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Discotic liquid crystals. A brief review

S. Chandrasekhar[a]
[a] Centre for Liquid Crystal Research, Bangalore, India

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Plenary Lecture

Discotic liquid crystals

A brief review

by S. CHANDRASEKHAR
Centre for Liquid Crystal Research,
P.B. No. 1329, Jalahalli, Bangalore 560 013, India

Progress in the field of discotic liquid crystals is summarized, with emphasis on experimental results rather than theoretical developments. Examples are given of discotic mesogens (including metallo-mesogens) and discotic polymers, and the structures of the mesophases identified to date are described.

1. Introduction

Liquid crystals of disc-shaped molecules, or discotic liquid crystals as they are now called, were discovered just about 15 years ago. The first examples of this kind of mesomorphism were observed in the hexa-alkanoyloxybenzenes [1] and the hexa-alkoxy- and -alkanoyloxy-triphenylenes [2, 3], and it was established by X-ray studies [1, 4] that the mesophases of these compounds have a columnar structure. The subject has since grown rapidly; about 1000 discotic compounds are known to date and some 800 papers have been published on the chemistry and physics of these materials [5]. The aim of this lecture is to give a brief account of advances in the field, laying greater emphasis on structural aspects rather than their theoretical implications [6].

2. Some examples of discotic mesogens

Generally speaking, discotic mesogens have flat or nearly flat cores surrounded by six or eight (sometimes four) long chain substituents. There are, of course, exceptions, as we shall see presently. Some examples are presented in figure 1. Figure 1 (*f*) gives the structure of an oligomeric triphenylene derivative which exhibits a hexagonal columnar phase [10]. The molecule in figure 1 (*g*) deserves special mention as it is the first and, to date, only example of a discotic compound giving a ferroelectrically switchable columnar liquid crystal [11]. Columnar phases are also formed when the flat core of the molecule is replaced by a conical or pyramidal-shaped one [12, 13] (see figure 2 (*a*)), and, in rare cases, even when the central core is absent altogether, as in certain macrocyclic molecules [14] (see figure 2 (*b*)).

Several variants of the columnar structure have been identified (see figure 3 (*a*)). In some cases, the columns are liquid-like, i.e. the molecular centres are arranged aperiodically within each column, whilst in others they are arranged in a regular ordered fashion. The ordered columnar structures will be discussed in greater detail later.

Figure 3 (*b*) gives a schematic representation of the molecular order in the discotic nematic (N_D) phase. The director now denotes the preferred axis of orientation of the disc-normals (or molecular short axes). Hence the medium is optically negative, unlike

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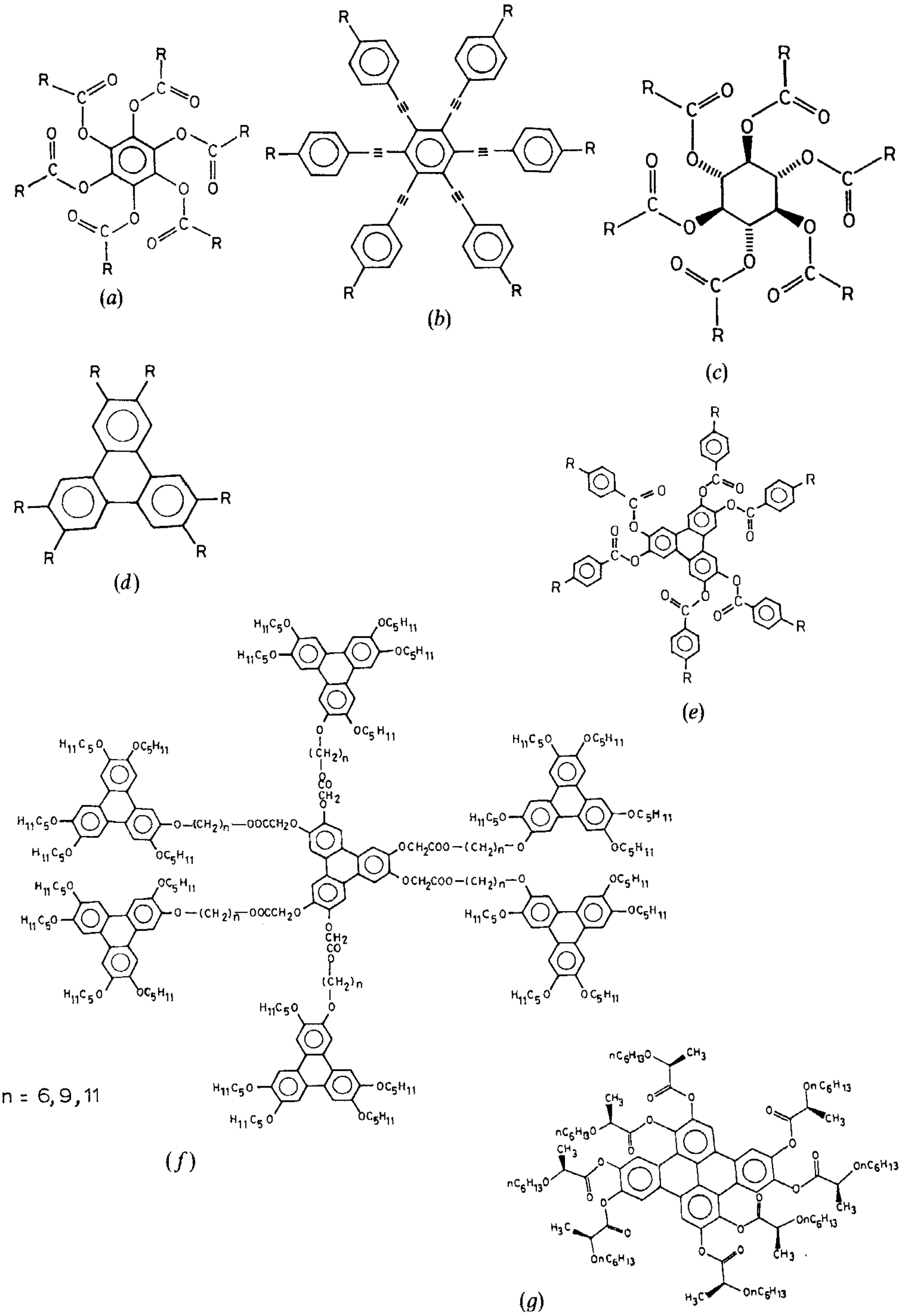


(a) (b) (c) (d) (e) (f) (g)

Figure 1. Examples of discotic mesogens: (*a*) hexa-*n*-alkanoyloxybenzenes [1]; (*b*) hexakis ((4-octylphenyl)ethynyl)benzene [7]; (*c*) hexa-*n*-alkanoates of *scyllo*-inositol [8]; (*d*) hexa-alkanoyloxy- and -alkoxy-triphenylenes [2, 3]; (*e*) hexa-*n*-alkyl- and -alkoxy-benzoates of triphenylene [9]; (*f*) star-like heptameric triphenylene derivatives [10]; (*g*) 1,2,5,6,8,9,12,13, -octa-(*S*-2-hexyloxypropanoyloxy)dibenzo-(e,l)-pyrene [11].

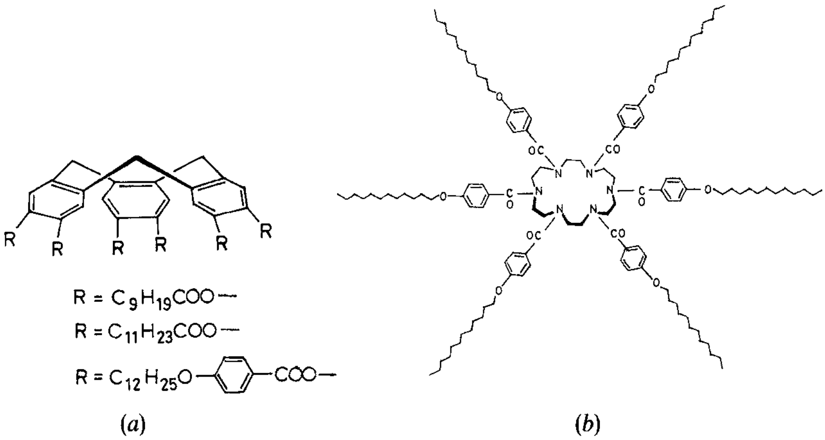


Figure 2. (*a*) Cyclotricatechylene hexa-esters [12]; (*b*) hexa(4-*n*-dodecyloxybenzoyl) derivatives of macrocyclic polyamines [14].

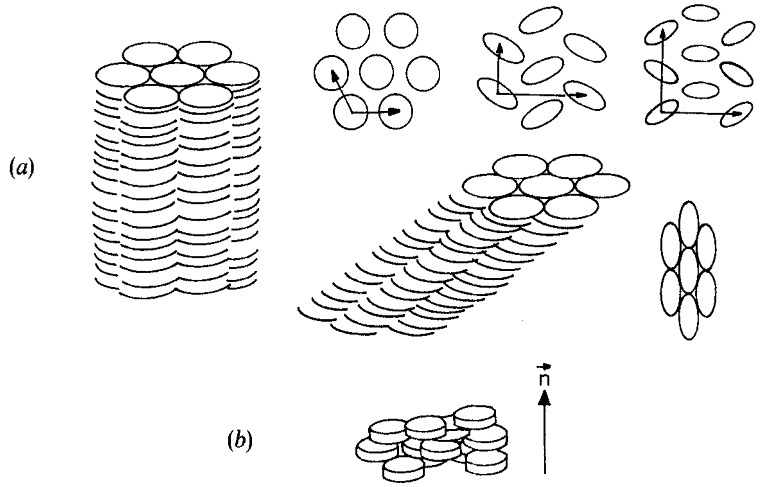


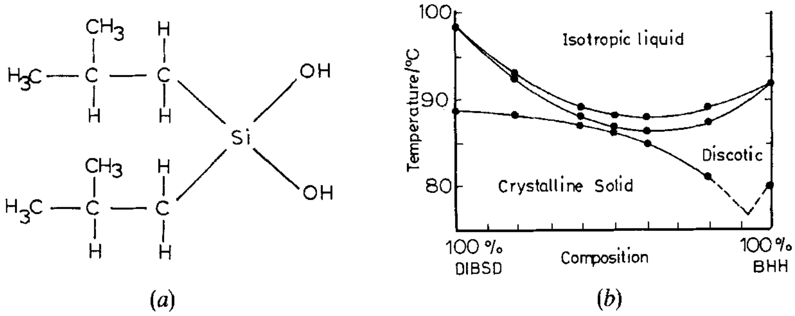
Figure 3. (*a*) Plan views of the two dimensional lattices of the columnar phases of discotic molecules. Ellipses denote discs that are tilted with respect to the basal plane [15]; (*b*) the discotic nematic phase.

the classical nematic which is optically positive. A twisted nematic (or cholesteric) phase with the helical axis normal to the director has also been identified [16, 17].

A smectic-like lamellar discotic phase has been reported [18–20]. It has been suggested [19] that this phase has a tilted smectic C type of structure, but the disposition of the molecules in each layer does not appear to have been resolved unequivocally.

3. Early work on discotics

The hexa-*n*-alkanoates of benzene were first synthesized as early as 1937 by Backer and Van der Baan [21] at the University of Groningen, but the authors did not report the mesogenic character of these compounds. However, de Jeu [22], who works in



(*a*) (*b*)

Figure 4. (*a*) The molecular structure of diisobutylsilanediol (DIBSD). (*b*) The miscibility diagram of DIBSD with benzene hexa-*n*-heptanoate (BHH) [24].

Groningen, found to his pleasant surprise that the materials were still in stock in the Department of Organic Chemistry, and sure enough they did show the expected mesomorphic properties.

Another interesting case is that of di-isobutyl silane diol [DIBSD; see figure 4(*a*)]. In the 1950s Eaborn and Hartshorne [23] observed that the compound showed a mesophase which they were unable to classify as belonging to any of the then known liquid crystal types. Much later, Bunning, Goodby, Gray and Lydon [24(a)] carried out miscibility studies of this compound with benzene hexa-*n*-heptanoate (BHH) and proved conclusively that the mesophase of DIBSD is a columnar liquid crystal (see figure 4(*b*). They have suggested that the molecule forms a dimer [24(a)] or a polymer [24(b)] which favours the occurrence of a columnar phase.

4. Discotic metallomesogens

Of late, metal containing liquid crystals are attracting a great deal of attention mainly because of their unusual electrical and magnetic properties. The first discotic metallo-mesogens were reported by Giroud-Godquin and Billard [18], and since then a variety of metal complexes have been synthesized which exhibit columnar or lamellar mesophases [25]. A few examples are given in figure 5. X-ray determinations of the crystal structures have been useful in elucidating the structures of the complexes and the mesophases formed by them [31–37].

Available electrical conductivity (σ) data on the columnar phases of metallo-mesogens indicate that they are molecular semiconductors [38, 39]. Figure 6 presents experimental plots of $\ln \sigma$ versus $1/T$ for copper phthalocyanines. Calculations indicate that the hopping probability for the electron is several orders of magnitude greater along the columnar axis than transverse to it, as is to be expected [39].

Eastman *et al.* [40] have carried out ESR studies on single crystals of the copper complex in figure 5(*a*). It is known from X-ray studies that the discs are arranged in columns in the crystalline state of this complex [34]. From an analysis of the ESR data, Eastman *et al.* have drawn the important conclusion that the spectra have the features associated with a spin $\frac{1}{2}$ one dimensional Heisenberg antiferromagnet, and that the exchange interactions are quite significant (i.e. an appreciable degree of antiferromagnetic long range order persists) even in the discotic phase.

5. High resolution X-ray studies

High resolution X-ray studies have been conducted on the columnar phases of a few compounds. The measurements were made on very well-oriented monodomain

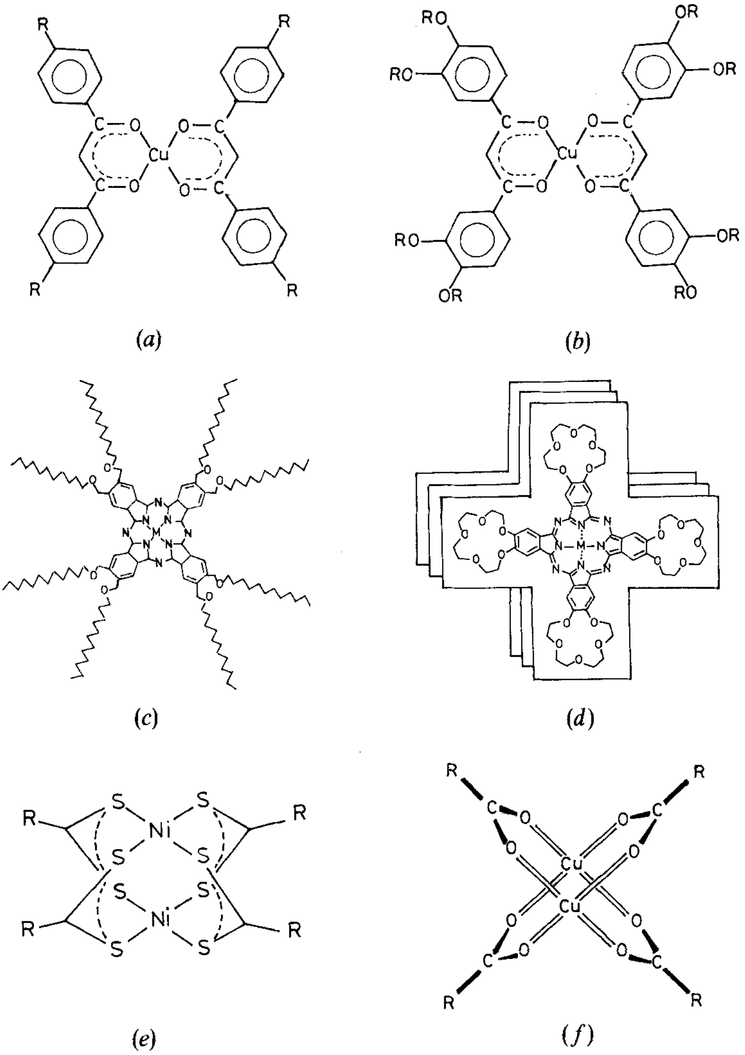


Figure 5. Metallo-mesogens: (*a*) bis-(4-*n*-decylbenzoyl)methanato copper(II) [18]; (*b*) bis-(3,4-di-nonyloxybenzoyl) methanato copper(II) [26]; (*c*) octa-substituted metallo-phthalocyanine [27]; (*d*) benzo-15-crown-5-substituted phthalocyanine [28]; (*e*) tetrakis(alkyldithiolato) dinickel(II) [29]; (*f*) binuclear copper carboxylate [30].

samples obtained by preparing freely suspended liquid crystal strands, typically about 200 μm in diameter and 1·5–2 mm long, with the column axis parallel to the axis of the strand [41–45]. The salient results of these studies are summarized below.

(i) $(C_{13}H_{27}COO)_6$-truxene [45]

The correlation length of the two dimensional lattice of the columnar phase of this compound is greater than 4000 Å, the lower limit being set by the instrumental resolution. On the other hand, within each column the flat molecular cores form an orientationally ordered one dimensional liquid, whilst the hydrocarbon chains are in a highly disordered state. These results are in conformity with those of deuterium NMR spectroscopy [46].

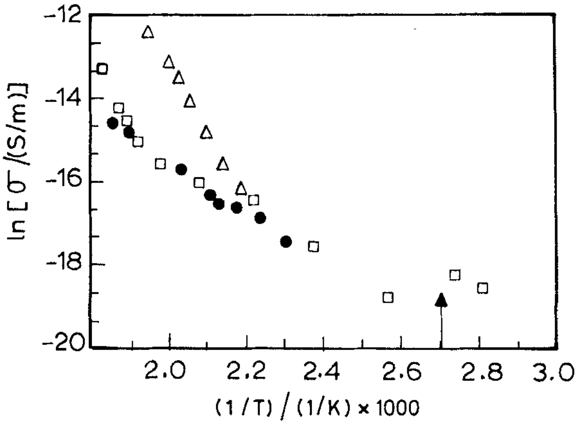


Figure 6. Arrhenius plots of the electrical conductivity (σ) for copper phthalocyanines: △ represents data points for $R = H$, ● for $R = OC_8H_{17}$ and ☐ for $R = OC_{12}H_{25}$. A slight increase in the conductivity is observed from the last compound at the transition from the discotic mesophase to the crystalline phase (indicated by the arrow) [38].

The structure of this columnar phase is essentially the same as that proposed originally for the benzene compounds [1]. The stability of such a structure (a three dimensional body in which the density is a periodic function of two dimensions only) was discussed many years ago by Landau, who concluded as follows: 'Thus, bodies having such a structure could in theory exist, but it is not known whether they do in fact exist in Nature' [47].

(ii) $(C_{12}H_{25}COO)_6$-triphenylene [42, 43]

This compound exhibits a transition from a hexagonal (D_h) to a rectangular (D_r) columnar phase. The transition, which is weakly first order, is associated with a small distortion of the lattice, consistent with a herringbone arrangement in the rectangular structure, with only the core of the molecule tilted with respect to the column axis (see figure 7). However, high resolution synchrotron X-ray studies on monodomain discotic strands have established that the tilt of the molecular core persists in the D_h phase as well, except that the tilts in neighbouring columns are rotationally uncorrelated, i.e. they are free to assume different azimuthal angles. Thus the D_h–D_r transition may be looked upon as an orientational order–disorder transition.

(iii) Hexa-hexylthiotriphenylene [44]

This compound shows a transition from a hexagonal ordered phase to a hexagonal disordered one. As mentioned earlier, the former is a phase in which there is regularity in the stacking of the triphenylene cores in each column, and the latter is one in which the column is liquid-like. X-ray studies reveal that in the ordered phase there is a helicoidal stacking of the cores within each column, the helical spacing being incommensurate with the intermolecular spacing [49]. In addition, a three column superlattice develops as a result of the frustration caused by molecular interdigitation in triangular symmetry. Ideally, if there is no intercolumn interaction, true long range order cannot exist within a column because of the Peierls–Landau instability. The existence of a regular periodicity in the stacking in each column therefore implies that neighbouring columns must be in register. Thus ordered columnar phases can

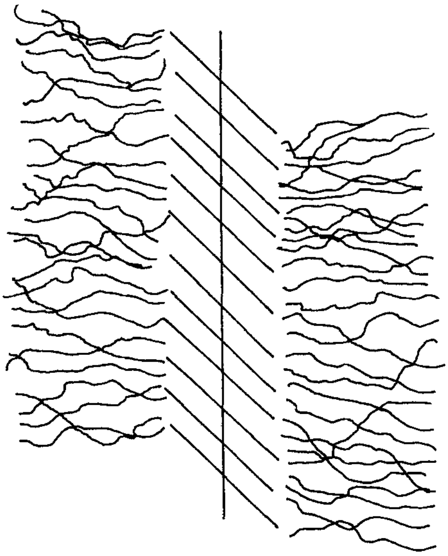


Figure 7. Schematic representation of the structure of a column in which the cores are tilted with respect to the column axis and the chains are in a highly disordered state [48].

probably be compared with highly ordered smectic phases of rod-like molecules, for example smectics B, E, G, H, etc., which possess three dimensional positional order. However, further high resolution studies are necessary before general conclusions can be drawn.

6. Biaxial nematics

Molecules that combine the features of the rod and the disc would appear to favour the formation of a biaxial nematic [50, 51]. Two examples are presented in figure 8. Other cases are discussed in an article by Praefcke *et al.* [51].

7. Discotic polymer liquid crystals

Finally we turn our attention briefly to a new class of liquid crystal polymers, viz. discotic polymers [54–60]. The basic monomer units are disc-shaped mesogenic moieties which are components of the polymer main chain itself or are attached to the polymer backbone as side groups (see figure 9). The types of mesophases that have so far been identified are illustrated in figure 10. A polyester with triphenylene as the repeating unit in the main chain separated by flexible spacers [55], or with triphenylene units attached as side groups to the polymer chain via flexible spacers [59] forms a hexagonal columnar mesophase (see figure 10 (*a*)). On the other hand, rigid aromatic polyamides and polyesters with disc-shaped units in the main chain form a sanidic (or board-like) nematic [57], with the boards stacked parallel to one another (see figure 10 (*b*)). The addition of electron acceptor molecules to discotic polymers results in the formation of charge transfer complexes which stabilize (or in certain, non-mesomorphic materials, induce) mesophases [60]. A new type of induced mesophase in such a system is the columnar nematic (see figure 10 (*c*)).

X-ray diffraction studies of the macroscopic alignment of the columnar structures produced by stretching a film or by drawing fibres (about 1 m long and several microns

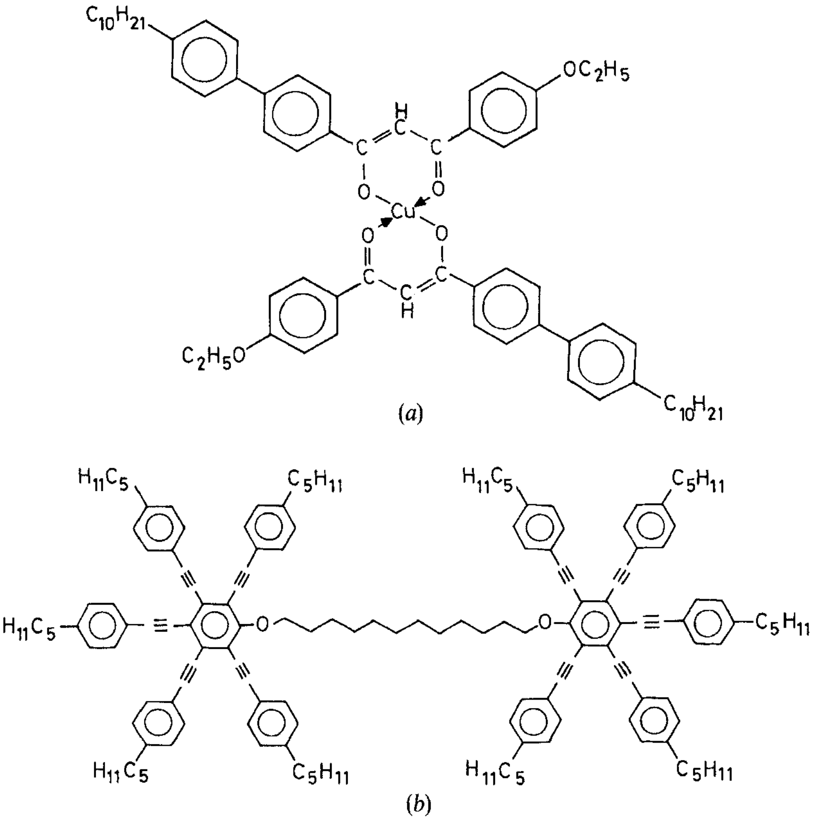


Figure 8. Molecules which exhibit the biaxial nematic phase: (*a*) (bis-(4-*n*-decylbiphenyl)-3-(4-ethoxyphenyl)propane-1,3-dionato copper(II) [52]; (*b*) bis-1,12-{[pentakis(4-pentylphenylethynyl)]phenoxy}dodecane [53].

thick) from the polymer melt have been reported [55, 59]. Hüser, Pakula and Spiess [59] have demonstrated that almost perfect alignment can be achieved by proper mechanical and thermal treatment. Interestingly, in the main chain polymer, the columns are oriented perpendicular to the stretch direction (or the chain direction) whereas in the side group polymer they are oriented parallel to it (see figure 11). A detailed study of the mechanical and other physical properties of these oriented polymers is evidently of the greatest interest.

8. Concluding remarks

I end this article by making a few remarks about terminology. The word discotic was introduced by Billard *et al.* [2] to distinguish disc-shaped mesogens from the familiar rod-shaped or calamitic mesogens. Soon, however, terms like discotic liquid crystals, discotic phases, discogens, etc., began to be used extensively in the literature, and as observed by the present writer at a Royal Society Discussion Meeting in 1982 [6 (b)], the word discotic came to be applied (rather loosely) to describe the molecules as well as the mesophases formed by them.

Strictly speaking, it is the molecules that are discotic and not the mesophases, which may be columnar, nematic or lamellar. Moreover, as we have seen, certain molecules which are themselves not disc-shaped may form columnar phases. A striking example is

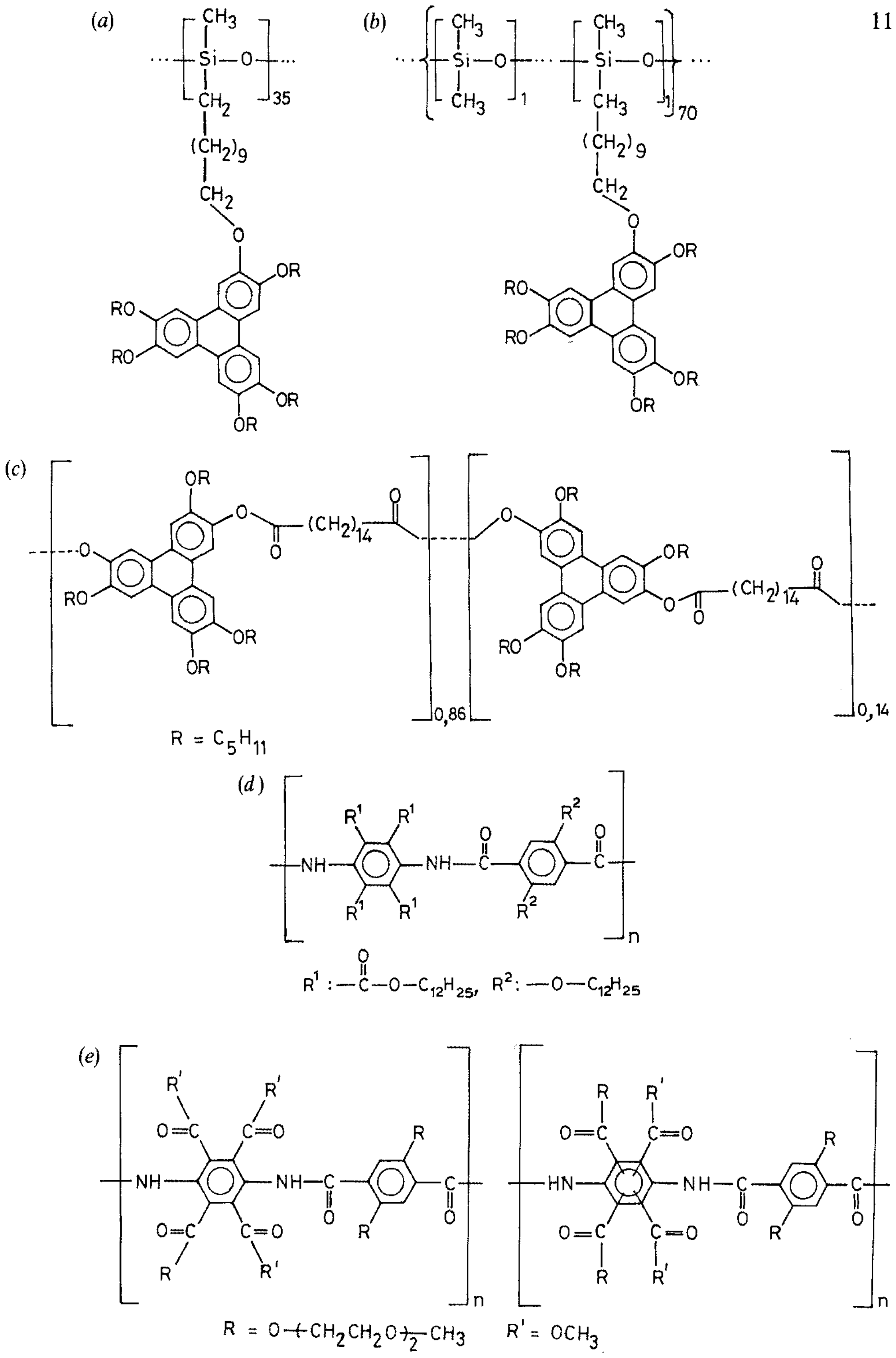


Figure 9. Discotic polymers: (*a*) annealed sample, g −19°C D 39°C I [54]; (*b*) annealed sample, g −29°C D 36°C I [54]; (*c*) 60°C D 150°C I [55]; (*d*) D_1 67°C D_2 130°C I [56]; (*e*) C 179°C Nσ 262°C I [57]; (*f*) C 131°C Nσ 216°C I [57]; g = glass, C = crystal, D = discotic columnar, Nσ = sanidic nematic, I = isotropic.

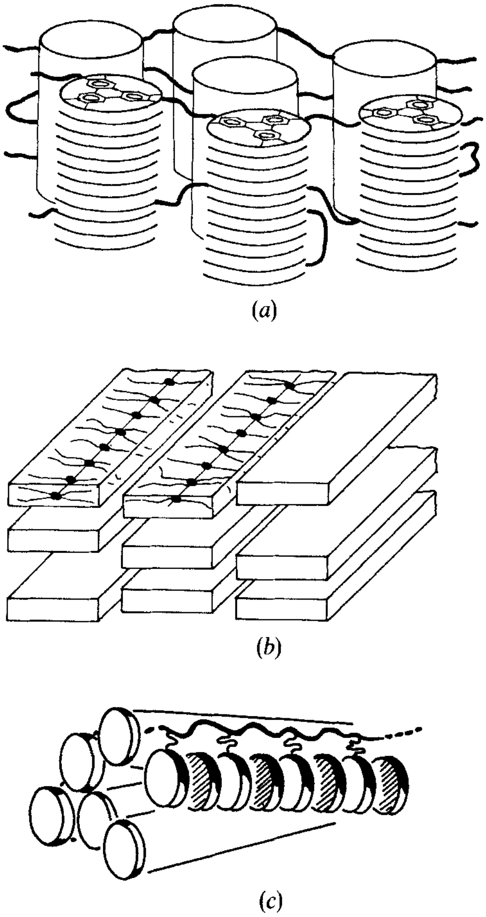


Figure 10. Mesophases of discotic polymers: (*a*) the hexagonal columnar phase [55, 59]; (*b*) the sanidic nematic phase (Nσ) [57]; (*c*) the columnar nematic phase [60].

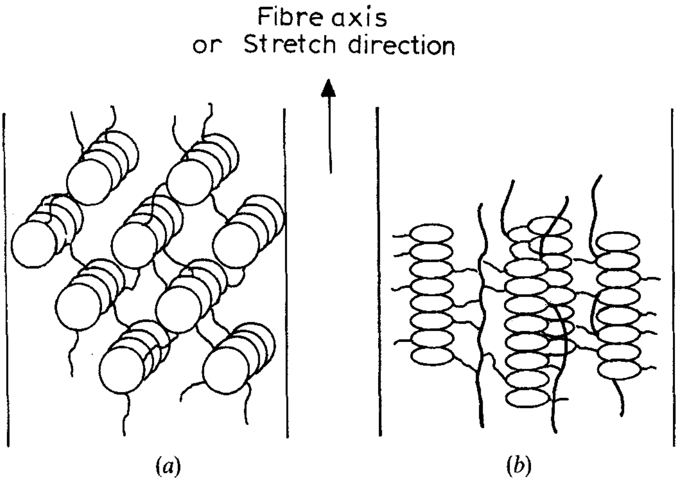


Figure 11. Orientation of the molecules in stretched samples: (*a*) main chain discotic polymer; (*b*) side group discotic polymer [59].

the so-called phasmid [61] which is more like a rod then a disc, but exhibits columnar mesomorphism. Now that the subject is expanding rapidly, it would be useful to redefine the terminology precisely and unambiguously. I have not, however, attempted to do so in the present paper.

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