

Comparative Study of Microwave Assisted and Conventional Synthesis of Bisphenols and Diols Containing Azomethine Groups

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Abstract

In this investigation we were interested in carrying out a comparative study of microwave assisted and conventional synthesis of bisphenols and mesogenic azomethine diols containing azomethine groups. Modified domestic microwave with ethanol solvent as a reaction mediator and conventional heating (oil bath) were used in this study. The condensation reaction of primary amines with active carbonyls was depended in this study. It is well known that azomethine group (Ar-CH=N-Ar), which is a rigid structure, is frequently used as the mesogenic unit for liquid crystals. The structures of all synthesized compounds were confirmed by FT-IR, ¹H-NMR spectroscopy and elemental analysis (CHN). All the reactions under microwave irradiation were carried out in short time with high yield of products, whereas similar reactions under conventional heating (oil bath) at refluxed temperature gave fewer yields with comparatively longer time.

Introduction

Microwave irradiation is emerging as an unconventional energy source power enough to accomplish chemical transformations in minutes instead of hours or even days. The application of microwave irradiation in the

organic synthesis has been the point of considerable attention in recent years, (Loupy, 2002), (Kappe and Stadler, 2005). Microwave irradiation has been applied to carry out synthesis in different ways, open vessels and closed vessels with and without solvents by

using single or multi-mode irradiation, (Varma, 1999), (Lidstrom *et.al*, 2001). The main advantages of microwave assisted organic chemistry are shorter reaction times, higher yields and a reduction of side reactions compared with synthesis performed under conventional heating. This is due to the direct interaction between the electromagnetic wave and the dipole moments of the starting materials, (Loupy *et.al*, 2001).

Azomethine group (CH=N) containing compounds (Schiff bases) belong to a widely used as important intermediates for the production of special chemicals, e.g. pharmaceuticals or rubber additives and amino protective groups in organic synthesis, (Arulmurugan *et.al*, 2010), (Satyanarayana *et.al*, 2008), (Macho *et.al*, 2004). It is well known that bisphenols containing azomethine group (Ar-CH=N-Ar) which they are rigid structures, are frequently used as the mesogenic unit for liquid – crystalline epoxy thermo - sets (LCTs). (Jang *et.al*, 2002), Cozan *et.al*, 2005), (Zhang *et.al*, 2007), (Zongyong *et.al*, 2007), (Marin *et.al*, 2005), (Zhang *et.al*, 2006). A series of mesogenic azomethine diols and diesters were synthesized by condensation of bisphenols containing azomethine with chloralkanes and carboxylic acids, (Ahmed *et.al*, 2010), (Lee *et.al*, 2007), (Sie-Tiong *et.al*, 2010). There are also many reported data about thermotropic liquid crystalline poly

azomethines, such as poly(azomethine–ester)s, (Shukla and Akshit, 2003), poly (azo- methane – ether)s, (Palaninathan *et.al*, 2004), (Marin *et.al*, 2006), poly(azomethine–carbonate)s, (Sun S-J *et.al*, 1993), poly (azomethine–urethane)s Issam and Ismail (2006), poly (azomethine–acrylate)s, (Prescher *et.al*, 1995) and poly (azomethine–enaryloxynitriles), (Sang – Gon *et.al*, 1995).

Conventionally, azomethine group containing bisphenols were synthesized by the condensation of primary amines with active carbonyls, using an external heat source and therefore transferred by conductance. Herein, we wish to present a detailed research work on the comparative study of microwave assisted and conventional synthesis of bisphenols and mesogenic azomethine diols containing azomethine groups, using modified domestic microwave and ethanol solvent as a reaction mediator which absorbs microwave energy efficiently.

Experimental

Materials: Most of the chemicals used in this work were purchased from either Fluka AG and BDH Ltd with high degree of purity and they were used without purification. All the solvents were dried by the explained methods, Vogel *et al* (2010).

Apparatus: A domestic microwave oven (Shownig, Model: MW-GA 38GSC9), multi-mode microwave device with 2.45 GHz

frequency (38 L capacity) and rated microwave power 1000 W was purchased from the local market and modified to carry out the chemical reaction. The modification included, reflux condenser mounted through hole in the roof of the cavity, a remote IR sensor (Pyrometer) through a hole inside of the cavity which was inserted for temperature measurement and a magnetic stirrer which was fixed down the cavity of the oven.

^1H NMR spectra were recorded on a Bruker 300MHz instrument, using DMSO as a solvent and chemical shift was given relative to tetramethyl silane (TMS). Multiplicities of proton resonances are designated as singlet (s), doublet (d), triplet (t), quartet (q), multiplet (m) and broad (br). FT-IR spectra were recorded on ABB Spectrolap MB 3000 England, transform infrared (FT-IR) spectrometer. Vibration transition frequencies were reported in wave number between $500 - 4000 \text{ Cm}^{-1}$. The band intensity was classified as weak (w), medium (m), shoulder (sh), strong(s) and broad (br).

Synthesis of Bisphenols Containing Azomethine Groups:

Method A (conventional): In a 50 ml round bottomed flask, 4-Hydroxy benzaldehyde (1.0 g, 8.19 mmol) and 1,4-phenylenediamine (0.443 g, 4.097mmol) were mixed in ethanol (20 ml) in the presence of CaCl_2 (0.01 g) at reflux temperature in oil bath for six hours with continuous stirring, then cooled to room

temperature. The precipitate was filtered and washed with ethanol. The yellowish green powder was recrystallized from 95% ethanol.

Method B (microwave irradiation): The above procedure was carried out by using modified domestic microwave under reflux with 400w power for 8 minutes, then cooled to room temperature. The precipitate was filtered and washed with ethanol. The yellowish green powder was recrystallized from 95% ethanol. Similar synthetic procedures were applied to prepare other bisphenols containing azomethine groups. The structures and physical properties were illustrated in Table 1.

Synthesis of Mesogenic Diols Containing Azomethine Groups (1-9) a:

Method A (conventional): In a 100 ml round bottomed flask, 6-Chloro-1-hexanol (5.5 ml, 41.2 mmol) and sodium carbonate (4.24 g, 40 mmol) were mixed in 40 ml of dimethylformamide (DMF). Then, a solution of 19.25 mmol of bisphenol (1-9) in DMF (6 ml) was added. The mixture was refluxed with continuous stirring at 130°C for 12 hours. The reaction mixture was cooled and poured into cold distilled water (250 ml). The obtained precipitate was then filtered, washed four times with diethyl ether and dried in vacuum oven at 90°C . Recrystallization was carried out by DMF: butanol (1:1) to obtain colored crystals.

Method B (microwave irradiation): The above procedure was carried out by using modified

domestic microwave under reflux with 400W power for 20 minutes with contentiously stirring. The reaction mixture was cooled and poured into cold distilled water (250 ml). The obtained precipitate was then filtered, washed four times with diethyl ether and dried in vacuum oven at 90°C. Recrystallization was

carried out by DMF:butanol (1:1) to obtain colored crystals. Similar synthetic procedures were applied to synthesize other mesogenicdiols containing azomethine groups. The structures and physical properties were illustrated in Table 2.

Table (1) Structures and Properties of the Synthesized Bisphenols Containing Azomethine Groups(1-9).

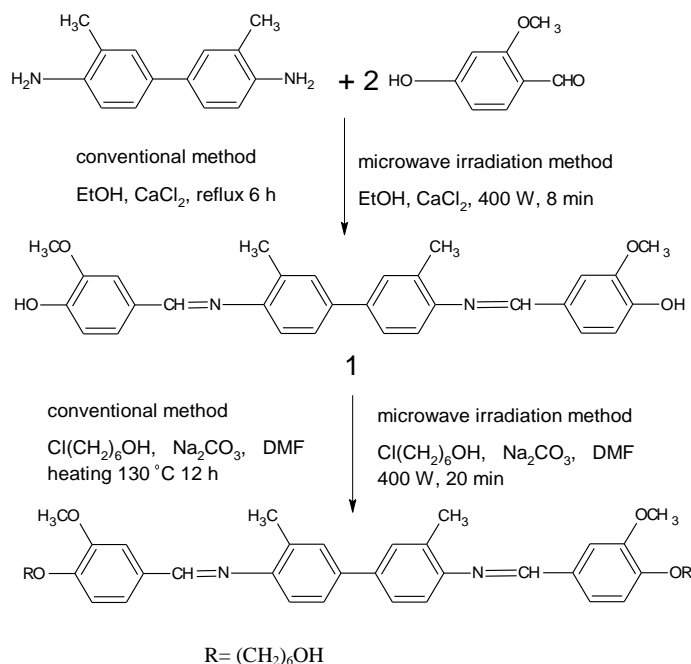
No.	Structure	Conventional*		Microwave**		mp(°C)	Color
		Time H	Yield %	Time min.	Yield %		
1		6	73	8	93	203	deep yellow
2		6	70	8	92	235	Light yellow
3		6	71	8	92	152	deep yellow
4		6	65	8	87	189	Yellow
5		6	62	8	89	252	Light yellow
6		6	59	8	88	243	Light yellow
7		6	70	8	92	285	Light yellow
8		6	66	8	90	299	Yellow
9		6	71	8	91	271	greenish yellow

* Reflux heating on oil bath, ** 400 W microwave power energy

Table (2) Structures and Properties of the Synthesized Mesogenic Diols Containing Azomethine Groups (1- 9) a, where R is (CH₂)₆OH.

No.	Structure	Conventional*		Microwave**		mp(°C)	Color
		Time H	Yield %	Time min.	Yield %		
1a		12	67	20	90	80	Yellow
2a		12	64	20	88	95	Light yellow
3a		12	63	20	87	152	Yellow
4a		12	60	20	87	77	Yellow
5a		12	59	20	89	100	Light yellow
6a		12	60	20	85	98	Light yellow
7a		12	62	20	90	127	Light yellow
8a		12	55	20	90	132	Yellow
9a		12	65	20	89	115	Yellow

* 130° C heating on oil bath, ** 400 W microwave power energy



Scheme 1

Results and discussion

Synthesis of Bisphenols Containing Azomethine Groups:

A series of bisphenol monomers containing azomethine groups were synthesized in this investigation by the condensation reaction of aromatic diamines with aromatic aldehydes in ethanol solvent and CaCl₂ as a catalyst. Conventional heating and microwave irradiation techniques were applied with some modifications for refluxing, Jignesh *et al* (2009). Scheme 1 summarized an example for this reaction. The comparative study in terms of yield and reaction time is shown in Table 1. All the reactions under microwave irradiation were carried out within 8 minutes at 400W microwave power energy and yields of products

ranged between 90-95%, whereas similar reactions under conventional heating (oil bath) at refluxed temperature gave less yields (didn't exceeded 80%) with comparatively longer reaction time (6 hours).

The structures of these compounds (1-9) were confirmed by FT-IR, ¹H-NMR spectroscopy and elemental analysis (CHN). Figures 1 and 2 show a typical examples of FT-IR and ¹H-NMR spectral, which corresponded to bisphenol monomer 1(N,N'- bis(4 - hydroxy - 3 - methoxy) benzylidene-o-tolidine), while FT-IR and ¹H-NMR spectral data for all compounds (1-9) were illustrated in Table 3.

FT-IR analysis of all compounds (1-9) showed a characteristic absorption band ranged of 1604 – 1623 cm⁻¹ indicating the formation of the

azomethine group (-CH=N-). The peak at 3270 – 3420 cm^{-1} for all compounds were assigned to phenolic hydroxyl group, while the aromatic rings skeletal vibrations and C=C bond stretching vibration appeared at 1580 – 1594, 1510 – 1520 and 1449 – 1460 cm^{-1} . Other functional groups (CH_3 and OCH_3) were also detected well.

$^1\text{H-NMR}$ spectral data for all compounds illustrated the presence of the azomethine group (-CH=N-) in the range of δ at (8.33 – 8.43) ppm. The multiple peaks in the range at δ (6.8 – 7.79) ppm and a single broad peak at δ (9.73 – 10.01) were observed for all compounds due to the aromatic and phenolic protons respectively.

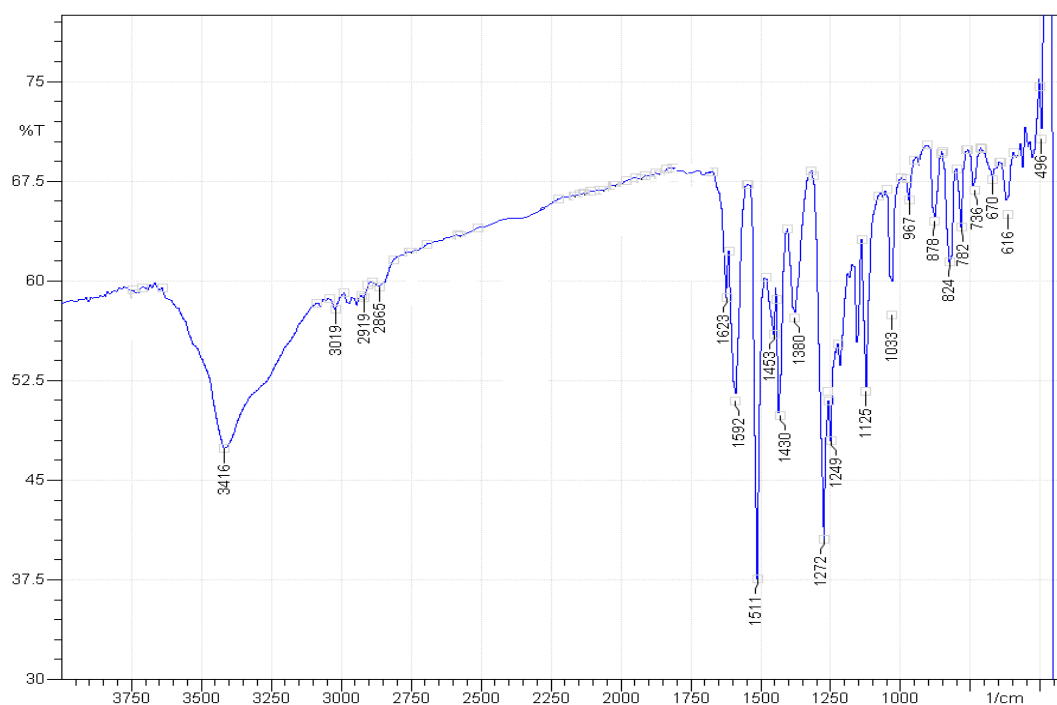


Figure (1) FT-IR Spectrum of Bisphenol Monomer 1, (N, N'- bis (4-hydroxy-3-methoxy)-benzylidene-O-tolidine).

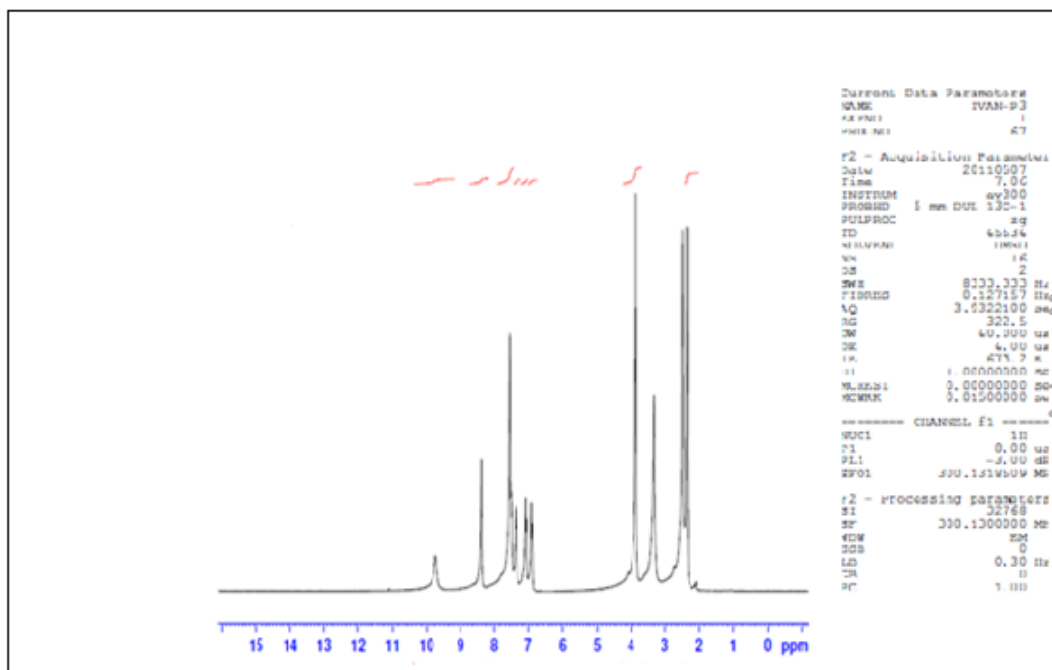


Figure (2) $^1\text{H-NMR}$ Spectrum of Bisphenol Monomer 1, (N, N'- bis (4-hydroxy-3-methoxy)benzylidene-O-tolidine).

Mesogenicdiols Containing Azomethine Groups:

Synthesis of mesogenic azomethane diols containing end terminal hexanol as promising thermotropic liquid crystal monomers was performed by the substitution reaction between bisphenols (1-9) and 6-chloro-1-hexanol. The reaction was carried out in DMF polar solvent by using conventional heating method (oil bath) Issam *et al* (2010), and unconventional heating method (microwave irradiation, 400W). Scheme 1 summarized an example for this reaction. As illustrated in Table 2 in terms of yield and reaction time, all the reactions under

microwave irradiation were carried out within 20 minutes at 400W microwave power energy and yields of products ranged 83 – 90 %, whereas similar reactions under conventional heating (130° C heating on oil bath) gave less yields (didn't exceed 68%) with a comparatively longer reaction time (12 hours).

The structures of these diols (1-9)a were confirmed by FT-IR, $^1\text{H-NMR}$ spectroscopy and elemental analysis (CHN). A good agreement was found between the calculated and experimental values obtained from elemental analysis of all diols (1-9) a.

Table (3) FT-IR, ¹H-NMR and UV-VIS Spectral Data of the Synthesized Bisphenols Containing Azomethine Groups (1-9).

No.	FT-IR Spectral Data (cm ⁻¹)	¹ H-NMR Spectral Data c (δ ppm)	UV-VIS Data λ _{max} (nm)
1	3416 (s, OH), 3016 (w, Ar. C-H), 2919, 2865 (w, CH ₃), 1623(sh, C=N), [1592 (s), 1511 (s), 1453 (m), Ar.C=C], 1380(m,C-N), [878(w), 824(m),736(m) Ar. Sub.]	2.37(s, 6H, CH ₃),4.09 (s, 6H, OCH ₃) 6.89(d, 2H), 7.08(d, 2H), 7.36(d, 2H) ,7.79(s,2H), 8.39(s, 2H, H-C= N), 9.73(s, 2H, OH)	350 , 287 , 231
2	3335 (s, OH), 3069 (w, Ar. C-H), 2957, (w,CH ₃), 1623(sh, C=N), [1584 (s), 1514 (s), 1450(m),Ar.C=C],1387(m,C-N) , [870(w), 820(m),747(m),635(m) Ar. Sub.]	4.11 (s, 6H, OCH ₃) 6.70 (d, 4H), 6.85(2H,d), 7.20(d, 4H), 7.35(d, 2H) , 7.84(s,2H), 8.42(s, 2H, H-C= N), 9.80(s, 2H, OH)	359 , 289 , 232
3	3331 (s, OH), 3077 (w, Ar. C-H), 2969, (w, CH ₃), 1617(sh, C=N), [1591 (s), 1514 (s), 1450(m), Ar.C=C], 1383(m, C-N), [863(m), 751(m), 630(m) Ar. Sub.]	4.07 (s, 6H, OCH ₃) 6.85 (d, 2H), 7.10(4H,d), 7.34(d, 2H), 7.77(s, 2H) , 8.40(s, 2H, H-C= N), 9.76(s, 2H, OH)	350 , 286 , 236
4	3381 (s, OH), 3042(w, Ar. C-H), 2957, 2857 (w, CH ₃), 1626 (s, C=N), [1580 (s), 1484 (s), 1453(m), Ar.C=C], 1379 (m,C-N),[809(m), 685(s) Ar. Sub.]	2.40(s, 6H, CH ₃), 6.69(d, 2H), 6.80(d, 2H), 7.01(d, 2H), 7.11(d, 2H), 7.22(d, 2H), 7.37(d, 2H)7.80(s,2H), 8.34(s, 2H, H-C= N), 9.90(s, 2H, OH)	350 , 277
5	3416 (s, OH), 3052(w, Ar. C-H), 1626(s, C=N), [1595 (s), 1500 (s), 1457(m), Ar.C=C], 1376(m, C-N), [855(m), 825(m), 680(s) Ar. Sub.]	6.66(d, 2H), 6.82(d, 2H), 7.01(d, 4H), 7.33(d, 4H), 7.42(d, 2H), 7.81(s,2H), 8.39(s, 2H, H- C= N), 9.92(s, 2H, OH)	350 , 279
6	3412 (s, OH), 3046(w, Ar. C-H), 1619(s, C=N), [1592(s), 1503 (w), 1453(s), Ar.C=C], 1387(m, C-N), [855(s), 758(m), 681(s) Ar.Sub.]	6.84(d, 2H), 7.15(d, 4H), 7.44(d, 2H), 7.80(s,2H), 8.40(s, 2H, H-C= N), 9.82(s, 2H, OH)	350 , 273
7	3439 (s, OH), 3069(w, Ar. C-H),2953 ,2880 (w,CH ₃), 1605 (sh,C=N), [1591(s),1514 (w), 1483 (s), Ar.C=C] , 1375 (m,C-N),820,770[Ar. Sub.]	2.39(s, 6H, CH ₃), , 6.66(d, 2H), 6.82(d, 2H), 7.07(d, 2H), 7.13(d, 2H), 7.20(d, 2H), 7.27(d, 2H)7.81(s,2H), , 8.37(s, 2H, H-C= N), 9.58(s, 2H, OH)	350 , 292
8	3420 (s, OH), 3076(w, Ar. C-H), 1608(sh, C=N), [1588(s), 1511 (m), 1455 (s), Ar.C=C], 1372(m, C-N), 828, (Ar. Sub.)	6.75 (d, 4H), 6.84(2H,d), 7.09 (d, 4H), 7.33(d, 2H) , 7.80(s,2H), 8.39(s, 2H, H-C= N), 9.65(s, 2H, OH)	350 , 291
9	3277 (s, OH), 3076(w, Ar. C-H), 1610(sh,C=N), [1599(s), 1518 (m), 1455 (s), Ar.C=C] , 1391(m,C-N),839, (Ar. Sub.)	6.81 (d, 2H), 7.09(4H,d), 7.33(d, 2H), 7.79(s, 2H) , 8.44(s, 2H, H-C= N), 9.57(s, 2H, OH)	350 , 290 , 225

FT-IR : s (strong), w (weak), m (medium), sh (shoulder) ,¹H-NMR : s (singlet) ,d (doublet)

Figures 3 and 4, show typical examples of FT-IR and $^1\text{H-NMR}$ spectral, which corresponded to mesogenic azomethine diol 1a (N, N'-bis (4-hydroxy octoxy - 3-methoxy) benzylidene-O-tolidine). FT-IR and $^1\text{H-NMR}$ spectral data for all compounds (1-9)a were illustrated in Table 4. FT-IR analysis of all diols (1-9) a showed strong absorption bands ranged 2870 – 2850 and 1450 – 1460 cm^{-1} related to stretching vibration and bending deformation of methylene (CH_2) groups which indicate the presence of aliphatic C-H in alkyl chain. The absorption band merged at 1608 – 1625 cm^{-1} indicating the presence of the azomethine group (CH=N). The peak at 3370 – 3420 cm^{-1} for all

compounds assigned to aliphatic hydroxyl group, while the aromatic rings skeletal vibrations and C=C bond stretching vibration appeared at, 1580 – 1590, 1570 – 1540 and 1520 – 1510 cm^{-1} . Other functional groups (CH_3 , OCH_3) were also detected well. $^1\text{H-NMR}$ spectral data for all diols (1-9)a indicated that, the chemical shift at $\delta = 1.1 - 2.0$ ppm assigned to methylene protons of hexyl chain. Other characteristic of bands related to (CH=N) and the protons in the aromatic rings were appeared as singlet at (8.33-8.43) and a multiplet at (6.8 – 7.79) ppm, respectively. Peaks at (3.57 – 3.60) ppm and (4.05 – 4.1) ppm are related to (OH-CH_2) and (Ph-O-CH_2) respectively.

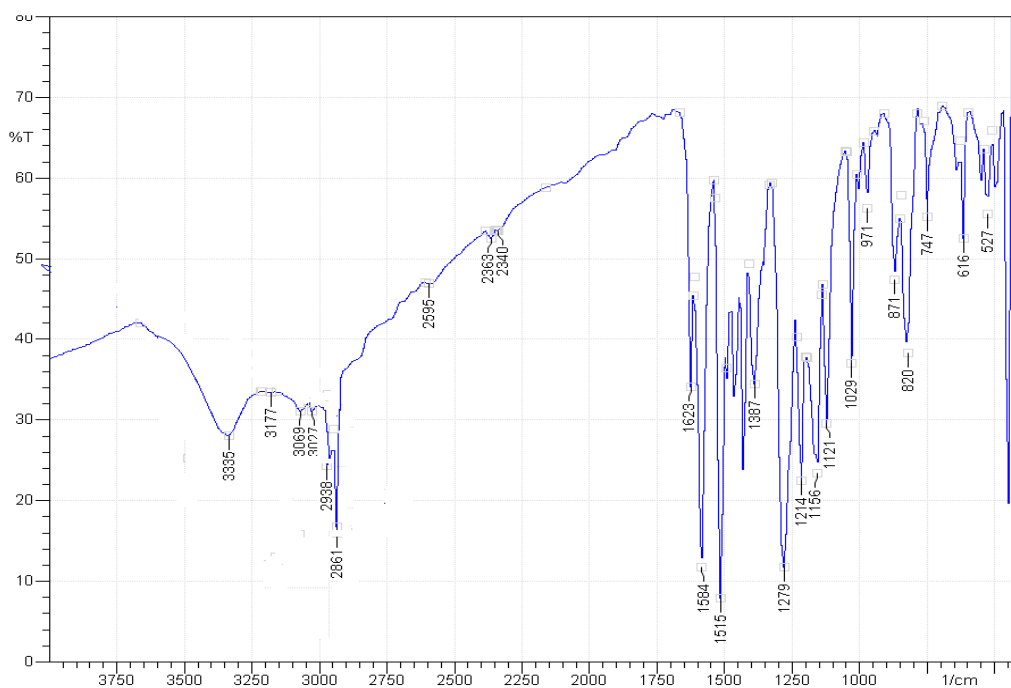


Figure (3) FT-IR Spectrum of Mesogenic Azomethinediol 1a (N, N'-bis (hydroxy- octoxy-3-methoxy) benzylidene-O-tolidine).

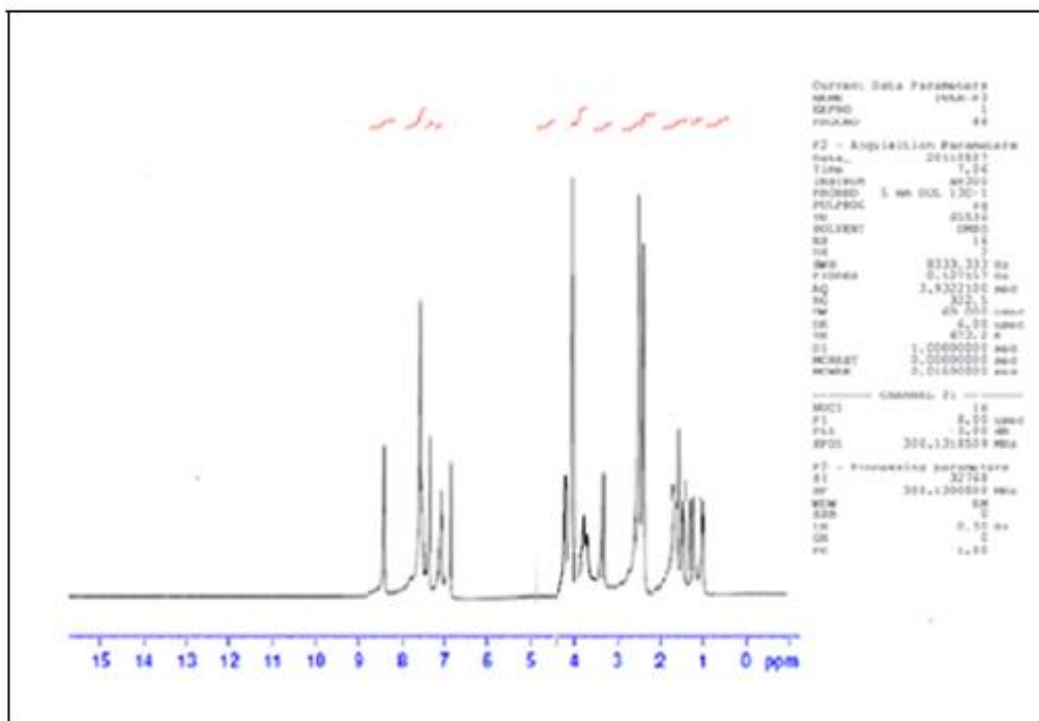


Figure (4) ^1H NMR Spectrum of Mesogenic Azomethine diol 1a, (N, N'-bis (4-hydroxy octoxy-3-methoxy) benzylidene-O-tolidine).

The synthesis under microwave irradiation for either bisphenols (1-9) or diols (1-9)a was accelerated because of the directly interaction of microwave irradiation with the dipole moments of the starting materials, demonstrating that the effect of microwave irradiation was not purely thermal. Otherwise, microwave irradiation produces efficient internal heat transfer (in situ heating), resulting in even heating throughout the sample as compared with the wall heat transfer that occurs when a water/oil bath applied as energy source. This is consistent with the reaction mechanism, which involves a polar transition state Varma

(1999). In early application of microwave technique in chemical reactions, open vessels (Erlenmeyer flasks) with solvents or in a dry condition (solvent -free) in domestic MW-oven have been used successfully Lidstrom *et al* (2001). However non uniform heating and difficulties with mixing in a dry condition were observed and organic solvents with opened vessels can lead to violent explosions. So in this work, we depended on a reflux system (the water condenser is outside the MW) in modified domestic microwave to prevent any vapor in the cavity of oven and to have uniform reactions.

Table (4) FT-IR and ¹H-NMR Spectral Data of the Synthesized Mesogenic Diols Containing Azomethine Groups(1- 9) a.

No.	FT-IR Spectral Data(cm ⁻¹)	¹ H-NMR Spectral Data (δ ppm)	λ _{max} (nm)
1a	3335 (s, OH), 3069 (w, Ar. C-H), 2938, 2861 (m, CH ₂ & CH ₃), 1623(sh, C=N), [1584(s), 1511 (s), 1450 (m), Ar.C=C], 1387(m, C-N), 1279(s, C-O-C), [871(w), 820(m), 7747(m) Ar. Sub.]	1.0 – 1.72 (m,16H,8 methylene), 2.39(s, 6H, CH ₃),3.57(d,2H,HO-CH ₂),3.90 (t ,4H, Ph -O-CH ₂ -),4.10 (s, 6H, OCH ₃), 4.2 (S,2H, OH), 6.84 (d, 2H), 7.05(d, 2H), 7.40(d, 2H) , 7.80(s,2H), 8.36(s, 2H, H-C= N)	350 , 286, 231
2a	3305 (s, OH), 3049 (w, Ar. C-H), 2969,2870 (m, CH ₂ & CH ₃), 1620(sh, C=N), [1588 (s), 1520 (s), 1460(m),Ar.C=C],1380(m,C-N) , 1256(s, C-O-C), [866(w), 822(m), 741(m), 635(m) Ar. Sub.]	1.09 – 1.74 (m,16H,8 methylene), 2.38(s, 6H, CH ₃),3.55(d,2H,HO-CH ₂),3.89 (t , 4H, Ph -O-CH ₂ -),4.10 (s, 6H, OCH ₃) 6.70 (d, 4H), 6.85(2H,d), 7.22(d, 4H), 7.35(d, 2H) , 7.84(s,2H), 8.42(s, 2H, H-C= N)	359 , 287 , 233
3a	3350 (s, OH), 3067 (w, Ar. C-H), 2969, 2855 (m, CH ₂ &CH ₃), 1617(sh, C=N), [1591 (s), 1520 (s), 1454(m), Ar.C=C], 1377(m, C-N), 1250(s, C-O-C) ,[860(m), 752(m), 636(m) Ar. Sub.]	1.10 – 1.80 (m,16H,8 methylene), 2.37(s, 6H, CH ₃),3.60(d,2H,HO-CH ₂),3.90 (t , 4H, Ph -O-CH ₂ -),4.09(s, 6H, OCH ₃) 6.83 (d, 2H), 7.07(4H,d), 7.30(d, 2H), 7.75(s, 2H) , 8.42(s, 2H, H-C= N)	350 , 286 , 235
4a	3400 (s, OH), 3055(w, Ar. C-H), 2960, 2866 (m, CH ₂ & CH ₃), 1624 (s, C=N), [1584 (s), 1480 (s), 1449(m), Ar.C=C], 1381 (m, C-N), 1248(s, C-O-C), [815(m), 681(s) Ar. Sub.]	1.20 – 1.83 (m,16H,8 methylene), 2.40(s, 6H, CH ₃),3.61(d,2H,HO-CH ₂),3.92(t , 4H, Ph -O-CH ₂ -), , 6.66(d, 2H), 6.80(d, 2H), 7.04(d, 2H), 7.15(d, 2H), 7.20(d, 2H), 7.40(d, 2H)7.81(s,2H), 8.39(s, 2H, H-C= N),	350 , 279
5a	3398 (s, OH), 3060(w, Ar. C-H), 2910, 2873(m, CH ₂), 1625(s, C=N), [1590 (s), 1510 (s), 1447(m), Ar.C=C], 1379(m, C-N), 1252 (s, C-O-C), [855(m), 825(m), 680(s) Ar. Sub.]	1.08 – 1.85 (m,16H,8 methylene), 3.66(d,2H,HO-CH ₂),3.94(t , 4H, Ph -O-CH ₂ -), 6.70(d, 2H), 6.83(d, 2H), 7.08(d, 4H), 7.29(d, 4H), 7.40(d, 2H), 7.79(s,2H), 8.42(s, 2H, H-C= N)	350 , 280
6a	3401(s, OH), 3055(w, Ar. C-H), 2905, 2870(m, CH ₂), 1619(s, C=N), [1592(s), 1513 (w), 1458(s), Ar.C=C], 1381(m, C-N), 1250(s, C-O-C), [860(s), 751(m), 673(s) Ar.Sub.]	1.11 – 1.82 (m,16H,8 methylene), 3.60(d,2H,HO-CH ₂),3.91(t , 4H, Ph -O-CH ₂ -), 6.73(d, 2H), 7.18(d, 4H), 7.25(d, 2H), 7.78(s,2H), 8.41(s, 2H, H-C= N)	350 , 273
7a	3419 (s, OH), 3065(w, Ar. C-H),2959 ,2882(m, CH ₂ &CH ₃), 1608(sh,C=N), [1590(s),1523 (w), 1477 (s), Ar.C=C], 1379 (m,C-N), 1254(s, C-O-C), 823,772[Ar. Sub.]	1.14 – 1.85 (m,16H,8 methylene), 2.38(s, 6H, CH ₃),)3.64 (d,2H,HO-CH ₂),3.97(t , 4H, Ph -O-CH ₂ -), , 6.68(d, 2H), 6.88(d, 2H), 7.09(d, 2H), 7.15(d, 2H), 7.20(d, 2H), 7.29(d, 2H)7.82(s,2H), , 8.39(s, 2H, H-C= N)	350 , 292
8a	3405 (s, OH), 3060(w, Ar. C-H),2914, 2850(m, CH ₂), 1613(sh, C=N), [1584(s), 1519 (m), 1457 (s), Ar.C=C], 1378(m, C-N), 1260(s, C-O-C), 830, (Ar. Sub.)	1.14 – 1.85 (m,16H,8 methylene),)3.64 (d,2H,HO-CH ₂),3.97(t , 4H, Ph -O-CH ₂ -), 6.74(d, 4H), 6.79(2H,d), 7.08 (d, 4H), 7.32 (d, 2H) , 7.84(s,2H), 8.40(s, 2H, H-C= N)	350 , 291
9a	3280 (s, OH), 3056(w, Ar. C-H), 2890 (m, CH ₂), 1612(sh, C=N), [1594(s), 1530 (m), 1459 (s), Ar.C=C], 1388(m, C-N), 1253(s, C-O-C), 840, (Ar. Sub.)	1.04 – 1.80 (m,16H,8 methylene),)3.64 (d,2H,HO-CH ₂),3.97(t , 4H, Ph -O-CH ₂ -), 6.84 (d, 2H), 6.98(4H,d), 7.21(d, 2H), 7.76(s, 2H) , 8.40(s, 2H, H-C= N)	350 , 292 , 229

FT-IR: s (strong), w (weak), m (medium), sh (shoulder) ,¹H-NMR : s (singlet) ,d (doublet)

Conclusion

This comparative study showed that, the microwave technique is an excellent method which can be used to prepare bisphenols and diols. The refluxing condenser system which was introduced in our modified domestic microwave gave a good safety method. In addition to, the method is characterized by the rationalization of energy and time. It also gave a high yield of products.

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دراسة مقارنة بين المايكرووف المساعد وطريقة التسخين الاعتيادي في تخليق البسفينولات والدايولات الحاويه على مجاميع الازوميثان

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الخلاصة

تضمن هذا البحث دراسة مقارنة تخليق مركبات البسفينول والدايول الحاويه على مجاميع الازوميثان الميزوجينيه باستخدام الاشعه المايكرويه والتسخين الاعتيادي المؤلف . استخدم جهاز المايكرووف المحور في مختبرنا كمصدر للاشعه المايكرويه مع مذيب الايثانول كوسط للتفاعل وطريقة التسخين الاعتيادي (حمام زيتي) . تم اعتماد التفاعل التكاتفي بين الامين الاولي والكاربونيل الفعال في تحضير مركبات البسفينول والتفاعل التعويضي بين البسفينول وهاليد الالكيل في تحضير الدايلول الحاويه على مجاميع الازوميثان الميزوجينيه . هذه المركبات الميزوجينيه تتصف بتركيبها القوي واستخداماتها في تحضير البلورات السائله . تم اثبات المركبات المحضره بواسطة تقنيات الاشعه تحت الحمراء و الرنين النووي المغناطيسي - البروتون وتحليل العناصر كاربون - هايدروجين - نايتروجين . ان جميع التفاعلات تحت المايكرووف اعطت ناتج عالي وأنجزت بوقت قصير اما التفاعلات بالتسخين الاعتيادي فكان الناتج اقل والوقت طويل .