown colour, and yields 74 %, MP = 213–215 °C.

Synthesis of **1,1'-(3,3'-dimethyl-[1,1'-biphenyl]-4,4'-diyl)bis(2-amino-5-oxo-4,5-dihydro-1H-pyrrole-3-carbonitrile) [II]**

Malononitrile (0.132 g, 0.002 mol) and (5mL) dioxane were added dropwise and stirred, that mixed in an ice bath with a mixture from compound [I] (0.365 g, 0.001 mol) and triethylamine (0.5 mL) in dioxane (30 mL). Then the reaction mixture was heated at reflux for 6 hours, then cooled at room temperature. The product was filtered off and recrystallized from ethanol to give the required compound. The compound [II] has a dark brown colour, and yields 85%, m.p = 248 - 250 °C.

Synthesis of N,N'-(((3,3'-dimethyl-[1,1'-biphenyl]-4,4'-diyl)bis(3-cyano-5-oxo-4,5-dihydro-1H-pyrrole-1,2-diyl))bis(2-chloroacetamide) [III]

A solution of compound [II] (0.424gm, 0.001 mol) in DMF (15mL) was mixed with triethyl amine (TEA) (18mL) and chloroacetylchloride (0.226 gm, 0.002 mol), then stirred on an ice bath for 3 hours. Then added the mixture to ice water, then filtration dried and recrystallized from ethanol. The compound [III] has a dark brown colour, and yields 70 %, m.p = 274 - 276 °C.

Synthesis of N,N'-(((3,3'-dimethyl-[1,1'-biphenyl]-4,4'-diyl)bis(3-cyano-5-oxo-4,5-dihydro-1H-pyrrole-1,2-diyl))bis(2-azidoacetamide) [IV]

A solution of compound [III] (0.577gm, 0.001mol) in (6 mL) DMF was added sodium azide (0.13 gm, 0.002mol) and ammonium chloride (0.107gm, 0.002mol). The mixture was refluxed for 4 hours with stirring. Then, the resultant solution was cooled in ice water and the product obtained was collected. The compound [IV] has a dark brown colour, and yields 68 %, m.p = 303 - 305 °C.

Synthesis of 1,1'-((((3,3'-dimethyl-[1,1'-biphenyl]-4,4'-diyl)bis(3-cyano-5-oxo-4,5-dihydro-1H-pyrrole-1,2-diyl))bis(azanediyl))bis(2-oxoethane-2,1-diyl))bis(4,5-dihydro-1H-1,2,3-triazole-4-carboxylic acid) [V]

A mixture of compound [IV] (0.59 gm, 0.001 mol) and acrylic acid (0.144 gm, 0.002 mol) in THF (5 mL) was refluxed for 24 hours. Then the solvent was evaporated under reduced pressure. The resulting precipitate was dried and recrystallized from ethanol. The compound [V] has a dark brown colour, and yields 81 %, m.p = 315 - 317 °C.

Synthesis of 1,1'-((((3,3'-dimethyl-[1,1'-biphenyl]-4,4'-diyl)bis(3-cyano-5-oxo-4,5-dihydro-1H-pyrrole-1,2-diyl))bis(azanediyl))bis(2-oxoethane-2,1-diyl))bis(4,5-dihydro-1H-1,2,3-triazole-4-carbonyl chloride) [VI]

The compound [V] (0.734gm, 0.001mol) was added to (0.238 gm, 0.002 mol) thionylchloride SOCl₂ in (3 mL) DMF. The reaction mixture refluxed for 3 hours, then cooled the resulting at room temperature, and recrystallized from ethanol. The compound [VI] has a dark brown colour, and yields 89%, m.p = 257 - 260 °C.

Synthesis of amide derivatives [VII]a-d

Different aromatic amines were added to a mixture of (1 mL) pyridine and (2 mL) DMF, in the ice bath, then added compound [VI] and stirred for 4 hours, after that, added hydrochloric acid (10%), then filtered and washed with water, then dried and recrystallized from ethanol. The resulting compounds have different nomenclature structural formulas, yields, and physical properties, as shown in Table (1).

Results and Discussions

The compound [I] was synthesized from a reaction of 3,3'-dimethyl-[1,1'-biphenyl]-4,4'-diamine with chloroacetylchloride in DMF and TEA. The FTIR

spectrum of the N-chloroacetyl compound showed disappearance absorbance bands of NH₂ for starting material. Bands observed at 3263 cm⁻¹, 1666 cm⁻¹, and 810 cm⁻¹ were assigned to NH, ν C=O amide, and ν C-Cl groups, respectively. ¹HNMR (300 MHz, DMSO- d₆) δ (ppm) : 9.68 (s, 2H of 2NH), 7.47-7.53 (m, 6H, Ar-H), 4.32 (s, 4H, 2(CH₂-Cl)), 2.26 (s, 6H, 2CH₃).

Table 1
The Nomenclature, yields, and physical properties of compounds [VII] a-d

Comp. No.	Nomenclature	X	Yield %	colour	M. (°C)	P
[VII] a	1,1'-((((3,3'-dimethyl-[1,1'-biphenyl]-4,4'-diyl)bis(3-cyano-5-oxo-4,5-dihydro-1H-pyrrole-1,2-diyl))bis(azanediyl))bis(2-oxoethane-2,1-diyl))bis(N-(p-tolyl)-4,5-dihydro-1H-1,2,3-triazole-4-carboxamide)	CH ₃	87 %	Dark brown	314-316	
[VII] b	1,1'-((((3,3'-dimethyl-[1,1'-biphenyl]-4,4'-diyl)bis(3-cyano-5-oxo-4,5-dihydro-1H-pyrrole-1,2-diyl))bis(azanediyl))bis(2-oxoethane-2,1-diyl))bis(N-(4-methoxyphenyl)-4,5-dihydro-1H-1,2,3-triazole-4-carboxamide)	CH ₃ O	85 %	Dark brown	226-228	
[VII] c	1,1'-((((3,3'-dimethyl-[1,1'-biphenyl]-4,4'-diyl)bis(3-cyano-5-oxo-4,5-dihydro-1H-pyrrole-1,2-diyl))bis(azanediyl))bis(2-oxoethane-2,1-diyl))bis(N-(4-nitrophenyl)-4,5-dihydro-1H-1,2,3-triazole-4-carboxamide)	NO ₂	88 %	Dark brown	310-312	
[VII] d	1,1'-((((3,3'-dimethyl-[1,1'-biphenyl]-4,4'-diyl)bis(3-cyano-5-oxo-4,5-dihydro-1H-pyrrole-1,2-diyl))bis(azanediyl))bis(2-oxoethane-2,1-diyl))bis(N-(4-hydroxyphenyl)-4,5-dihydro-1H-1,2,3-triazole-4-carboxamide)	OH	83 %	Dark brown	323-325	

The reaction of compound [I] and malononitrile with dioxane and triethylamine gave compound [II] 1,1'-(3,3'-dimethyl-[1,1'-biphenyl]-4,4'-diyl)bis(2-amino-5-oxo-4,5-dihydro-1H-pyrrole-3-carbonitrile). The FTIR spectrum of compound [II] showed characteristic absorption bands (NH₂), (CN) and (C=O) functional groups at (3271-3116) cm⁻¹, 2337 cm⁻¹ and 1666 cm⁻¹, respectively. ¹HNMR (300 MHz, DMSO- d₆) δ (ppm): 7.53-6.50 (m, 6H, Ar-H), 4.32 (s, 4H, 2 NH₂), 3.52 (s, 2CH₂ in cyclic), 1.28 (s, 6H, 2CH₃). In order to obtain the compound [III], we reacted compound [II] with chloroacetyl chloride in DMF and TEA, the FTIR spectrum of compound [III] observed bands at 1666cm⁻¹ and 791 cm⁻¹ assigned to ν C=O amide and ν C-Cl group, respectively. ¹HNMR (400 MHz, DMSO- d₆) δ (ppm)

: 9.68 (s, 2H, 2NH), 7.53 - 7.47 (m, 6H, Ar-H), 4.32 (s, 4H, 2 CH₂ in cyclic), 3.52 (s, 4H, 2 CH₂Cl), 1.28 (s, 6H, 2CH₃).

The reaction of compound [III] (1mmol) with sodium azide (2mmol) and ammonium chloride in DMF produced a compound [IV]. The FTIR spectrum of this compound showed stretching, vibration in 2198 cm⁻¹ for (N≡N) group. In the later compound [IV] reaction with two moles of acrylic acid to product compound [V], the FTIR spectrum appearance bands at 3278 cm⁻¹, 1666 cm⁻¹ were assigned to OH and C=O carboxyl groups, respectively. ¹HNMR (400 MHz, DMSO- d⁶) δ (ppm): 10, 17 (s, 2H, 2OH), 7.56-7.38 (m, 6H, Ar-H), 4.26 (s, 4H, 2 CH₂ in cyclic), 4.24 (s, 4H, 2 CH₂ in cyclic), 3.36 (s, 4H, 2 CH₂), 1.23 (s, 6H, 2CH₃). Then carboxylic acid compound [V] reacted with SOCl₂ to obtain acid chloride [VI], the FTIR spectrum display band at 1750 cm⁻¹ of carbonyl acid chloride. Finally amide compounds [VII] a-d product from reactive compound [VI] with different aromatic amines in pyridine medium with TEA. The characterization of FTIR absorption band values for these compounds were listed in Table (2).

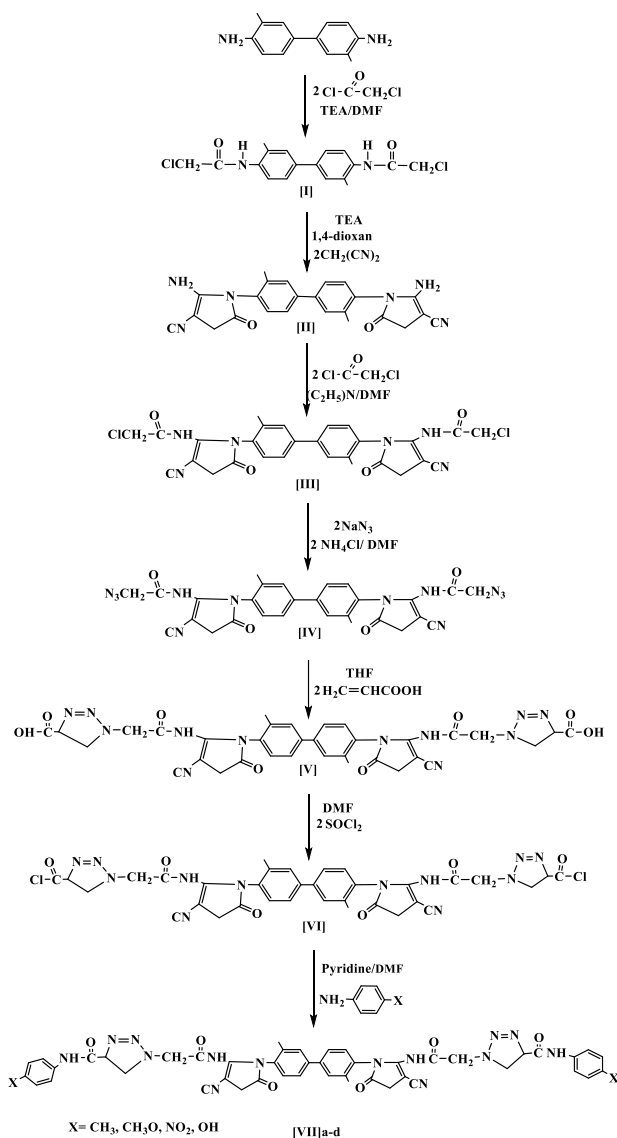
Scheme 1. The synthetic route of compounds [I]-[VII]_{a-d}

Table 2
The characterization of FTIR absorption bands of compounds [VII]_{a-d}

Comp. No.	Characteristic bands FT-IR spectra (cm ⁻¹)						
	ν N-H	ν C-H arom.	ν C-H aliph.	ν C≡N	ν C=O	ν C=C arom.	Other
[VII] a	3363	3041	2916-2854	2337	1687	1624	-
[VII] b	3394	3070	2923-2874	2337	1697	1619	ν C-O 1249
[VII] c	3386	3085	2916-2854	2337	1708	1605	ν NO ₂ 1554
[VII] d	3394	3048	2916-2854	2337	1697	1620	ν OH

Liquid crystalline properties

The textures and phase-transition temperature behaviour of the novel liner-shaped compounds were evaluated using a polarizing optical microscope (POM). In Table (3) the transition temperature values were presented and identified the texture of the mesophase by microscopy studies using the classification systems reported by Richter (Richter, 1980) and Gray & Goodby (Gray & Goodby, 1984).

Table 3
The phase transition temperatures for compounds [VII] a-d

Comp. No.	Phase transition
[VII] a	Cr $\xrightleftharpoons{230}$ N $\xrightleftharpoons{370}$ I
[VII] b	Cr $\xrightleftharpoons{220}$ N $\xrightleftharpoons{350}$ I
[VII] c	Cr $\xrightleftharpoons{250}$ N $\xrightleftharpoons{380}$ I
[VII] d	Cr $\xrightleftharpoons{100}$ N $\xrightleftharpoons{290}$ I

Cr, Crystalline Phases, N, Nematic phase, and I, Isotropic Liquid.

In the present work, we investigate the liquid crystal properties of the compounds [VII] a-d that bear four amide groups and the correlation between the properties of the amide functional groups and their number (Bujosa, Greciano, Martínez, Sánchez, & Soberats, 2021). Also, the compound [VII] b displays a nematic phase, that can be attributed to the chain length of an alkoxy terminal group that interferes with the order of liquid crystalline. The compounds give less ordered mesophases (nematic mesophase) when the ratio of terminal/lateral (t/l) interaction is high, while the compounds give high order mesophases (smectic phase) when this ratio is low (Karam & Ibraheem, 2018).

In addition, the compound [VII] d showed a nematic phase. This can be attributed to the presence of the hydroxyl group as a terminal group which could form inter molecular hydrogen bonding as shown in Figure (2), H-bonds are very important for the stabilization and tuning of functional liquid crystal properties. The results indicate that the phase transition temperatures are influenced by the site of hydrogen-bonding (Xu, Zhang, Wang, & Gu, 2009). The compounds [VII] a-d displays enantiotropic nematic phases. The nematic textures for compounds [VII] a, b as in Figures (1a) and (1b), respectively.

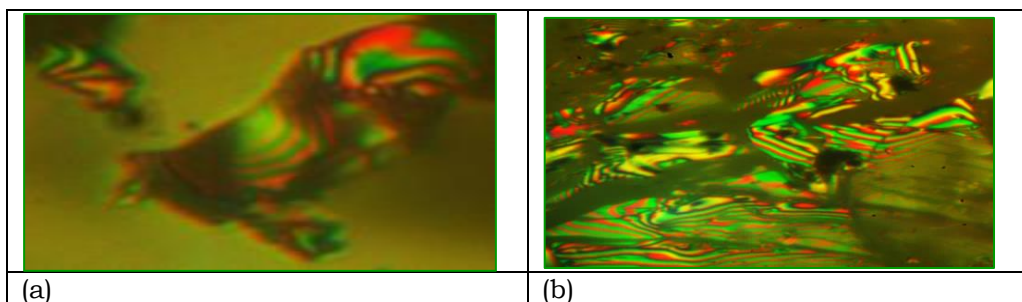


Figure 1. Cross - polarizing optical textures (a) nematic phase for compound [VII] a on heating at 320 ° C (b) nematic phase for compound [VII] b on heating at 230 ° C.

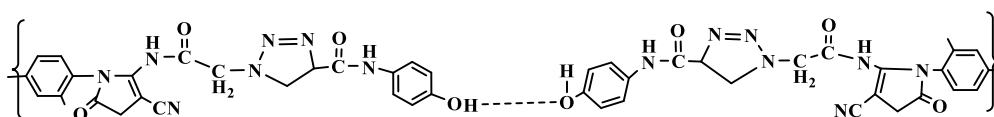


Figure 2. Hydrogen bonding formation of compound [VII] d

Conclusion

This work explains the synthesis and mesomorphic behaviour of new compounds comprising biphenyl central linkage and dihydro pyrrole and 1,2,3-triazole heterocyclic core. It was found that the central linkage, heterocyclic ring and the terminal substituted effect on mesophase type. These compounds gave liquid crystalline properties, with nematic mesophases and good thermal stability.

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