New Ether-Azo- Anil Derivatives(Preparation, Characterization , Crystal Behavior , Solubility in Solvents)

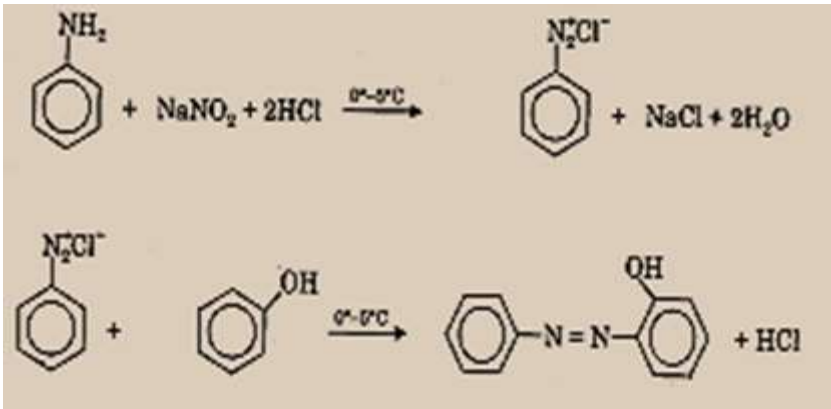
Athraa Sahib Alrammahi
M.Sc in Chemistry , Teacher in School , Alnajaf Government ., Iraq .

ABSTRACT
New chemical compounds were prepared in our work represented ether- azo- anil compounds from types of reactions such as azotation, coupling , substitution , condensation , imination reaction to formation new organic compounds which have several applications in many fields such as organic chemistry , liquid crystal fields , and other fields. All formatted new compounds investigated by many chemical and spectral methods (FT.IR , H.NMR , Mass) – spectrophotometric , studying liquid crystals behavior for our compounds , their solubility with many solvents , physical characterization .

Keywords: ethe , cry ,solub .

I.INTRODUCTION

The reaction of aromatic primary amines with hydrochloric acid in azotation reaction (diazonium salt) is named diazotization[1-3] . Diazonium salts are important synthetic intermediates which undergo coupling step to form azo dyes and aromatic azo dyes., aromatic azo compounds[4-8] tend to be brightly colored because of conjugated systems .



Picture .1 : Mechanism od Azotation step

Azo dyes are an important part from organic compounds, which include the coloring group (N=N-). The azo group is often bound to an aromatic system , and the dye can then be broken down to an aromatic amine, aromatic amine. Many types[9-16] of dyes can also be broken down to aromatic amines during storage due to light and high temperature. Some aromatic amines[17-23] have been judged to be carcinogenic.

The compounds of dyes are applied in dyeing textile fiber , cotton but also silk, wool, viscose and synthetic fiber . They are considered to be easy to use[24-33], very cheap and to provide clear, strong colors .

The mainly of dyes are soluble in water . Azo compounds may also be toxic to aquatic organisms and cause effects in the aquatic environment., azo dyes[34-41] are used in synthetic chemistry , as a starting material in several compounds and many fields[42-48] .

II.EXPERIMENTAL & MATERIALS

The formatted new compounds were investigated by : FT-IR spectra (FT-IR 8300 Shimadzu) in the range (400-4000) cm^{-1} as KBr discs ., ^{1}H.NMR– Spectra in DMSO–solvent., Differential , Polarized Optical Microscope (POM) . physical with chemical applications carried out for compounds.

EXPERIMENTAL PART:

STEP . 1 : Formation of Ether – Anil Compounds { 1 }:

Para -Alkoxy benzaldehyde derivative (0.1 mole) heated with (0.1mole) of para –methoxy aniline for (2 hrs) in ethanol and drops of glacial acetic acid according to literatures[20 ,24] ,to produce precipitation ,then filtered and dried, re crystallized to yield new compound { 1 }.

STEP .2 : Formation of Ether – Anil Compounds { 2 }:

Para -Alkoxy benzaldehyde derivative (0.1 mole) heated with (0.1mole) of para –amino aniline for (2 hrs) in ethanol and drops of glacial acetic acid according to literatures[20, 24] ,to produce precipitation ,then filtered and dried, re crystallized to yield new compound { 2 }.

STEP .3 : Formation of Ether – Anil Compounds { 3 }:

Compound { 2 } (0.1 mole) dissolved in (3 ml) of hydrochloric acid with sodium nitrite in ice temperature , then addition solution of coupling compound (meta- dinitrobenzene) according to literatures[20, 24] ,to produce precipitation ,then filtered and dried, re crystallized to yield new compound { 3 }.

STEP .4 : Formation of Ether – Anil Compounds { 4 }:

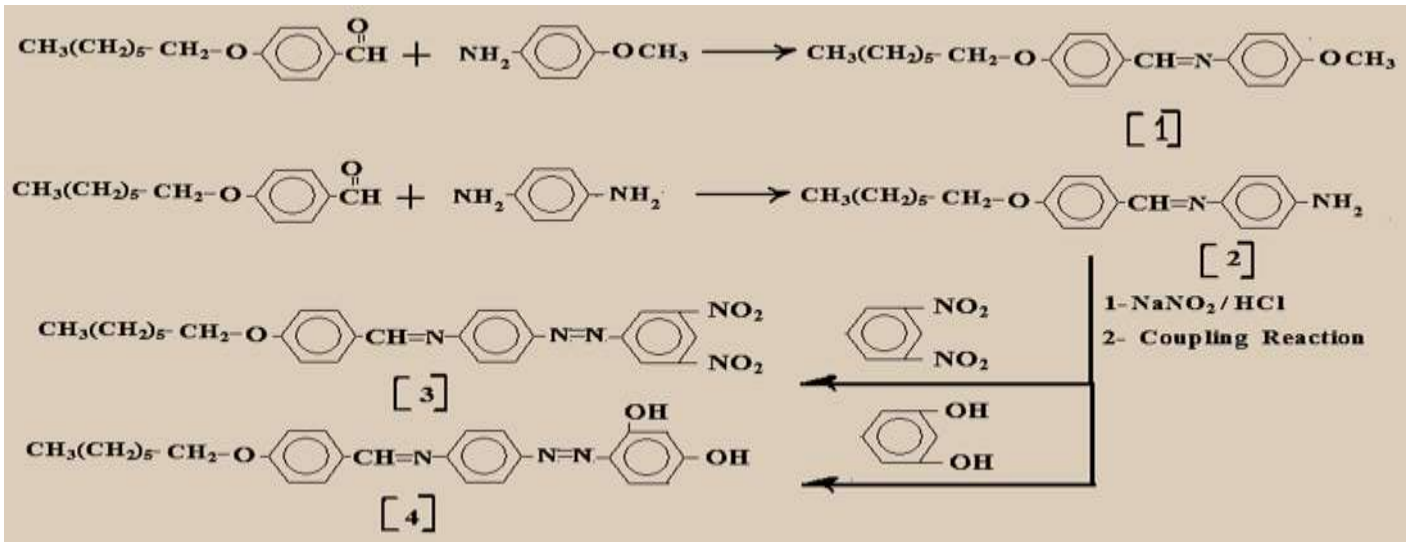
Compound { 2 } (0.1 mole) dissolved in (3 ml) of hydrochloric acid with sodium nitrite in ice temperature , then addition solution of coupling compound (resorcinol) according to literatures[210, 24] ,to produce precipitation ,then filtered and dried, re crystallized to yield new compound { 4 }.

STEP .5 : Formation of Ether – Anil Compounds { 5 }:

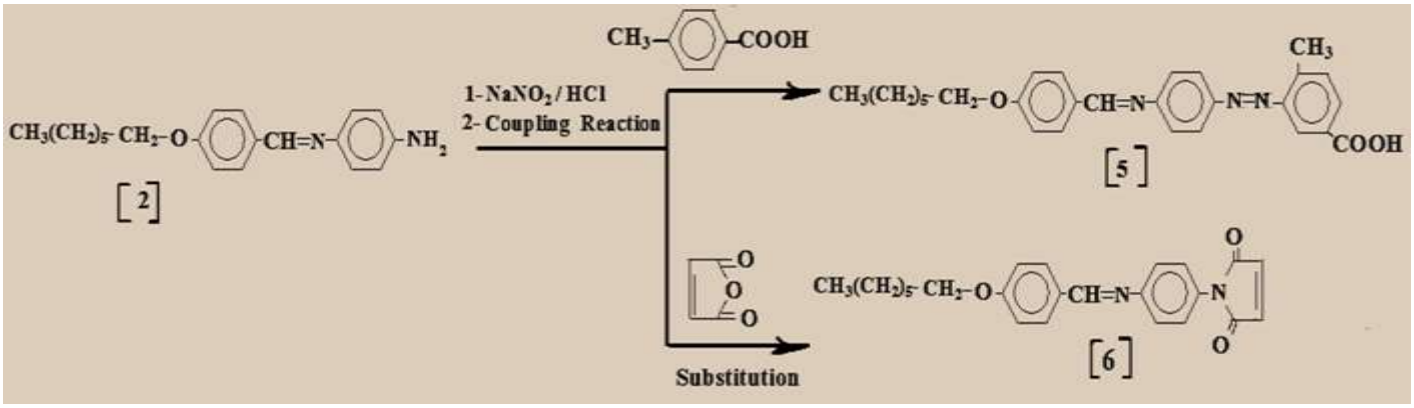
Compound { 2 } (0.1 mole) dissolved in (3 ml) of hydrochloric acid with sodium nitrite in ice temperature , then addition solution of coupling compound (para- toluic acid) according to literatures[20, 24] ,to produce precipitation ,then filtered and dried, re crystallized to yield new compound { 5 }.

STEP .6 : Formation of Ether – Anil Compounds { 6 }:

Compound { 2 } (0.1 mole) was refluxed with maleic anhydride according to literatures[20, 24] ,to produce precipitation ,then filtered and dried, re crystallized to yield new compound { 6 }.



Scheme. 1: Synthesis of Ether – Azo- Anil {1- 4 }



Scheme . 2: Formation of Ether – Azo- Anil {5 , 6 }

III.RESULTS AND DISCUSSION

Our work involved ,preparation of new six ether-Anil – Azo compounds (1-6) which were investigated by spectral methods like (FT.IR , H.NMR , Mass) spectra and physic - chemical studying and other applications (crystal ,POM).

Chemical and Spectral Investigation:

The FT.IR- Investigation : absorption bands appeared at (-C-O-C-) ether : 1236 ., (CH=N) Imine group : 1619 ., (CH)Aliph : 2978 in compound(1) , bands are appeared at (-C-O-C-) ether : 1243 ., (CH=N) Imine group : 1612 ., (CH)Aliph : 2966 ., (NH_2-) Amine : (3256 , 3247) in compounds (2) ,while other bands appeared at (-C-O-C-) ether : 1254 ., (CH=N) Imine group : 1626 ., (CH)Aliph : 2974 ., (-NO_2) : (1376 , 1514) ., (-N=N-)Azo : 1498 in compound (3) ., bands at (-C-O-C-) ether : 1262 ., (CH=N) Imine group : 1620 ., (CH)Aliph : 2975 ., (-OH) : 3411 ., (-N=N-)Azo : 1481 in compound (4) , bands at (-C-O-C-) ether : 1267 ., (CH=N) Imine group : 1622 ., (CH)Aliph : 2991 .,
(-COOH)Carbonyl of carboxyl group : (1718) ., (-N=N-)Azo : 1495 in compound (5) ., compound (6) : appeared bands at (-C-O-C-) ether : 1264 ., (CH=N) Imine group : 1620 ., (CH)Aliph : 2956 ., (-N=N-)Azo : 1482 ., (N-CO-) Carbonyl of amide : 1692 ., all bands abstracted in Table (1) .

Table (1): FT.IR- data (cm^{-1}) of Compounds (1- 6).

Comp	Other Groups
(1)	(-C-O-C-) ether : 1236 ., (CH=N) Imine group : 1619 ., (CH)Aliph : 2978 .
(2)	(-C-O-C-) ether : 1243 ., (CH=N) Imine group : 1612 ., (CH)Aliph : 2966 ., (NH_2-) Amine : (3256 , 3247) .
(3)	(-C-O-C-) ether : 1254 ., (CH=N) Imine group : 1626 ., (CH)Aliph : 2974 ., (-NO_2) : (1376 , 1514) ., (-N=N-)Azo : 1498 .
(4)	(-C-O-C-) ether : 1262 ., (CH=N) Imine group : 1620 ., (CH)Aliph : 2975 ., (-OH) : 3411 ., (-N=N-)Azo : 1481 .
(5)	(-C-O-C-) ether : 1267 ., (CH=N) Imine group : 1622 ., (CH)Aliph : 2991 ., (-COOH)Carbonyl of carboxyl group : (1718) ., (-N=N-)Azo : 1495 .
(6)	(-C-O-C-) ether : 1264 ., (CH=N) Imine group : 1620 ., (CH)Aliph : 2956 ., (-N=N-)Azo : 1482 ., (N-CO-) Carbonyl of amide : 1692

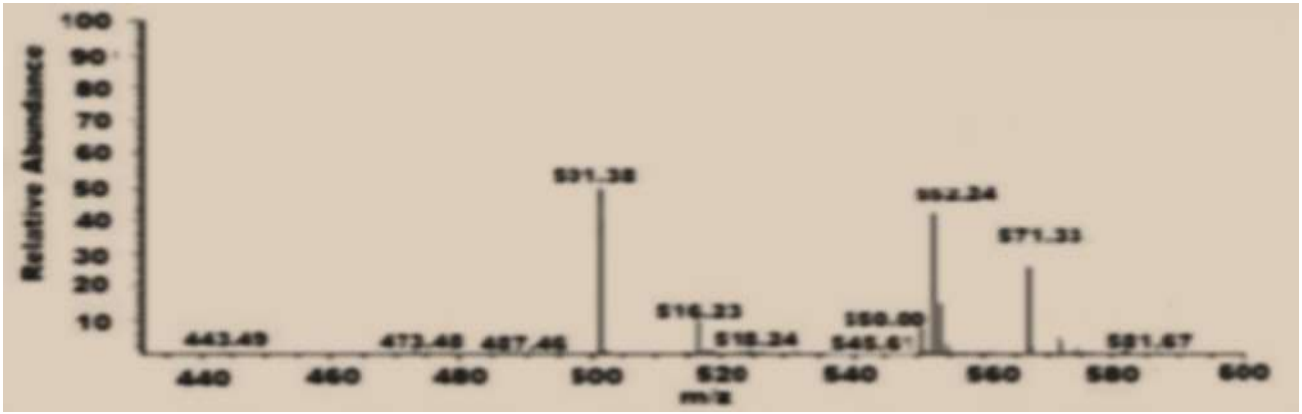
The ^{1}H.NMR- Investigation : showed peaks for ϐ DMSO-d6(solvent): 2.50 .,(CH=N) Proton of Imine: 8.12 .,Protons of Phenyl ring: (6.74-7.59) ., {CH_3-$(CH_2)_5$-CH_2-O -} : (0.61 , 0.67 , 0.69 , 0.70 , 0.72 , 0.73 ,0.94 , 0.65) in compound (1) .While compound (2) showed signals at .,(CH=N) Proton of Imine: 8.19 .,Protons of Phenyl ring: (6.79-7.43) ., {CH_3-$(CH_2)_5$-CH_2-O -} : (0.56 , 0.57 , 0.58 , 0.60 , 0.61 , 0.66 ,0.67) .,(NH_2): 5 ., compound(3) appeared peak at .,(CH=N) Proton of Imine: 8.21 .,Protons of Phenyl ring: (6.74-7.59) ., {CH_3-$(CH_2)_5$-CH_2-O -} : (0.61 , 0.67 , 0.69 , 0.70 , 0.72 , 0.73 ,0.94) ., while compound (4) showed signals at .,(CH=N) Proton of Imine: 8.31 .,Protons of Phenyl ring: (6.93-7.80) ., {CH_3-$(CH_2)_5$-CH_2-O -} : (0.65 , 0.66 , 0.70 , 0.74 , 0.79 , 0.84 ,0.87 , 0.88) .,(OH): 11.38 ., While compound (5) showed signals at .,(CH=N) Proton of

Imine: 8.24 .,Protons of Phenyl ring: (6.71-7.48) ., {CH_3-$(CH_2)_5$-CH_2-O -} : (0.51 , 0.59 , 0.67 , 0.68 , 0.73 , 0.78 ,0.82 , 0.85) **.,** (COOH)Proton of carboxyl : 12. 57 ., (CH_3) : 0.97 , compound (6) appeared signals at (CH=N) Proton of Imine: 8.37 .,Protons of Phenyl ring: (6.95-7.55) ., {CH_3-$(CH_2)_5$-CH_2-O -} : (0.75 , 0.76 , 0.79 , 0.80 , 0.83 , 0.86 ,0.89 , 0.97) ., (-CH_2=CH_2-) : (6.03 , 6.10) ., and other peaks in table (2) .

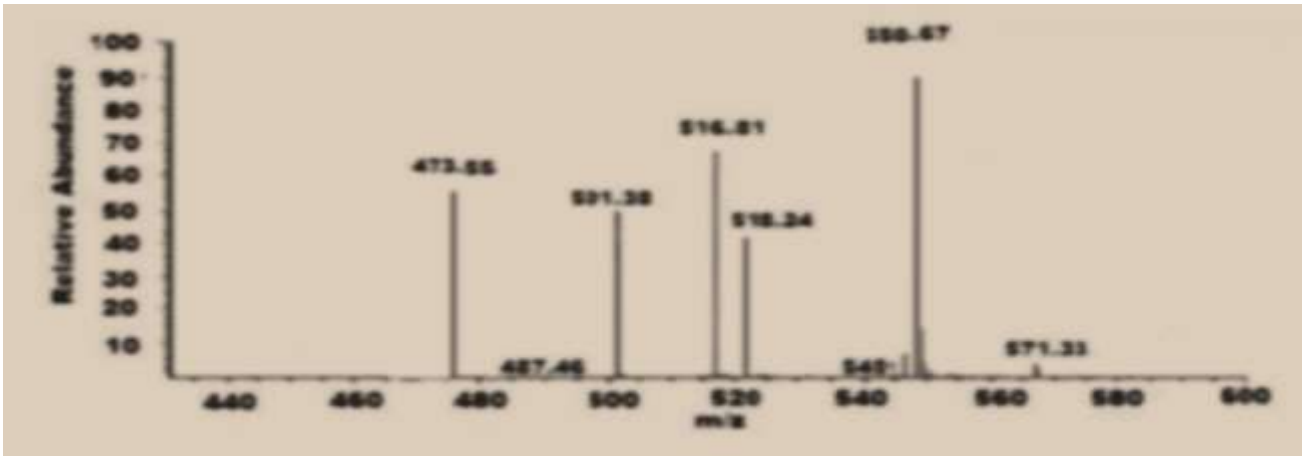
Table (2): H.NMR-data (ɓ - ppm) of Compounds (1- 6)

Comp	Other groups
(1)	DMSO-d6(solvent): 2.50 .,(CH=N) Proton of Imine: 8.12 .,Protons of Phenyl ring: (6.74-7.59) ., {CH_3-$(CH_2)_5$-CH_2-O -} : (0.61 , 0.67 , 0.69 , 0.70 , 0.72 , 0.73 ,0.94 , 0.65) .
(2)	DMSO-d6(solvent): 2.50 .,(CH=N) Proton of Imine: 8.19 .,Protons of Phenyl ring: (6.79-7.43) ., {CH_3-$(CH_2)_5$-CH_2-O -} : (0.56 , 0.57 , 0.58 , 0.60 , 0.61 , 0.66 ,0.67) .,(NH_2): 5.02 .
(3)	DMSO-d6(solvent): 2.50 .,(CH=N) Proton of Imine: 8.21 .,Protons of Phenyl ring: (6.74-7.59) ., {CH_3-$(CH_2)_5$-CH_2-O -} : (0.61 , 0.67 , 0.69 , 0.70 , 0.72 , 0.73 ,0.94) .
(4)	DMSO-d6(solvent): 2.50 .,(CH=N) Proton of Imine: 8.31 .,Protons of Phenyl ring: (6.93-7.80) ., {CH_3-$(CH_2)_5$-CH_2-O -} : (0.65 , 0.66 , 0.70 , 0.74 , 0.79 , 0.84 ,0.87 , 0.88) .,(OH): 11.38.
(5)	DMSO-d6(solvent): 2.50 .,(CH=N) Proton of Imine: 8.24 .,Protons of Phenyl ring: (6.71-7.48) ., {CH_3-$(CH_2)_5$-CH_2-O -} : (0.51 , 0.59 , 0.67 , 0.68 , 0.73 , 0.78 ,0.82 , 0.85) **.,** (COOH)Proton of carboxyl : 12. 57 ., (CH_3) : 0.97 .
(6)	DMSO-d6(solvent): 2.50 .,(CH=N) Proton of Imine: 8.37 .,Protons of Phenyl ring: (6.95-7.55) ., {CH_3-$(CH_2)_5$-CH_2-O -} : (0.75 , 0.76 , 0.79 , 0.80 , 0.83 , 0.86 ,0.89 , 0.97) ., (-CH_2=CH_2-) : (6.03 , 6.10) .

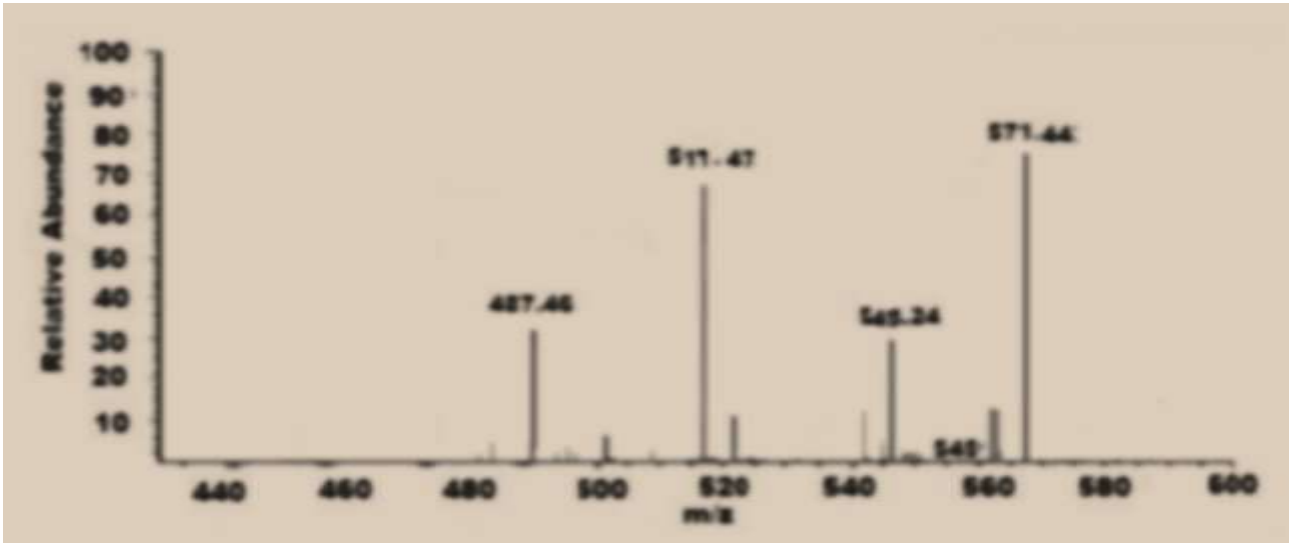
The Mass Investigation : Showed all fragments about parts of our formatted compounds in figures(1-3):



Fig(1): Mass Identification of Compound (3)



Fig(2): Mass Identification of Compound (4)



Fig(3): Mass Identification of Compound (5)

Optical Microscopic Study :

The results of optical microscopic study gave good result for behavior of crystal field for all compounds because of length and linearity[41, 44] of our compounds ., the behavior are shown in figures (4 - 9):

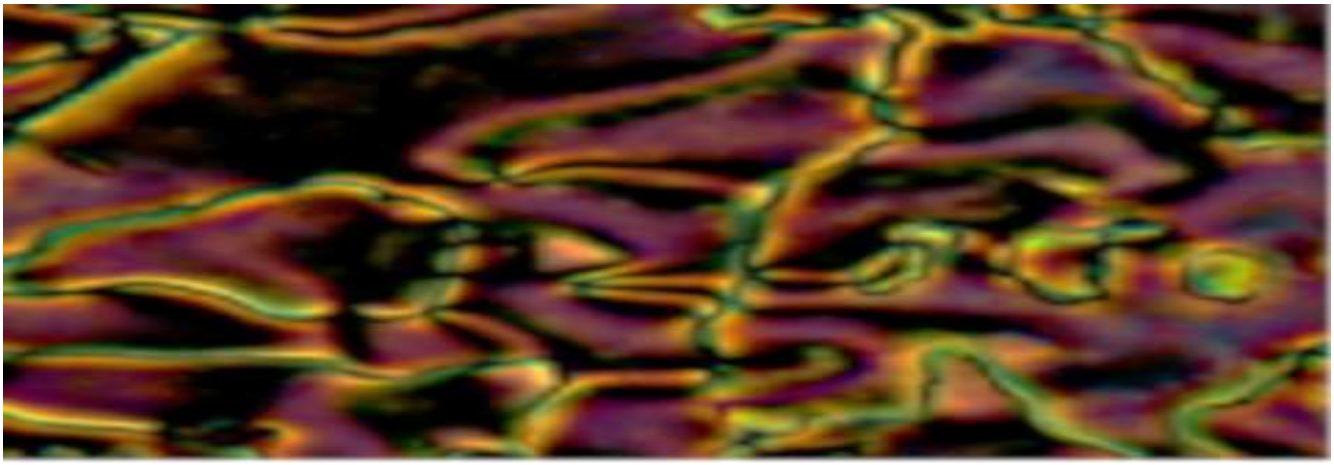


Fig. 4: Nematic Phase at 80 °C for Compound {1}

Fig. 5 : Nematic Phase at 98 °C for Compound {2}

Fig. 6 : Nematic Phase at 76 °C for Compound {3}

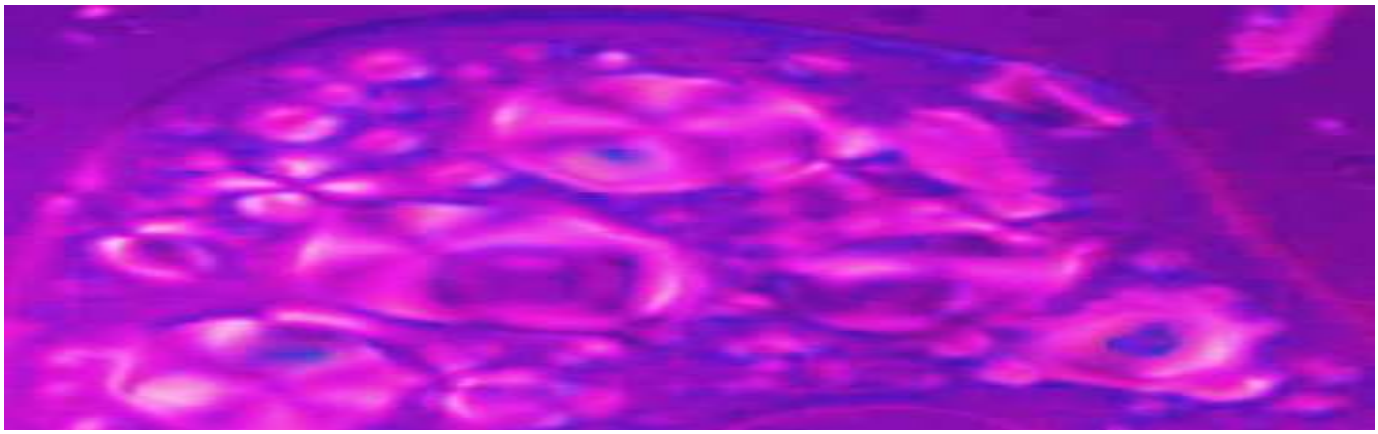


Fig. 7 : Nematic Phase at 120 °C for Compound{4}

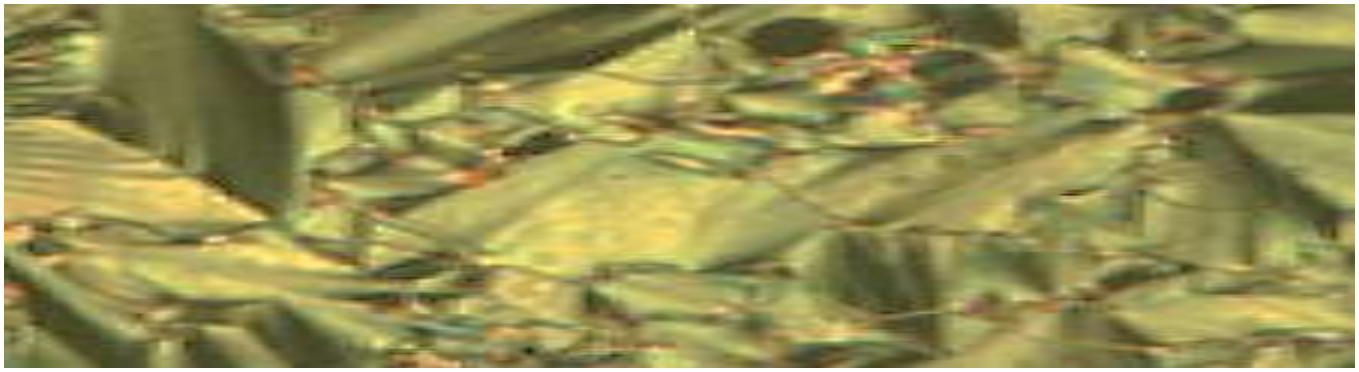


Fig. 8 :Nematic Phase at 92 °C for Compound{5}

Fig. 9 :Nematic Phase at 92 °C for Compound{6}

Solvation in Chemical Solvents :

Our formatted compounds were studied in different chemical solvents according to interaction of functional groups with solvents and solvation process , all results are abstracted in Table (3).

Table (3) : Solvation of compounds in organic Solvents.

Compounds	Solvents					
	CH_3OH	DMSO	DMF	$CHCl_3$	CCl_4	Dioxan
{ 1 }	+	+	+	-	-	-
{ 2 }	+	+	+	-	-	-
{ 3 }	+	+	+	-	-	-
{4 }	+	+	+	-	-	-
{5 }	+	+	+	-	-	-
{6 }	+	+	+	-	-	-

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