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Mesomorphic properties, microwave-assisted synthesis, and antimicrobial evaluation of novel Schiff base functionalized resorcin[4]arene derivatives

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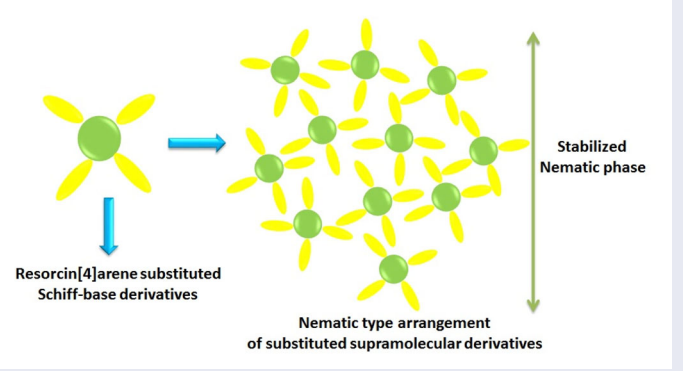
ABSTRACT

Schiff bases of Resorcin[4]arenes were synthesized under microwave irradiation in high yield and characterized through various spectroscopic techniques. Mesomorphic behavior of synthesized materials **(8a-f)** were studied by polarizing optical microscope, differential scanning calorimetry (DSC), and thermal gravimetric analysis. Most of compounds displayed isotropic phase and nematic mesophase in enantiotropically manner. Electronic properties of the highest occupied molecular orbital (HOMO) and the lowest unoccupied molecular orbital (LUMO) of compound **(8a)** was simulated for the first time. Among them, most of derivatives exhibited outstanding antibacterial activity. Some molecules also displayed excellent antifungal potency.



KEYWORDS

Resorcin[4]arene; microwave irradiation; resorcin[4]arene; Schiff bases; antimicrobial; liquid crystals

Introduction

In supramolecular chemistry, studies of macrocycles like calix[4]arenes [1–3] is much encouraging and growing branch. Resorcin[4]arene is one type of calix[4]arene and its made from resorcinol units. Resorcin[4]arene is a flexible subclass, having eight hydroxyl groups at their upper rim, which is the best active site for substitution to

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increasing their applications. They are broadly described as a form of their oxygen bridged derivatives, the cavitands (it is a container shaped molecule), and the different type of supramolecular assemblies are widely found in literature [4–9].

Resorcin[4]arene and their derivatives were synthesized by different synthetic protocols utilizing different Lewis acid catalysts like concentrated HCl [10], trifluoroacetic acid [11], TSA (Tungsten sulfuric acid) [12], MSA(molybdate sulfuric acid) [13], iodine [14], and so on. The resorcinol react with various aldehyde gives all cis cone stereoisomer [15,16], that reported by Niedel and Vogel and their boat, saddle, and chair stereoisomer was discovered by Hogberg [17]. In resorcin[4]arene structure, different aldehyde groups attached in feet to give the ordering of stereoisomers of resorcin[4]arene [18].

Resorcin[4]arene are popular cyclic tetrameric host compounds for ions, sugars, and organic molecules thus their applicability is very wide [19]. They are also used as a starting material for different cavitands [20–24]. Resorcin[4]arenes have a broad range of applications such as a macrocyclic receptor [25], dendrimers in biological systems [26], nanoparticles and nano-capsules [27], supramolecular tectonics [28], optical chemosensors [29], host-guest complexes [30], components in liquid crystals [31], photoresists [32], selective membranes [33], HPLC stationary phases [34], surface reforming agents [25], ion channel mimics [35], and metal ion extraction agents [36] and also in host-guest chemistry with Nonlinear optical (NLO) property of material chemistry also taken consideration [37–42].

Nowadays, liquid crystalline (LC) a state investigation has become a multidisciplinary research area, due to its exceptional property to flow like a liquid and optical properties as crystals are beneficial to various industrial applications [43–48]. Study on LC state is measured with a view to understanding the effect of molecular structure on LC properties [49–51] as a significance of molecular stiffness and flexibility [52–55]. Liquid crystals can be classified into lyotropic, thermotropic, and metallotropic phases. Lyotropic and thermotropic liquid crystal state mostly seen in organic compounds, however, few minerals are also known [56]. Liquid crystals in the nematic group are most extensively used in the manufacture of liquid crystal displays (LCD) primarily due to their exceptional physical properties as well as a wide temperature range.

Calix[4]arene rigid core as liquid crystalline materials was reported and utilized first time in 1990 [57]. Such type of materials, which are an important part of macrocycles based liquid crystal materials were examined by some scientist [58–62]. Furthermore, the calix[4]arene with substitution of long aliphatic side chains showed excellent liquid crystalline behavior [63,64]. Yonetake and coworkers have prepared two liquid crystal based on *tert*-butyl calix[8]arene to show smectic and nematic mesophases [65]. Yang and associates displayed mesomorphic property with the presence of columnar type molecular arrangement of calixarene with cholesterol derivatives [30]. Furthermore, Yang et al. have investigated the liquid crystalline behavior of calix[4]arene-linked triphenylene derivatives [66–69]. In addition, his group has reported the mesomorphic properties of gallic-calix[4]arene derivatives with stable cone conformation [70]. Menon and coworkers have disclosed a review on lower rim substituted calixarenes and their various applications [71]. In addition, her group has synthesized Schiff base and azoester derivatives of calix[4]arene and studied their liquid crystalline and dielectric

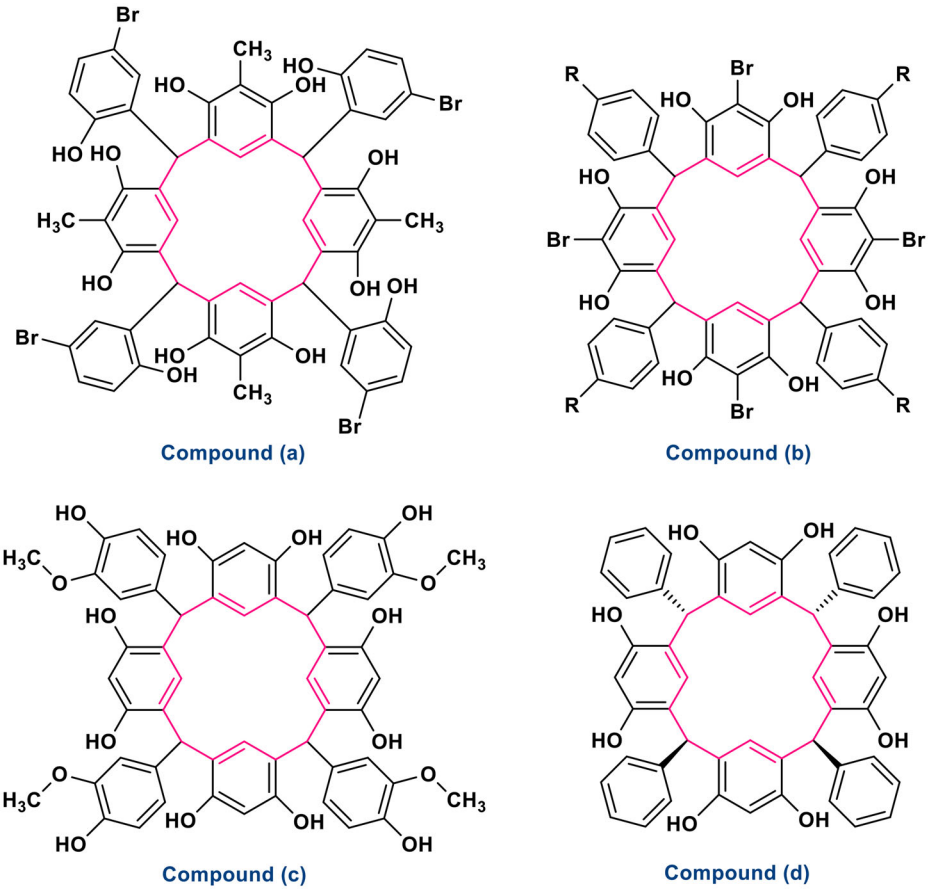
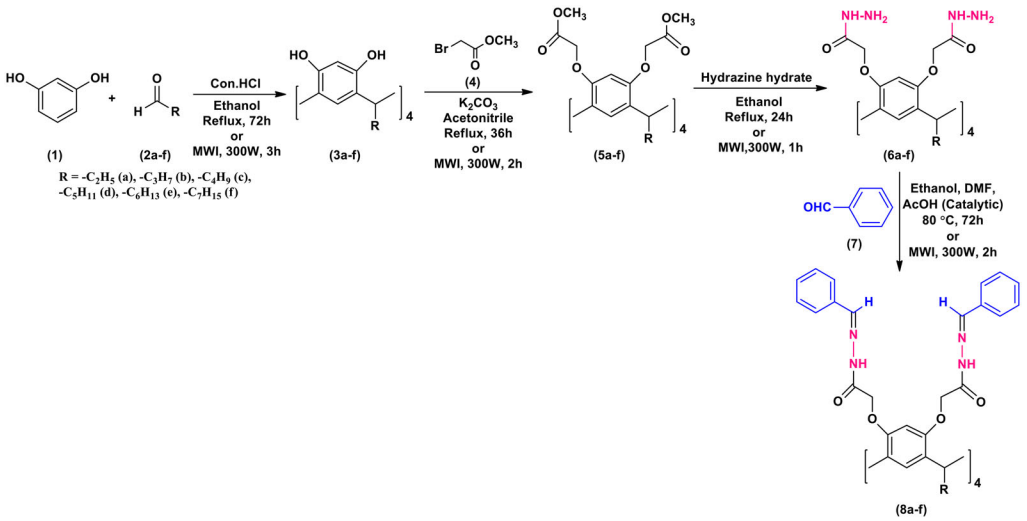


Figure 1. Some bio-active resorcin[4]arene derivatives.

properties at various temperatures [72–74]. Fang et al. have prepared calix[4]arene hybrid dimeric cholesteryl derivative with the presence of naphthalene linker [75]. Recently, Sharma et al. developed various supramolecular liquid crystals based on calix[4]arene core and studied their optical and luminescence properties [76].

Due to their simply modifiable framework, resorcin[4]arene is a major class of supramolecular host that can be used in the structure to add particular functionality [77,78]. To change the property of resorcin[4]arene system, various functional groups are introduced and used to replace the hydroxyl groups. Resorcin[4]arene has a capability for molecular identification, interacted maximum guest molecules, modification, and further manufactured in high amounts [24].

Resorcin[4]arene were used to design and synthesized biological active compounds [79,80] and also rare host molecules for chemical and biological study [81]. Resorcin[4]arene derivatives (Figure 1) exhibited some bio-active molecules in which **Compound (a)** [82] gives moderate antibacterial activity, good antiviral activity, and antiHSV-1 (Herpes simplex virus) activity, whereas **Compound (b)** [83] gives good antibacterial activity. Here, **Compound (c)** [84] gives excellent antioxidant activity with low cytotoxicity and **Compound (d)** [85] gives moderate antimicrobial activity against Gram-positive bacteria.



Scheme 1. Synthesis of Schiff bases of resorcin[4]arene derivatives **(8a–f).**

The green preparation of organic compounds in the field of medicinal chemistry can be recognized as an attractive research prospect, because of energy savings, waste reduction, atom economy, avoiding the use of hazardous chemicals, and easy work-up processes are progressive benefits related with these syntheses [86,87].The expansion of effective anti-microbial derivatives becomes one of the most important areas of antibacterial research today, because of their resistance to the existing antibacterial and antifungal drugs [88–90]. Thus, new antimicrobial agents are urgently essential to survive this situation.

Inspired from the above facts, we have synthesized novel Schiff bases of resorcin[4]arene derivatives and evaluate their antimicrobial activity. Here, resorcin[4]arene **(3a–f)**derivatives were synthesized from the reaction of resorcinol**(1)** with different aldehydes **(2a–f)** in presence of acatalytic amount of conc. HCl in refluxing ethanol. After that, we have synthesized Resorcin[4]arene methyl ester **(5a–f)** and their corresponding hydrazine derivatives **(6a–f)** in excellent yield. These hydrazide derivatives finally reacted with benzaldehyde to form different Schiff bases of resorcin[4]arene derivatives **(8a–f)** (Scheme 1).

Result and discussion

Initially, we have synthesized resorcin[4]arene derivatives **(3a–f)**from the reaction of resorcinol with a various aldehyde in presence of a catalytic amount of conc. HCl under-refluxing ethanol for 72 h or under microwave irradiation (MWI) at 300 W for 3 h. Here, we have used only aliphatic aldehydes, which are attached at feet of resorcin[4]arene. Further, we have carried out the synthesis of resorcin[4]arene methyl ester **(5a–f)** through the reaction of derivatives **(3a–f)** with methyl 2-bromoacetate in the presence of K_2CO_3 in acetonitrile at 70 °C for 36 h or under MWI at 300 W for 2 h. To optimize the reaction conditions, the same reaction was carried out in the presence of solvents like DMF and THF. However, in both cases comparatively lower yield of

Table 1. Preparation of Schiff base of hydrazine carbonyl methyl Resorcin[4]arene and their derivatives.

		Thermal heating (80 °C)		Microwave irradiation (300 W)		
Comp. code	*R*	Time (h)	Yield[a](%)	Time (min)	Yield[a](%)	Mp (°C)
8a	C_2H_5	72	64.71	3	82.34	121–123
8b	C_3H_7	72	89.36	3	92.60	139–140
8c	C_4H_9	72	64.76	3	85.82	143–144
8d	C_5H_{11}	72	54.34	3	76.22	134–135
8e	C_6H_{13}	72	90.00	3	93.56	128–130
8f	C_7H_{15}	72	57.43	3	79.00	145–146

[a]Isolated yields.

product was obtained. After that, hydrazide derivatized resorcin[4]arenes **(6a-f)** were prepared by the reaction of derivatives **(5a–f)** and hydrazine hydride under refluxing ethanol for 24 h or under MWI at 300 W for 1 h. Finally, the benzaldehyde was treated with derivatives **(6a–f)** in ethanol: DMF as a dual solvent media in the presence of a catalytic amount of acetic acid at 80 °C for 3 days or under MWI at 300 W for 2 h to obtain the different Schiff base derivatives **(8a–f)**. All the newly synthesized Schiff bases **(8a–f)** were purified either by column chromatography or by recrystallization with ethanol. Compared to conventional heating, reaction under MWI showed higher proficiency in terms of product yields and reaction times (Table 1).

The reaction proceeded smoothly and provided excellent yields in all cases (Table 1). Various long carbon chain aldehydes reacted smoothly with this protocol to afford the excellent yields of the products. The purity of the synthesized compounds was confirmed by TLC and elemental analysis. The structure of the final products was well characterized by using spectral analysis techniques (IR, Mass, ^{1}H-NMR, and ^{13}C-NMR).

IR spectra displayed characteristic -NH stretching bands between 3200 and 3400 cm^{-1} is corresponding to -CONH group as well as the peak between 1680 and 1700 cm^{-1} also confirms the presence of amide in the structure. The ^{1}H-NMR spectrum showed a singlet nearer 4.4 to 4.7 ppm, which indicated a proton of the –O-CH_2-CO- linkage, while singlet nearer 7.6–8.2 ppm, which indicated a proton of the -CONH group. In addition, peaks between 6.6 and 8.8 ppm were observed for respective aromatic protons. The ^{13}C-NMR spectrum displayed peak nearer 170–176 ppm, which indicated C = O group of amide. The ESI-MS spectra of compounds **(8a–f)**, showed corresponding $(M+1)^+$ peak.

Electronic properties

Electronic properties such as ionization potential (IP), electron affinities (EA), and coefficients of HOMO and LUMO of compound **(8a)** are simulated at the R83LYP/3-21G (d,p) level (Figure 2).

The negatives of HOMO and LUMO are estimated as IP and EA, respectively. The IP and EA of compound **(8a)** are −0.20 and −0.08 eV. The electron transitions occur from the ground state to the excited state due to their $\pi \rightarrow \pi$ transition. Here, the negative value of HOMO–LUMO gap indicates that the molecule is stable. Also, the reason for the decreasing energy of the HOMO–LUMO energy gap is the eventual charge

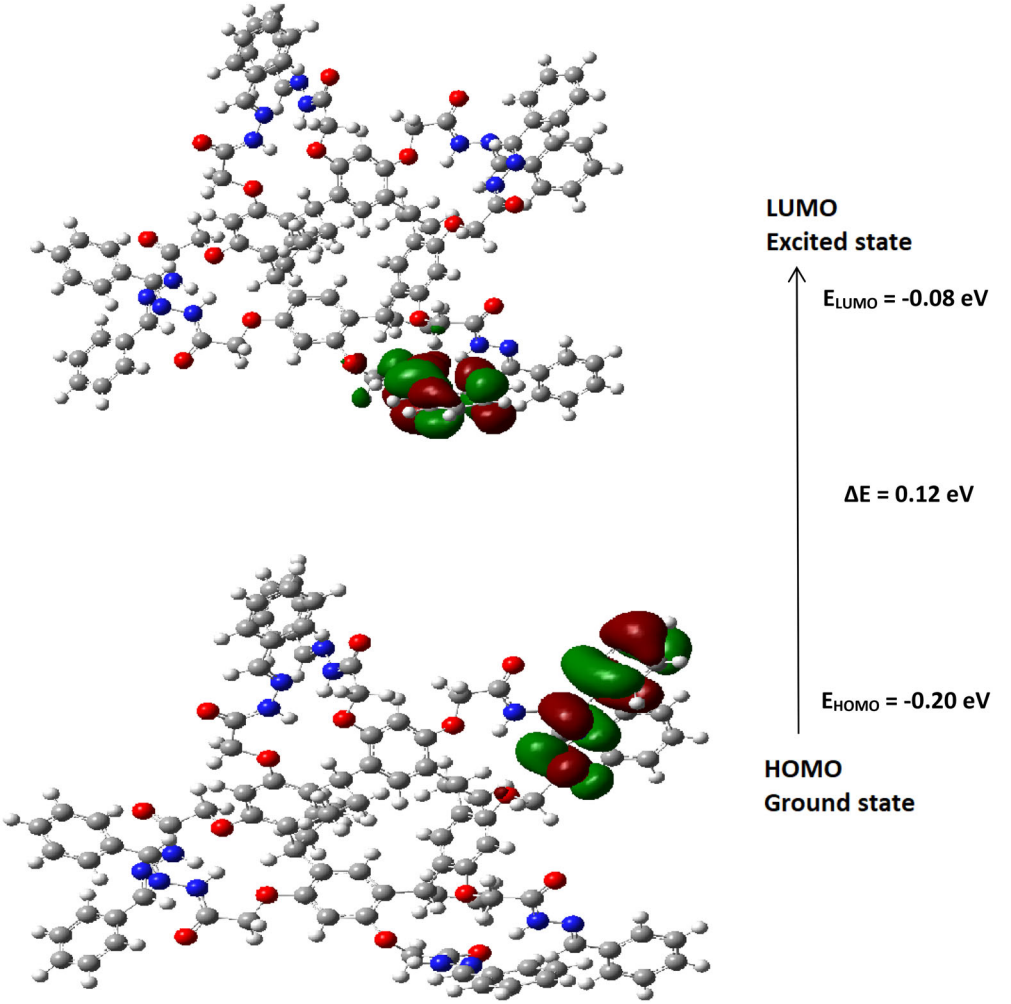


Figure 2. The HOMO–LUMO isodensity surface plots for the (**8a**).

transfer interaction taking place within the molecules because of the strong electron-accepting ability of the electron acceptor group [91].

POM investigation

The liquid crystalline phase of present synthesized Schiff base based resorcin[4]arene derivatives **(8a–f)** were primarily investigated by polarizing optical microscope (POM) (Figure 3). It can be observed that the texture pattern of nematic phase in compounds **(8a–8b)** on heating as well as cooling condition respectively. Compounds **(8a)** showed droplets of nematic phase at 112 °C which is further isotropic at 128 °C. Further, a similar texture pattern is observed in the cooling phase. Compound **(8 b)** displayed a nematic phase at 126 °C on heating and 121 °C on cooling condition. In addition, compound **(8c–8d)** showed a similar texture pattern of the nematic phase. Compound

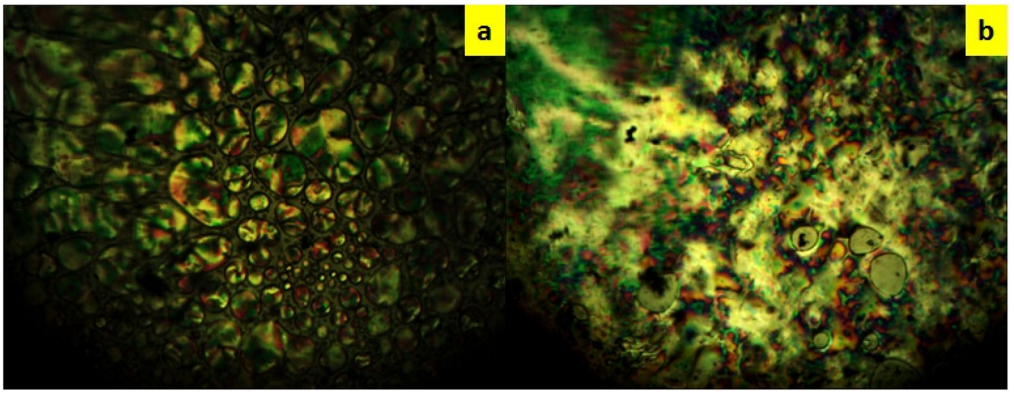


Figure 3. (a) Nematic phase at 50 °C for compound **8e**; (b) Nematic phase at 80 °C for compound **8c**.

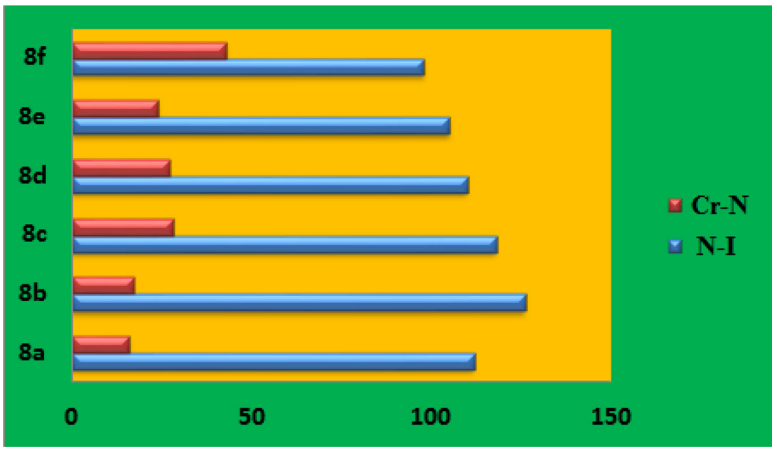


Figure 4. Bargraph showing the thermal stability of compounds (**8a-f**) by POM.

(8e) showed nematic phase at 105 °C on heating and 109 °C on cooling temperature. Similarly, Compound **(8f)** shows threaded type texture image of a nematic phase at 98 °C on heating condition. The mesophase temperature range and phase transition curve is given in below (Figures 4 and 5).

DSC investigation

The mesomorphic behavior of present synthesized Schiff base functionalized resorcin[4]arene core based derivatives **8a–8f** were preliminarily studied by using differential scanning calorimetry (DSC) (Figure 6). The transition temperatures along with enthalpy changes obtained from the DSC scans of second heating and cooling cycles are mention in Table 2. In DSC scans, compound **8a–8f** exhibited two endothermic peaks corresponding to crystal to nematic mesophase and nematic mesophase to isotropic liquid phase on heating as well as cooling condition. From these results, it can be noted that all the synthesized derivatives showed enantiotropical nematic mesophase. Compound **8a** with ethylchain exhibited two endothermic peaks at 119.2 °C and 131.6 °C on heating

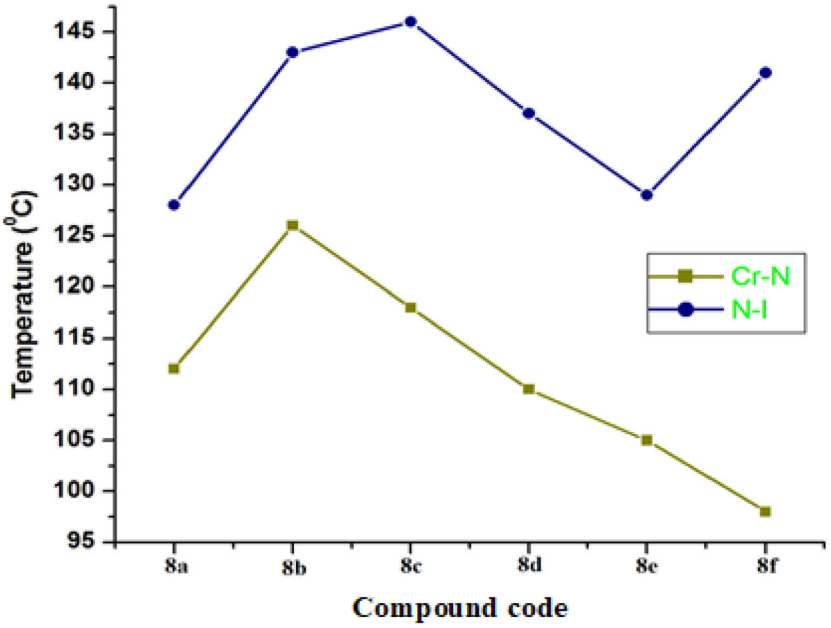


Figure 5. Phase diagram of compound (**8a–f**) by POM.

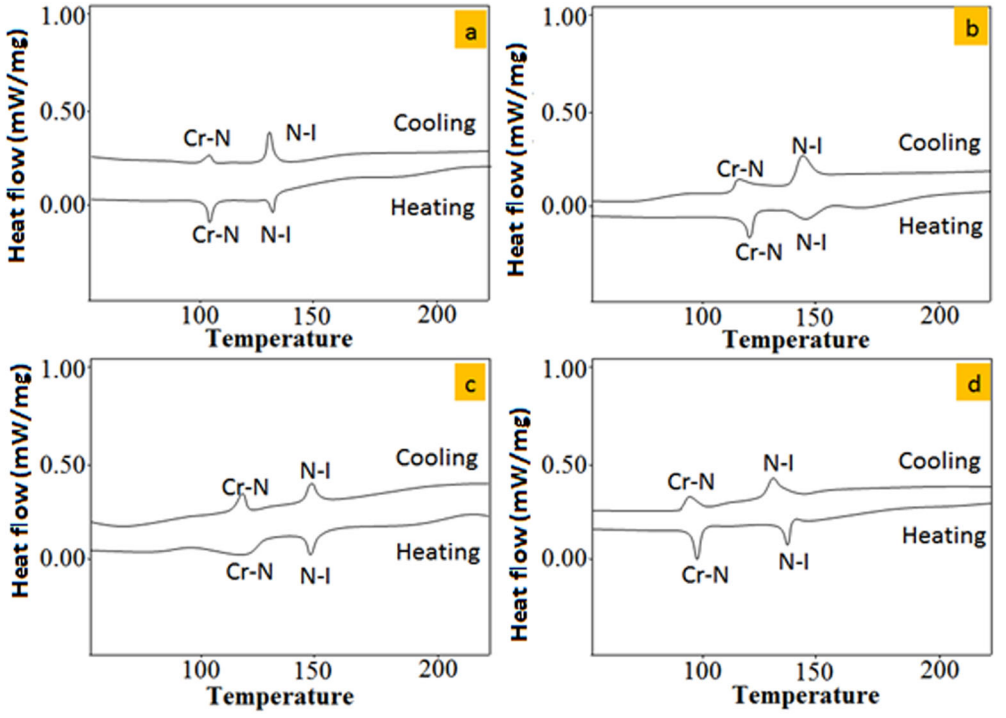


Figure 6. The DSC traces of compounds (a) **8a**, (b) **8b**, (c) **8c**, and (d) **8d** on first heating and cooling (scan rate 10 °C/min).

Table 2. Phase transition temperatures (°C) and corresponding enthalpies (kJ/mol) of synthesized compounds.

	Phase sequence	
Compound	Heating	Cooling
8a	Cr 119.2 (2.3) N 131.6 (9.4) I	I 128.4 (11.3) N 116.3 (2.3) Cr
8b	Cr 123.5 (1.4) N 147.3 (11.5) I	I 144.6 (9.4) N 124.8 (3.6) Cr
8c	Cr120.1 (1.9) N 151.8 (8.6) I	I 149.4 (6.1) N 118.8 (4.2) Cr
8d	Cr108.5 (2.1) N 143.8 (9.6) I	I 139.7 (8.4) N 105.7 (3.2) Cr
8e	Cr107.3 (3.1) N 131.5 (11.6) I	I 129.4 (6.2) N 104.6 (2.4) Cr
8f	Cr98.7 (4.2) N 142.6 (12.7) I	I 139.6 (7.3) N 96.1 (3.1) Cr

[a]Peak temperatures in the DSC thermograms obtained during the second heating cooling cycles at 10 °C/min; N = nematic phase; I = isotropic phase.

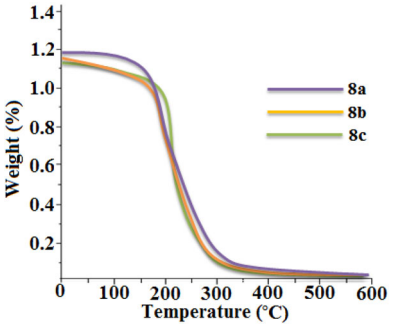


Figure 7. TGA curves of the compounds **8a–8c** carried out at a rate of 10 °C/min.

condition while on cooling condition, two endothermic peaks again traced at 128.4 °C and 116.3 °C. Compound **8b** with propylchain showed two endothermic peaks at 123.5 °C and 147.3 °C on heating and at 144.6 °C and 124.8 °C on cooling condition respectively. One can see that the higher value of enthalpy change in N-I phase transition is occurred as compare to Cr-N phase transition due to disordered molecular arrangement of 3D-shaped functionalized resorcin[4]arene central core. Compound **8c**and compound **8d** showed first endothermic peaks at 120.1 °C and 118.8 °C on heating condition and second endothermic peaks at 151.8 °C and 149.4 °C with a lower enthalpy change value to form N-I phase transition peak noticed in DSC scans. It can be noted thatthe compounds **(8d–8f)** with higher periphery alkyl chain showed higher mesophase range as compared to compounds (**8a–8c)**, which contain lower mesophase transition temperature. The result obtained from DSC study is further confirmed by using POM analysis.

Thermogravimetric analysis

Thermal stability of compounds **8a–8c** were studied by thermogravimetric analysis (TGA) under nitrogen atmosphere mention in Figure 7. All the synthesized Schiff base functionalized calix[4]resorcinarene based supramolecular derivatives were stable at least up to ≈ 230°C and complete degradation occurs at around 320 °C which showed the good stability of these synthesized compounds, respectively. As there is no loss seen up to temperature at around 130 °C that means no water or any other solvent entrapped in mesophase formation. All the synthesized supramolecular compounds show good

Table 3. *In vitro* antibacterial activity of compounds (8a–f).

	MIC (μg/mL)			
Comp. Code	*E. Coli* MTCC 443	*P. Aeruginosa* MTCC 1688	*S. Aureus* MTCC 96	*S. Pyogenus* MTCC 442
8a	100	125	200	250
8b	62.5	200	62.5	100
8c	100	62.5	200	250
8d	50	100	100	125
8e	100	200	125	100
8f	25	100	125	250
Gentamycine	0.05	1	0.25	0.5
Ampicillin	100	100	250	100
Chloramphncol	50	50	50	50
Ciprofloxacin	25	25	50	50
Norfloxacin	10	10	10	10

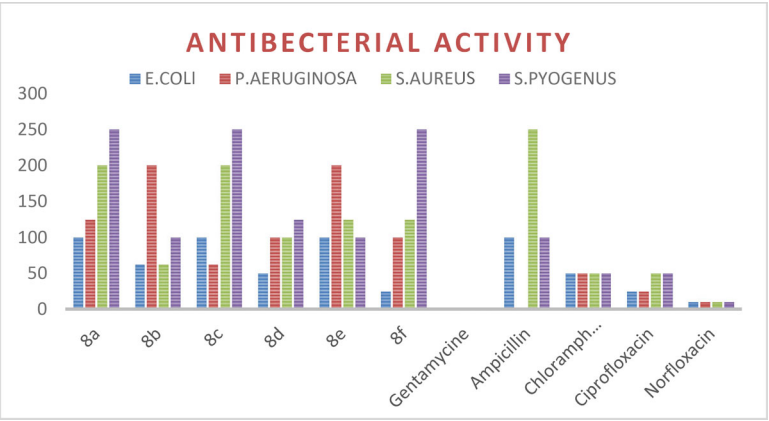


Figure 8. Graphical representation of antibacterial activity of compound (**8a–f**).

thermal stability as also confirmed by the POM temperature range as well. From the POM study, compounds **8a–c** showed mesophase temperature range for nematic to isotropic phase is 16°, 17°, and 28°, respectively. Further increasing alkyl chain in functionalized resorcinarene resulted in an increment of the mesophase temperature range.

Pharmacology

In vitro antibacterial activity

The newly synthesized Schiff base of resorcin[4]arene derivatives were screened for their *in vitro* antibacterial activity (Table 3). The bioassay results demonstrated that Schiff bases **(8a–f)** succeeded to indicate remarkably activity against the mentioned microorganisms as compared to standard drugs (Figure 8). In general, most of the tested derivatives showed better activity against the Gram-positive strain *S. aureus* as well as the Gram-negative bacteria *E. coli*.

Among the Gram-positive bacterial strain, *S. aureus* exhibited comparatively higher sensitivity toward the screened derivatives. In this view, the derivative **(8b)** was found to be the most active and that inhibits the *S. aureus* bacterial growth at the lowest minimum inhibitory concentration (MIC) value of 62.5 µg/mL than standard drug Ampicillin (MIC 250 µg/mL). Furthermore, compounds **(8e)** and **(8f)** showed excellent

Table 4. *In vitro* antifungal activity of compounds (**8a–f**).

	MIC (μg/mL)		
Compound code	*C. albicans* MTCC 227	A. *niger* MTCC 282	A. *clavatus* MTCC 1323
8a	250	500	500
8b	50	>1000	>1000
8c	250	500	1000
8d	100	>1000	>1000
8e	500	500	500
8f	250	1000	1000
Nystatin	100	100	100
Greseofulvin	500	100	100

activity with MIC value 125 µg/mL against *S. aureus* as compared to standard drug Ampicillin. Furthermore, molecules **(8d)** showed excellent potency (MIC 100 µg/mL) against *S. aureus* as compared to standard reference Ampicillin (MIC 250 µg/mL), but 50% less active than Chloramphenicol (MIC 50 µg/mL), and Ciprofloxacin (MIC 50 µg/mL). With regard to the activity against *S. pyogenes*, derivatives**(8b)** and **(8e)** showed good activity (MIC 100 µg/mL), which is equally potent to the Ampicillin (MIC 100 µg/mL) but 50% less active than Chloramphenicol (MIC 50 µg/mL) and Ciprofloxacin (MIC 50 µg/mL). Rest of the compounds displayed moderate activity against *S. pyogenus* bacterial strain.

On the other hand, activity against the two tested Gram-negative strains revealed that derivative**(8f)** (MIC 25 µg/mL) displayed outstanding activity against *E. coli* as compared to standard drug Ampicillin (MIC 100 µg/mL). Furthermore, derivative**(8d)** (MIC 50 µg/mL) and derivative**(8b)** (MIC 62.5 µg/mL) exhibited excellent potency against *E. coli* as compared to standard drug Ampicillin. Furthermore, compounds **(8a)**, **(8c)**, and **(8e)** were showed equipotent activity to Ampicillin (MIC 100 µg/mL) against *E. coli*, but 50% less active than Chloramphenicol (MIC 50 µg/mL). Moreover, compound **(8c)** was inhibits the Gram-negative *P. Aeruginosa* bacterial growth at the lowest minimum inhibitory concentration (MIC) value of 62.5 µg/mL as compared to standard reference ampicillin (MIC 100 µg/mL). Furthermore, compound **(8f)** was showed equipotent activity to Ampicillin (MIC 100 µg/mL) against *P. aeruginosa* bacterial strain.

In vitro antifungal activity

Antifungal activity data (Table 4) displayed that among the derivatives **(8a–f)**, compound **(8b)** displayed excellent activity with MIC 50 μg/mL against *C. albicans* as compared to Nystatin (MIC 100 µg/mL) and Greseofulvin (MIC 500 µg/mL). Furthermore, compound **(8d)** showed excellent activity with MIC 100 μg/mL against *C. albicans* equipotent to Nystatin (MIC 100 µg/mL). Moreover, compounds **(8a)**, **(8c)**, and **(8f)** exerted good inhibitory efficiency (MIC 200 µg/mL) against the *C. albicans* as compared to standard drug Greseofulvin (MIC 500 µg/mL). Moreover, compounds **(8a–f)**, exhibited moderate inhibitory activity against the *A. niger* and *A. clavatus* (Figure 9).

Experimental

Materials and methods

All chemicals of the highest purity available were purchased from commercial sources and used as received. The progress of the reaction was monitored by thin layer

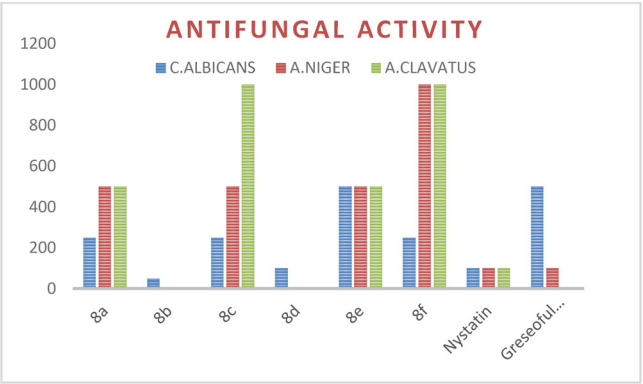


Figure 9. Graphical representation of antifungal activity of compound (**8a–f**).

chromatography (TLC) on Merck precoated silicagel 60 F254 aluminum sheets, visualized by UV light.

The reactions were performed under Samsung modified microwave oven. Melting points were measured on an Optimelt MPA 100 melting point apparatus and are uncorrected. FTIR spectra were taken on a Perkin-Elmer FT-IR 377 spectrometer using KBr pellets in cm^{-1}. Microanalysis was performed on Perkin-Elmer PE 2400 CHN analyzer. The texture images were studied on a trinocular optical polarizing microscope (POM) equipped with a heating stage. The phase transition temperatures were measured using Shimadzu DSC-50 at heating and cooling rates of 10 °C min^{-1}. The samples were heated from room temperature to 550 °C at 10 °C/min. ^1H NMR spectra were recorded on a Bruker AV 400 MHz spectrometer using $CDCl_3$ as a solvent, TMS as an internal reference. Mass spectra were recorded on Advion appearance CMS, USA. Ethyl acetate was utilized as mobile phase and electron spray ionization (ESI) is used as the ion source. Elemental analysis was performed on a CHNS elemental analyzer. Resorcin[4]arene derivatives **(3a–f)** were prepared according to previously reported methodology [92]. The resorcin[4]arene methyl esters **(5a–f)** were prepared according to procedure reported by Pansuriya and coworker [23].

General process for preparation of Resorcin[4]arene derivatives (3a–f)

Take ethanolic solution of resorcinol (1 mol) in RBF and add con. HCl (1.81 mol) dropwise with constant stirring during 15 min at 0 °C. Now, add aldehyde (1 mol) (Propanal, Butanal, Pentanal, Hexanal, Heptanal, Octanal) into the reaction mixture at same temperature. After that, reaction mixture was stirred for 3 days at reflux temperature or irritated under microwave oven at 300 W for 3 h. After completion of reaction, indicated by TLC, the reaction mixture was cooled to room temperature and poured into crushed ice-water mixture. Obtained precipitates were filtered and washed with ethanol/water (1:1) and dried in oven at 45 °C for 24 h.

General process for preparation of Resorcin[4]arene methyl ester (5a–f)

In three neck RBF take resorcin[4]arenes (1 mol) in dry acetonitrile. Anhydrous potassium carbonate (20 mol) was added into the reaction mixture and stirred for 10 min at 70 °C.

After that, methyl acetyl bromide (16 mol) was added drop-wise into the reaction mixture and heated 70 °C for 2 days or irritated under microwave oven at 300 W for 2 h. After completion of reaction, indicated by TLC, solvent was removed under vacuum. The residue extracted with MDC and water. The organic layer was separated and dried over anhydrous magnesium sulfate ($MgSO_4$) to remove the water molecules. Remove the solvent under vacuum and obtained solid was enough pure and no further purification was needed.

General process for preparation of hydrazide derivative of carbonyl methyl resorcin[4] arene (6a-f)

Take ethanolic solution of resorcin[4]arene methyl ester (1 mol) in RBF. Then add hydrazine hydrate (25 mol) with constant stirring. The reaction mixture was refluxed for 24 h or irritated under microwave oven at 300 W for 1 h. After completion of reaction, indicated by TLC, cool the reaction mixture at room temperature. The obtained precipitates were filtered out and washed with Ethanol/water (1:1) and dried the product in oven.

General process for preparation of Schiff base of resorcin[4] arene (8a-f)

Take hydrazide derivative of carbonyl methyl resorcin[4]arene (1 mmol) into EtOH: DMF (4:1) as dual solvent media in RBF, add benzaldehyde (16 mol) into the reaction mixture followed by few drops (3–4 drops) of acetic acid. The reaction mixture was then heated 80 °C for 3 days or irritated under microwave 2 h with a fixed power of 300 W. The reaction progress was monitored by TLC. After completion of the reaction, the mixture was added into cold water. Obtained precipitates were filtered and washed with Ethanol/water (1:1) and dried in oven. All compounds were purified by either column chromatography or by crystallization with ethanol.

Antibacterial and antifungal activity

Mueller-Hinton broth and Sabouraud's broth were used as nutrient medium to grow bacteria and fungus, respectively. Inoculum size for the test strain was adjusted to 10^6 colony-forming unit (CFU) per milliliter by comparing the turbidity. Serial dilutions were prepared in primary and secondary screening. The control tube containing no antibiotic was immediately subcultured (before inoculation) by spreading a loopful evenly over a quarter of plate of medium suitable for the growth of test organism and incubated at 37 °C for bacteria and 22 °C for fungi overnight. The MIC of the control organism was read to check the accuracy of the drug concentrations. The lowest concentration inhibiting growth of the organism was recorded as the MIC. Each test compound was diluted, obtaining 2,000 µg/mL concentration, as a stock solution. In primary screening 1000, 500, 250, and 125 µg/mL concentrations of the test compounds were taken. The active synthesized compounds found in this primary screening were further tested in a second set of dilution against all organisms. The drugs found active in primary screening were similarly diluted to obtain 100, 50, 62.5, 25, 12.5, and 6.25 µg/mL concentrations. The highest dilution showing at least 99% inhibition is taken as MIC.

All newly synthesized Schiff base of resorcin[4]arene derivatives**(8a-f)**were examined for antimicrobial activity against two gram-positive bacterial strains (*Staphylococcus aureus* MTCC 96 and *Streptococcus pyogenes* MTCC 442), two gram-negative bacterial strains

(*Escherichia Coli* MTCC 443, *Pseudomonas aeruginosa* MTCC 1688) as well as three fungal strains (*Aspergillus clavatus* MTCC 1323, *Candida albicans* MTCC 227 and *Aspergillus niger* MTCC 282) using the agar dilution method [93]. Ampicillin, Ciprofloxacin, and Chloramphenicol were used as standard control drugs for antibacterial activity, whereas Nystatin and Greseofulvin were used as standard control drugs for antifungal activity.

Physical and spectral data of compounds

N′-(phenylmethylidene)-2-{[2, 8, 14, 20 – tetraethyl – 6, 10, 12, 16, 18, 22, 24 – heptakis ({[N′-phenylmethylidene) hydrazinecarbonyl] methoxy}] pentacyclo [19.3.1.13′7.19′13.115′19] octacosa – 1 (24), 3 (28), 4, 6, 9 (27), 10, 12, 15 (26), 16, 18, 21 (25) dodecaen-4-yl] oxy} acetohydrazide (8a)

White solid. IR (KBr) (λ_{max}) cm^{-1}: 3443.63 (-N-H), 1696.20 (>C = O), 3028.16 (Ar-CH), 1443.27 (-CH_2), 1365.95 (-CH_3), 1696.90 (-C = O-NH-), 3028.16 (=C-H), 1224.19 (-C-O-C-), 1498.17 (-C = C-Ar).^{1}H NMR(400 MHz, $CDCl_3$, δ ppm): 8.25 (s, 8H, -N-H-), 7.64-7.52(s, 8H, -N = CH-), 7.35–7.19 (m, 40H, Ar-H),7.10 (s, 4H, Ar-H), 6.32–6.08 (s, 4H, Ar-H), 4.54–4.12 (s, 16H, -OCH_2CO-), 3.60–3.50 (t, 4H, -Ar-CH-Ar-), 1.95–1.86 (q, 8H, -CH_2), 0.78–0.76 (s, 12H, -CH_3). ^{13}C-NMR (100 MHz, DMSO, δ ppm): 12.7, 26.9, 37.3, 69.3, 95.5, 125.7, 126.0, 156.3, 128.8, 129.2, 131.0, 133.71, 144.1, 156.3, 171.0. Mass (m/z): 1883.20 $[M+1]^+$. Elemental Analysis: Calculated for $C_{108}H_{104}N_{16}O_{16}$ (1882.08): C 68.92%, H 5.57%, N 11.91%; Found: C 68.90%, H 5.55%, N 11.90%.

2-{[6,10, 12, 16, 18, 22, 24 – Heptakis ({[N′- (phenylmethylidene) hydrazinecarbonyl] methoxy}) -2, 8, 14, 20 tetrapropyl-pentacyclo[19.3.1.13′7.19′13.115′19] octacosa-1 (24), 3 (28), 4, 6, 9 (27), 10, 12, 15 (26), 16, 18, 21 (25), 22 – dodecaen– 4 - yl} - N′- phenylmethylidene acetohydrazide (8b)

White solid. IR (KBr) (λ_{max}) cm^{-1}: 3417.27 (-N-H), 1686.18 (>C = O), 2955.18 (Ar-CH), 1499.21 (-CH_2), 1383.11 (-CH_3), 1596.02 (-C = O-NH), 759.42 (=C-H), 1090.92 (-C-O-C-), 1596.02 (-C = C-Ar). ^{1}H NMR (400 MHz, $CDCl_3$, δ ppm): 8.22 (s, 8H, -N-H-), 7.69–7.63 (s, 8H, -N = CH-), 7.32-7.16 (m, 40H, Ar-H),7.14 (s, 4H, Ar-H), 6.34–6.11 (s, 4H, Ar-H), 4.59–4.14 (s, 16H, -OCH_2CO-), 3.65–4.49 (t, 4H, -Ar-CH-Ar-), 1.93–1.84 (q, 8H, -CH_2), 1.38–1.13 (m, 8H, -CH_2), 0.80–0.78 (s, 12H, -CH_3).^{13}C-NMR (100 MHz, DMSO, δ ppm): 14.4, 19.7, 34.8, 41.4, 69.3, 96.5, 125.7, 126.0, 128.8, 129.2, 131.0, 133.7, 144.1, 156.3, 171.0. Mass (m/z): 1938.7 $[M]^+$, 1939.77 $[M+1]^+$, 1940.7 $[M+2]^+$. Elemental Analysis: Calculated for $C_{112}H_{112}N_{16}O_{16}$ (1938.19): C 69.41%, H 5.82%, N 11.56%; Found: C 69.40%, H 5.80%, N 11.55%.

N′-(phenylmethylidene)-2-{[2, 8, 14, 20 – tetrabutyl – 6, 10, 12, 16, 18, 22, 24 – heptakis ({[N′-(phenylmethylidene) hydrazinecarbonyl] methoxy}) pentacyclo [19.3.1.13′7.19′13.115′19] octacosa – 1 (24), 3 (28), 4, 6, 9 (27), 10, 12, 15 (26), 16, 18, 21 (25), 22 – dodecaen – 4 - yl] oxy} acetohydrazide (8c)

White solid. IR (KBr) (λmax) cm^{-1}: 3060.37 (-N-H), 1697.22 (>C = O), 1441.59 (-CH_2-), 1352.27(-CH_3), 1628.23 (-C = O-NH), 3060.37 (=C-H), 1071.80 (-C-O-C-), 1498.40 (-C = C-).^{1}H NMR (400 MHz, $CDCl_3$, δ ppm): 8.26 (s, 8H, -N-H-), 7.70-7.62 (s, 8H,

-N = CH-), 7.35–7.19 (m, 40H, Ar-H),7.12 (s, 4H, Ar-H), 6.28–6.05 (s, 4H, Ar-H), 4.64–4.14 (s, 16H, -OCH_2CO-), 3.69–3.49 (t, 4H, -Ar-CH-Ar-), 2.02–1.72 (q, 8H, -CH_2), 1.35–1.08 (m, 16H, -CH_2), 0.85–0.80 (s, 12H, -CH_3).^{13}C-NMR (100 MHz, DMSO, δ ppm): 14.1, 23.0, 34.4, 35.1, 38.9, 69.3, 95.5, 125.7, 128.8, 129.2, 131.0, 133.7, 144.1, 156.3, 171.0. Mass (m/z): 1994.8 $[M]^+$, 1995.7 $[M+1]^+$. Elemental Analysis: Calculated for $C_{116}H_{120}N_{16}O_{16}$ (1994.33): C 69.86%, H 6.07%, N 11.24%; Found: C 69.85%, H 6.05%, N 11.22.

N′- (phenylmethylidene) – 2 - {[2, 8, 14, 20 – tetrapentyl – 6, 10, 12, 16, 18, 22, 24 – heptakis ({[N′-(phenylmethylidene) hydrazinecarbonyl] methoxy}) pentacyclo [19.3.1.13,7.19,13.115,19] octacosa – 1 (24), 3 (28), 4, 6, 9 (27), 10, 12, 15 (26), 16, 18, 21 (25), 22 – dodecaen – 4 – yl] oxy} acetohydrazide (8d)

White solid. IR (KBr) (λ_{max}) cm^{-1}: 3213.58 (-N-H), 1694.36 (>C = O), 3061.28 (Ar-CH), 3213.58 (-C = O-NH-), 3061.28 (=C-H), 1442.66 (-CH_2-), 1070.86 (-C-O-C-), 1498.14 (-C = C-Ar).^{1}H NMR (400 MHz, $CDCl_3$, δ ppm): 8.20 (s, 8H, -N-H-), 7.68-7.63 (s, 8H, -N = CH-), 7.51-7.19 (m, 40H, Ar-H), 7.10 (s, 4H, Ar-H), 6.23-6.09 (s, 4H, Ar-H), 4.63-4.11 (s, 16H, -OCH_2CO-), 3.67–3.51 (t, 4H, -Ar-CH-Ar-), 2.00–1.74 (q, 8H, -CH_2), 1.37–1.07 (m, 24H, -CH_2), 0.84–0.78 (s, 12H, -CH_3).^{13}C-NMR (100 MHz, DMSO, δ ppm): 14.1, 22.7, 26.3, 32.1, 35.1, 39.2, 69.3, 96.5, 125.7, 126.0, 128.8, 129.2, 131.0, 133.7, 144.1, 156.3, 171.0.Mass (m/z): 2050.8 $[M]^+$, 2051.87 $[M+1]^+$. Elemental Analysis: Calculated for $C_{120}H_{128}N_{16}O_{16}$ (2050.44): C 70.29%, H 6.29%, N 10.93%; Found: C 70.26%, H 6.27%, N 10.92%.

N′ - (phenylmethylidene) – 2 - {[2, 8, 14, 20 – tetrahexyl – 6, 10, 12, 16, 18, 22, 24 – heptakis ({[N′- (phenylmethylidene) hydrazinecarbonyl] methoxy}) pentacyclo [19.3.1.13′7.19′13.115′19] octacosa – 1 (24), 3 (28), 4, 6, 9 (27), 10, 12, 15 (26), 16, 18, 21 (25), 22 – dodecaen – 4 - yl] oxy} acetohydrazide (8e)

White solid. IR (KBr) (λ_{max}) cm^{-1}: 3211.78 (-N-H), 1693.81 (>C = O), 3061.73 (Ar-CH), 3211.78 (-C = O-NH), 693.17 (=C-H), 1444.96 (-CH_2-), 1071.19 (-C-O-C-), 1497.78 (-C = C-Ar).^{1}H NMR (400 MHz, $CDCl_3$, δ ppm): 8.24 (s, 8H, -N-H-), 7.71–7.64 (s, 8H, -N = CH-), 7.34–7.15 (m, 40H, Ar-H), 7.15 (s, 4H, Ar-H), 6.25–6.07 (s, 4H, Ar-H), 4.62–4.09 (s, 16H, -OCH_2CO-), 3.67–3.52 (t, 4H, -Ar-CH-Ar-), 2.05–1.77 (q, 8H, -CH_2), 1.37–1.06 (m, 16H, -CH_2), 0.89–0.82 (s, 12H, -CH_3).^{13}C-NMR (100 MHz, DMSO, δ ppm): 14.1, 22.7, 26.6, 29.6, 31.8, 35.8, 39.2, 69.3, 96.5, 125.7, 126.6, 128.8, 129.2, 131.0, 133.7, 144.1, 156.3, 171.0.Mass (m/z): 2106.06 $[M]^+$, 2107.8 $[M+1]^+$. Elemental Analysis: Calculated for $C_{124}H_{136}N_{16}O_{16}$ (2106.55): C 70.70%, H 6.51%, N 10.64%; Found: C 70.69%, H 6.50%, N 10.62%.

N′-(phenylmethylidene) – 2 - {[2, 8, 14, 20 – tetraheptyl - 6, 10, 12, 16, 18, 22, 2heptakis ({[N′ - (phenylmethylidene) hydrazinecarbonyl] methoxy}) pentacyclo [19.3.1.1^{3,7}.1^{9,13}.1^{15,19}] octacosa – 1 (24), 3 (28), 4, 6, 9 (27), 10, 12, 15 (26), 16, 18, 21 (25), 22 – dodecaen – 4 - yl] oxy} acetohydrazide (8f)

White solid. IR (KBr) (λ_{max}) cm^{-1}: 3403.20 (-N-H), 1693.77 (>C = O), 1598.91 (-C = O-NH), 758.80 (=C-H), 1439.48 (-CH_2-), 1382.01 (-CH_3), 1071.29 (-C-O-C-),

1498.71 (-C = C-). ^{1}H NMR (400 MHz, $CDCl_3$, δ ppm): 8.22 (s, 8H, -N-H-), 7.72-7.64 (s, 8H, -N = CH-), 7.34–7.18 (m, 40H, Ar-H), 7.17 (s, 4H, Ar-H), 6.24–6.03 (s, 4H, Ar-H), 4.62–4.07 (s, 16H, -OCH_2CO-), 3.69–3.51 (t, 4H, -Ar-CH-Ar-), 2.03–1.79 (q, 8H, -CH_2), 1.37–1.04 (m, 16H, -CH_2), 0.88–0.80 (s, 12H, -CH_3).^{13}C-NMR (100 MHz, DMSO, δ ppm): 14.1, 22.7, 26.6, 29.3, 31.9, 35.1, 39.2, 69.3, 96.5, 125.7, 126.0, 128.8, 129.2, 131.0, 133.7, 144.1, 156.3, 171.0. Mass (m/z): 2162.9 $[M]^+$, 2163.9 $[M+1]^+$. Elemental Analysis: Calculated for $C_{128}H_{144}N_{16}O_{16}$ (2162.61): C 71.09%, H 6.71%, N 10.36%; Found: C 71.08%, H 6.70%, N 10.35%.

Conclusion

The Schiff base of resorcin[4] arene derivatives were synthesized in excellent yield via microwave assisted methodology. All synthesized compounds displayed nematic mesophase due to the presence of short alkyl spacer and flexibility in Schiff base in alkoxy tail group. Themesogenic behavior of newly synthesized compounds were investigated by POM and DSC study. The electronic properties of compound **(8a)** was simulated for the first time. Compared to conventional heating, reaction under MWI showed higher proficiency in terms of product yields and reaction times. From the bioassays it is clear that the all Schiff base of resorcin[4]arene derivatives leads to outstanding antimicrobial potency. In the present study, derivatives **(8b), (8d),** and **(8f)** exhibited highly potent activity against most of the tested bacteria. In addition, derivative **(8b)** displayed excellent activity with MIC 50 μg/mL against *C. albicans* as compared to standard drug Nystatin.

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Disclosure statement

The authors declare no conflict of interest.

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