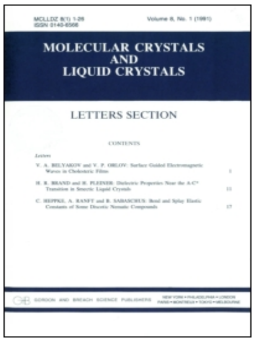

MCLLDZ 8(1) 1-28
ISSN 0140-6566
Volume 8, No. 1 (1991)

MOLECULAR CRYSTALS
AND
LIQUID CRYSTALS

LETTERS SECTION

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Molecular Crystals and Liquid Crystals

ISSN: 1542-1406 (Print) 1563-5287 (Online) Journal homepage: http://www.tandfonline.com/loi/gmcl20

[1,2,3]-triazole derivatives: Mesomorphic property dependence on the molecular shape

Souria Benallou, Salima Saidi-Besbes, Eric Grelet & Ahmed Bentaleb

To cite this article: Souria Benallou, Salima Saidi-Besbes, Eric Grelet & Ahmed Bentaleb (2017) [1,2,3]-triazole derivatives: Mesomorphic property dependence on the molecular shape, Molecular Crystals and Liquid Crystals, 647:1, 290-298, DOI: 10.1080/15421406.2017.1289614

To link to this article: http://dx.doi.org/10.1080/15421406.2017.1289614

MOLECULAR CRYSTALS AND LIQUID CRYSTALS
2017, VOL. 647, 290–298
http://dx.doi.org/10.1080/15421406.2017.1289614

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[1,2,3]-triazole derivatives: Mesomorphic property dependence on the molecular shape

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ABSTRACT
Nowadays, heterocyclic units have been extensively studied for the preparation of thermotropic liquid crystals due to their ability to impart lateral and/or longitudinal dipoles and induce changes in molecular shape. Key parameters to accede to stable mesophases are the position of the heterocycle and the nature of terminal and linking functions. We present in this paper, an overview of several series of 1,2,3-triazole liquid crystalline derivatives that we designed and characterized. We highlight the effect of different connecting functions and the geometry of mesogenic core on the mesomorphic behavior of these derivatives.

KEYWORDS
1,2,3-triazole; catalyzed 1,3-dipolar cycloaddition; X-ray diffraction; smectic mesophases

Introduction

Heterocyclic units attracted considerable attention as building blocks for the preparation of liquid crystalline compounds due to their particular properties and their structural diversity allowing for various mesogenic designs. Different supramolecular organizations have been targeted and tuned through appropriate functionalization of the heterocyclic core [1–5]. The rich polymorphism usually observed of such structures was attributed to their medium to strong lateral dipole, high polarizability and anisotropy, low symmetry and/or non-planar structure caused by nitrogen, oxygen, sulfur heteroatoms, etc [6–7].

Six membered aromatic heterocyclic rings containing one or two nitrogen atoms such as pyridine [8], tetrazine [9], pyrimidine [10] are known to be conducive to liquid crystalline organizations and in particular to smectic mesophases. Even if, such heterocycle ring changes slightly the geometry of mesogenic molecules in comparison with benzene homologous, the lone pairs of electrons on the nitrogen atoms introduce attractive forces which favor layer formation. The position of nitrogens may have a substantial effect on the polarisability and polarity of mesogenic molecules and thus on the mesophase stabilities. For calamitic mesogens, the mesophase is even more stable than the polarity and polarisability are high due to the intensification of side-chain interactions.

For a long time, the nonlinearity of five-membered heterocyclic rings was considered as a brake on the achievement of mesomorphic properties. The unfavorable deviation from linearity could be compensated by [11]: a) the lengthening of the mesogenic core, b) the introduction of functional goups allowing for the straightening out of the molecules (as imine

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Color versions of one or more of the figures in the article can be found online at www.tandfonline.com/gmcl.

function) or, c) the introduction of the heterocyclic ring at the end of the mesogenic core. Several five-membered heterocyclic rings have been investigated such as pyrrole, thiophene, isoxazole [12], tetrazole [13] and 1,2,4-oxadiazole [14] in the aim to establish a structure-liquid crystalline properties relationship. Nematic and smectic mesophases have been usually obtained with lower melting points than those obtained for classical 1,4-phenylene core owing to the reduced packing efficiency of this type of molecules.

For instance, nitrogen based heterocyclic liquid crystals [14–17] that exhibit tilted smectic liquid crystalline packing (SmC) have been explored, as achiral host LC, in mixtures with chiral dopants having a large spontaneous polarization to prepare ferroelectric liquid crystal materials [13]. The aim was to accede to materials with low viscosities and melting points and a wide range SmC phase for use in "guest-host" approach to ferroelectric mixtures.

1,2,3-triazole mesogenic derivatives

Since the development of copper-catalyzed azide-alkyne cyloaddition also known as 'Click Chemistry', [1,2,3]-triazole compounds have attracted much more attention in different fields of pharmaceutical industry, supramolecular chemistry and material chemistry for realizing highly efficient phosphorescent OLEDs [18–19] or for nonlinear optical applications [20–21]. 1,2,3-triazole combines multiple interesting properties including a high chemical and thermal stabilities, a strong dipole moment, an aromatic character and a good hydrogen-bond-accepting ability [22–23]. Despite, these outstanding features, 1,2,3-triazole heterocyclic has gained less interest to devise mesogenic compounds.

In view of this lack of 1,2,3-triazole LC materials, we have developed different triazole based mesogenic compounds with different shapes [24–26] (Figure 1).

The majorly of reported calamitic derivatives in literature contains a central rigid core composed of a phenyl or biphenyl ring directly linked to triazole heterocyclic at N1 and/or C4 position. We have shown in our previous studies the positive effect of the introduction of an ester function group within the triazole heterocycle, as a connector group, on the enhancement of thermal stability of mesophases (**1a-d, 2a-c**) [24]. This can be explained by the contribution of

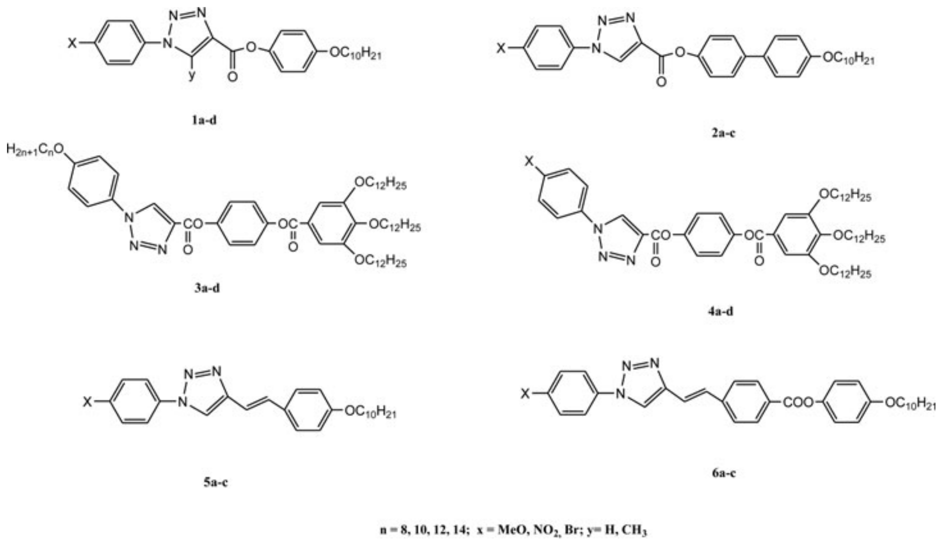

Figure 1. Synthesized 1,2,3-triazole mesogenic derivatives [24–26].

the ester group on the enhancement of dipole moment of mesogen molecules favoring layer organization of molecules within smectic A mesophase. It is worth mentioning that this type of organization was obtained even for short mesogens bearing only three aromatic rings.

This feature was exploited to prepare unsymmetrical tetracatenar compounds containing ester type [1,2,3]-triazole derivatives (**3a-d, 4a-d**) by the esterification of 1H-[1,2,3]-triazole-4-carboxylic acid compounds bearing octyl, decyl, dodecyl or tertadecyl oxychain with a tricatenar alcohol.

Stable hexagonal columnar organization of these derivatives was evidenced from X-ray diffraction (XRD) analyses with a very weak correlation of the molecules within the columns [25]. The building block was composed of a disc-like structure containing two molecules lying side by side. The lamellar ordering was maintained even in the solid structure.

The effect of alkene function as a linking group has been also investigated (**5a-c, 6a-c**). We demonstrated that the nature of obtained mesophases (SmA, SmC and N) and their stability could be tuned through a suitable terminal functionalization [26]. The elongation of the aromatic rigid core decreased by a factor of 2.5 the temperature range of smectic A mesophase stability of and induced the disappearance of SmC in favor of less ordered nematic phase. Trans isomers were more conducive to liquid crystalline packing in comparison with cis homologous and induced significantly higher stable smectic A mesophase and higher melting points. This type of molecules presents an extended conjugated structure and can potentially act as electron-transporting materials.

Recently, as part of our continuing effort in triazole derivatives, we investigate the effect of oxymethylene function as a connector group on mesomorphic behavior of triazole based mesogens. Symmetrical bis-1,2,3-triazole ether derivatives with a biphenyl central core were designed that exhibit an enantiotropic smectic C mesophase at high temperature.

These compounds (**11a-d**) were prepared according to the synthetic pathway described in Figure 2. They present a dimeric structure consisting of a 4,4′-biphenyl central core connected on both sides, through an oxymethylene unit, to 1-(4-(alkyloxy)phenyl)-1*H*-[1,2,3]-triazoyl group. The precursor used to accede to these compounds is the 4-azido-1-alkyloxybenzene (**9a-d**) which was obtained from 4-aminophenol using a three-step synthesis strategy as reported in a previous work [25]. Meanwhile, 4,4′-bis(propargyloxy)biphenyl (**10**) was synthesized by etherification of 4,4′-biphenol with two equivalents of propargylbromide in the presence of sodium hydride. Catalytic 1,3-dipolar cycloaddition of the arylazide derivatives (**9a-d**) with 4,4′-bis(propargyloxy)biphenyl (**10**) in the presence of CuI and catalytic amount of triethyamine affords the final products (**11a-d**). Under click chemistry condition, only the 1,4-regioisomers of compounds (**11a-d**) were obtained with good yields (between 56 and 77%). Their structures were supported by ^{1}H and ^{13}C NMR analyses and High Resolution Mass Spectroscopy.

The synthesized derivatives showed enantiotropic liquid crystalline behavior with very high transition temperatures (above 220°C) that cause partial thermal decomposition of few compounds after extensive annealing at high temperature (Table 1, Figure 3). On cooling from the isotropic liquid, elongated "bâtonnets" nucleated from the dark background and grew up to broken fan-shaped texture (Figure 4c-d). Schlieren textures were also observed for compounds **11b** and **11c** in cooling runs indicating the existence of SmC phase (Figure 4a-b).

Differential scanning calorimetry analysis confirmed the mesomorphic behavior. Three endothermic peaks, corresponding to crystallization, melting and isotropic transitions were observed. A typical thermogram obtained by DSC analysis of compound **11a** is presented in Figure 5.

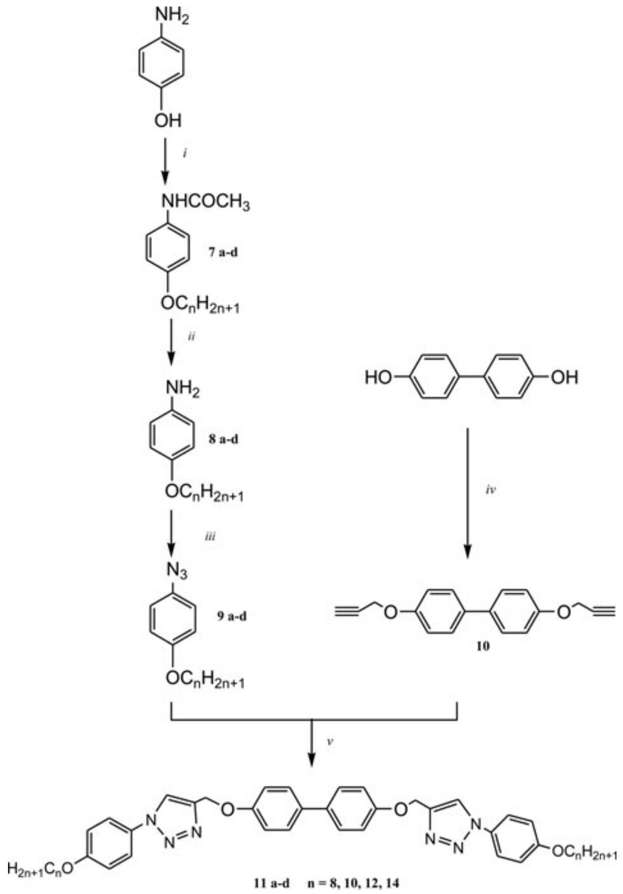

Figure 2. Reagents for the synthesis of triazole derivatives *(i)* 1. acetic anhydride, H_2O, 2. $C_nH_{2n+1}Br$ ($n = 8$, 10, 12, 14), K_2CO_3, butanone; *(ii)* HCl, H_2O; *(iii)* 1. HCl, $NaNO_2$, H_2O, 2. NaN_3, H_2O; *(iv)* propargyl bromide, NaH, DMF; *(v)* CuI, $N(C_2H_5)_3$, water/EtOH 1:1.

Table 1. Transition temperatures (°C) and transition enthalpies ΔH (KJ mole^{-1}) of compounds **5 a-d** determined by DSC (5°C min^{-1}) during the second cycle on heating.

Compound	n	Transitions temperatures (°C) [transition enthalpies ΔH (KJ mole^{-1})
11a	8	Cr(139.38)[10.21]Cr′ (212.5)[0.10]SmC(225.4)[47.31]I
11b	10	Cr(121.32) Cr′(139.1)[Cr+Cr′ = 35.8]Cr″(214.89)[40.74]SmC(227.31)[9.08]I
11c	12	Cr(141.54)[29.70]Cr′(213.39)[16.89]SmC(225.01)[1.32]I
11d	14	Cr(144.09)[52.03]Cr′(179.67)[[1.32]Cr‴(210.03)[34.79]SmC(225.64)[16.86]I

The thermal stability of the mesophase is independent of the length of the alkoxy terminal chain and is in the range of (12.4 to 15.6°C).

Cristano *et al.* reported analogue derivative of general formula 4-(4′-Decyloxybiphenyl-4-yl)-1-(4-decyloxyphenyl)-1H-[1,2,3]-triazole, that showed SmC phase in the thermal range of 184.9°C-246.1°C [27]. The structural difference with our compounds and especially compound **11b** bearing the same decyloxy chain is in the number of triazole ring and the nature of the connector linking the latter to biphenyl core (Cristano's compound contains one triazole ring and no connector). Compound **11b** exhibits higher melting transition temperature and

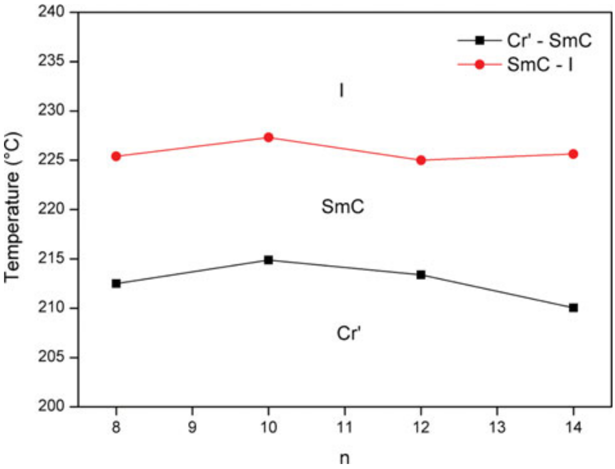

Figure 3. Comparative thermal behavior of compounds (**11a-d**).

narrow mesomorphic range. Two factors should be at the origin of the short mesophase stability: the rotation around the oxymethylene connector group that is free to take place and contributes to the loss of linearity and planarity of the mesogenic core disfavoring the mesophase formation and the linearity deviation caused by the two 1,2,3-triazole units. The bis-triazole

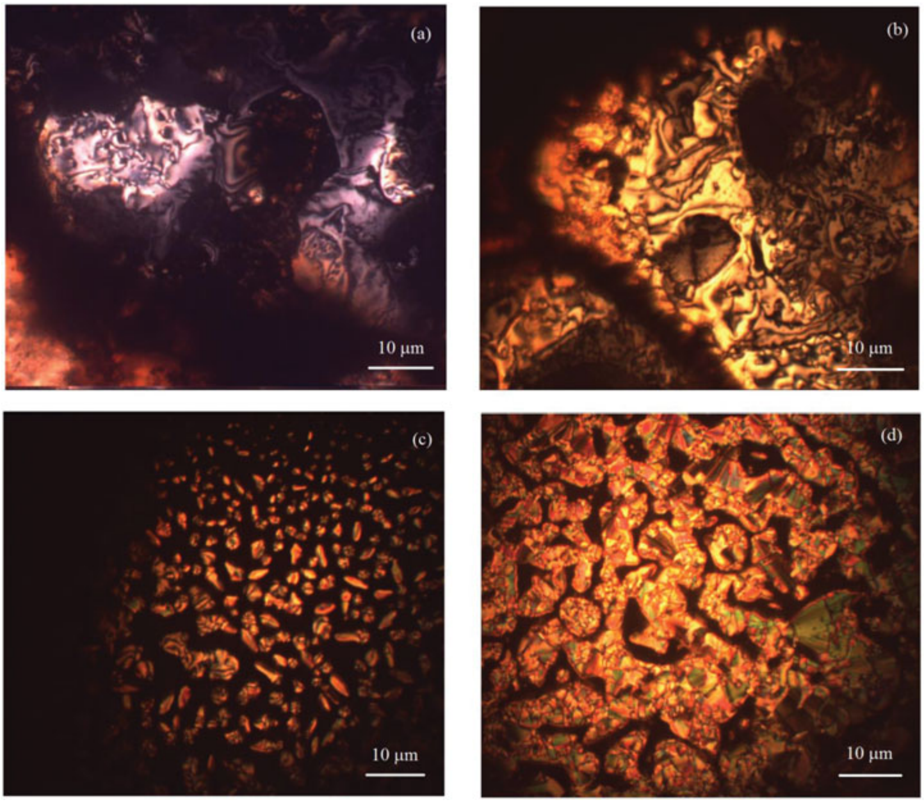

Figure 4. Optical textures observed on cooling (5°C/min) between cover slip and glass slide by polarized microscopy for (a) **11b** at 225°C, (b) **11c** at 226°C, (c) **11d** at 224°C and (d) **11d** at 212°C.

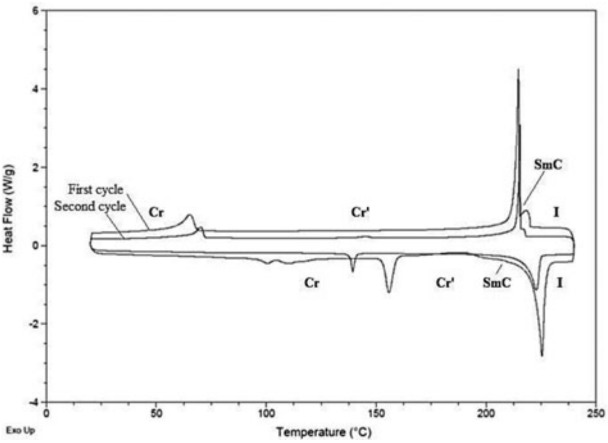

Figure 5. Thermogram of compound **11a** during the first and second cycles. The heating and cooling rates are 5°C min^{-1}.

mesogens reported in this paper are less linear than monotriazolic derivative; the heterocyclic ring may acts as a polar terminal substituent rather than a part of the central rigid core.

XRD experiments were carried out on compound **11b** to determine the nature of the mesophase. The XRD pattern for compound **11b** (Figure 6) showed two reflection peaks characterized by the main Bragg peaks $q_{100} = 1.61\ \mathrm{nm}^{-1}$ and the second-order reflection (200) at $3.19\ \mathrm{nm}^{-1}$ with ratio $q_{200}/q_{100} \approx 2$, confirming the lamellar organization. The signal of the disordered aliphatic chains corresponds to the broad reflection at about $13.7\ \mathrm{nm}^{-1}$, is characteristic of a liquid-like order. Due to the instability of investigated compounds to prolonged exposure at high temperatures, we were not able to carry out X-ray diffraction experiments to determine the temperature dependence of the layer spacing and therefore the nature of the local smectic order [25]. However, the comparison of the interlayer spacing $d = 3.90$ nm ($d = 2\pi/q_{100}$) with the molecular length $L = 4.61$ nm estimated from the energy minimized structure obtained using semi-empirical Hartree-Fock AM1 (Austin Model 1) level, indicated that the molecules are tilted within the layer. In this case, the tilted angle estimated from this

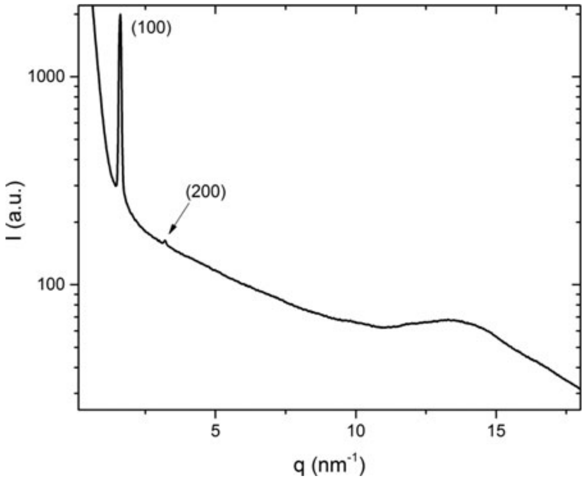

Figure 6. XRD pattern of compound **11b** at 225°C in the SmC mesophase.

molecular length using the following equation $\cos\theta = d/L$ will be around 32° and is consistent with SmC orgnaization deduced from POM textures.

Conclusion

Several liquid crystalline 1,2,3-triazole derivatives with different molecular shapes have been synthesized in order to investigate the relationship between molecular structure and mesomorphic properties. Rich polymorphism was exhibited by this heterocyclic synthon such as nematic, smectic and columnar phases. We demonstrated that the introduction of a polar connector group within the heterocyclic unit may have a significant effect on the nature of observed mesophases and their stability. Ester and alkene fucntion were more conducive to liquid crystalline phases than the oxymethylene group probably due to the lack of linearity of the latter mesogenic core.

Many scientific issues in this field still need to be explored in the feature in particular for the development of 1,2,3-triazole based discotic liquid crystals that have been very little reported in the litterature. This class of materials will be potentially interesting for optoelectronic devices and solar cell applications due to the thermal stability of these derivatives and their long-range self-assembly.

Experimental section

Characterization

^{1}H and ^{13}C NMR spectra were recorded on a Bruker 300 MHz spectrometer (Wissembourg, France). Tetramethylsilane was used as an internal reference for chemical shifts. Column chromatographies were carried out using Merck, Darmstadt silica gel (Kieselgel 60, 230–400 mesh) as the stationary phase (Merck Millipore, Gernsheim, Germany). Thin-layer chromatography was carried out on aluminum plates pre-coated with Merck silica gel $60F_{254}$ and visualized by means of ultraviolet fluorescence quenching. The melting points, transition temperatures and phase transition enthalpies were determined using a differential scanning calorimetry (DSC Q200®, TA Instruments) at a heating and cooling rate of 5°C min^{-1}. Mesomorphic textures were observed using a Zeiss Scope. A1 polarizing microscope equipped with Mettler Toledo heating stage.

X-ray diffraction experiments were performed by using a rotating anode generator (Rigaku Nanoviewer MicroMax 007HF) coupled to a confocal Max-Flux® Osmic mirror (Applied Rigaku Technologies, Austin, USA) producing beam with a wavelength of 1.54 Å together with a homemade heating stage with a thermal stability of 0.1°C. The spectra were recorded with a bi-dimensional detector (MARResearch-345) located at 308 mm from the sample, which was introduced as a powder in glass capillary tubes (Glas, Muller, Germany) having a diameter of 1.5mm. The spectra were analyzed using FIT2D software (ESRF; http://www.esrf.eu/).

Synthesis

All the reagents were purchased from Aldrich (Prochima Sigma, Tlemcen, Algeria) and used as received. The solvents were of commercial grade quality and were dried and distilled before use. Methylene chloride was distilled over calcium hydride. 4-azido-1-alkyloxybenzene (9a-d) were prepared according to our previous study from 4-aminophenol [25].

4,4′-bis(propargyloxy)biphenyl (10)

In a two-necked round bottom flask, is placed (0.97 g, 5.3 mmol) of 4,4′-biphenol solubilized in a minimum of DMF and (0.25 g, 10.6 mmol) of NaH. The reaction is stirred during 2 hours at room temperature under nitrogen atmosphere. The solution is cooled to 0°C then 10.6 mmol of propargyl bromide are added dropwise. The reaction is stirred at room temperature during 48 h. Afterwards, distilled water is added and the obtained precipitate is filtered, abundantly washed with water then recrystallized from ethanol.

Yield: 93%, mp = 176°C, *^1H NMR (C_2D_6O)*: 2.85 (s, 2H, C≡CH), 4.83 (s, 4H, HC≡C–$\underline{CH_2}$), 7.07(d, 4H, Ar-H, $^3J = 8.61$ Hz), 7.56(d, 4H, Ar-H, $^3J = 8.62$ Hz). *^{13}C NMR (C_2D_6O)*: 55.42, 76.18, 78.93, 115.18, 127.49, 156.97.

4,4′-bis (1-(4-alkyloxyphenyl)-1H-[1,2,3]-triazole)oxymethylenebiphenyl (11a-d)

(0.76 mmol) of the appropriate 4-azido-1-alkyloxybenzene (9a-d) and (0.1 g, 0.38 mmol) of 4,4′-bis(propargyloxy)biphenyl (10) are dissolved in 15 mL of a water/ethanol (1:1) mixture. Afterward, (14 mg, 0.076 mmol) of cupper iodide (CuI) and a catalytic quantity of triethylamine are added. The mixture is stirred at 70°C during 48 hours. After cooling to room temperature, the reaction mixture is filtered and the obtained solid is washed with ethanol then purified by flash column chromatography using acetone then dichloromethane as eluents.

4,4′-Bis (1-(4-octyloxyphenyl)-1H-[1,2,3]-triazole)oxymethylenebiphenyl (11a)

Yield: 77%, *^1H NMR ($CDCl_3$, 50°C)*: 0.91 (t, 6H, CH_3, $^3J = 6.33$ Hz), 1.31 (m, 20H, $CH_3\underline{(CH_2)}_5$), 1.83 (m, 4H, $OCH_2\underline{CH_2}$); 4.02 (t, 4H, $O\underline{CH_2}CH_2$, $^3J = 6.90$ Hz), 5.35 (s, 4H,OCH_2), 7.00 (d, 4H, Ar-H, $^3J = 8.96$ Hz), 7.11 (d, 4H, Ar-H, $^3J = 8.37$ Hz), 7.50 (d, 4H, Ar-H, $^3J = 8.37$ Hz, 7.62 (d, 4H, Ar-H, $^3J = 8.96$), 7.99 (s, 2H, H_5-triazole). ***^{13}C NMR** ($CDCl_3$)*: 14.09, 22.41, 25.75, 28.90, 28.97, 29.09, 31.55, 61.85, 68.43, 114.84, 115.06, 122.25, 127.62, 129.97, 133.95, 140.29, 157.15, 159.29. ESI-HRMS $[M+H]^+$: calculated for $C_{46}H_{57}N_4O_6$: 757.4436; found 757.4426.

4,4′-Bis (1-(4-decyloxyphenyl)-1H-[1,2,3]-triazole) oxymethylenebiphenyl (11b)

Yield: 56%, *^1H NMR ($CDCl_3$, 50°C)*: 0.91 (t, 6H, CH_3, $^3J = 6.33$ Hz), 1.31 (m, 28H, $CH_3\underline{(CH_2)}_7$), 1.83 (m, 4H, $OCH_2\underline{CH_2}$); 4.03 (t, 4H, $O\underline{CH_2}CH_2$, $^3J = 6.9$ Hz), 5.35 (s, 4H, OCH_2), 7.03 (d, 4H, Ar-H, $^3J = 8.81$ Hz), 7.11 (4H, Ar-H, $^3J = 8.54$ Hz), 7.52 (d, 4H, Ar-H, $^3J = 8.54$ Hz), 7.64 (d, 4H, Ar-H, $^3J = 8.81$ Hz), 7.96 (s, 2H, H_5-triazole). ***^{13}C NMR** ($CDCl_3$)*:13.92, 22.57, 25.95, 29.22, 29.30, 29.38, 29.56, 29.97, 31.82, 68.58, 68.68, 115.31, 115.50, 119.58, 122.31, 127.91, 134.15, 136.18, 140.67, 157.62, 159.70. ESI-HRMS $[M+H]^+$: calculated for $C_{50}H_{67}N_6O_4$: 813.5062; found 813.5067.

4,4′-Bis (1-(4-dodecyloxyphenyl)-1H-[1,2,3]-triazole)oxymethylenebiphenyl (11c)

Yield: 75%, *^1H NMR ($CDCl_3$)*: 0.91 (t, 6H, CH_3, $^3J = 6.33$ Hz), 1.30 (m, 36H, $CH_3\underline{(CH_2)}_9$), 1.83 (m, 4H, $OCH_2\underline{CH_2}$), 4.03 (t, 4H, $O\underline{CH_2}CH_2$, $^3J = 6.9$ Hz), 5.35 (s, 4H, OCH_2), 7.01 (d, 4H, Ar-H, $^3J = 7.57$ Hz), 7.11 (d, 4H, Ar-H, $^3J = 7.80$ Hz), 7.49 (d, 4H, Ar-H, $^3J = 7.80$ Hz); 7.64 (d, 4H, Ar-H, $^3J = 7.57$); 7.96 (s, 2H, H_5-triazole). ***^{13}C NMR** ($CDCl_3$)*: 14.02, 22.67, 26.05, 29.24, 29.34, 29.38, 29.59, 29.64, 29.66, 29.70, 31.93, 62.48, 68.69, 115.33, 115.51, 119.07, 122.32, 125.32, 127.92, 134.18, 146.12, 157.63, 159.72. ESI-HRMS $[M+H]^+$: calculated for $C_{54}H_{73}N_6O_4$: 869.5688; found 869.5695.

4,4′-Bis (1-(4-tetradecyloxyphenyl)-1H-[1,2,3]-triazole)oxymethylenebiphenyl (11d)

Yield: 66%, *^1H NMR ($CDCl_3$)*: 0.91 (t, 6H, CH_3, $^3J = 6.33$ Hz), 1.31 (m, 44H, $CH_3\underline{(CH_2)_{11}}$), 1.84 (m, 4H, $OCH_2\underline{CH_2}$), 4.04 (t, 4H, $O\underline{CH_2}CH_2$, $^3J = 6.9$ Hz), 5.35 (s, 4H, OCH_2), 7.03 (d, 4H, Ar-H, $^3J = 8.93$ Hz), 7.10 (d, 4H, Ar-H, $^3J = 8.66$ Hz), 7.50 (d, 4H, Ar-H, $^3J = 8.66$ Hz), 7.61 (d, 4H, Ar-H, $^3J = 8.93$ Hz), 7.96 (s, 2H, H_5-triazole). ***^{13}C NMR** ($CDCl_3$)*: 13.98, 22.66, 26.07, 29.28, 29.35, 29.40, 29.58, 29.60, 29.58, 29.60, 29.67, 29.68, 31.95, 62.62, 68.79, 115.44, 115.61, 121.02, 122.36, 127.94, 130.59, 137.6, 149.00, 157.74, 159.79. ESI-HRMS $[M+H]^+$: calculated for $C_{58}H_{81}N_6O_4$: 925.6314; found 925.6329.

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