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(71) Applicant: **THE UNIVERSITY OF BIRMINGHAM**
 [GB/GB]; Edgbaston, Birmingham B15 2TT (GB).

(72) Inventors: **BARANOFF, Etienne David**; 15 Harborne
 Park Road, Birmingham B17 0DE (GB). **HERBAUT,**
Antoine Joel Maurice; 100, route de Don, 59112 An-
 noeuillin (FR).

(74) Agent: **BAILEY, Jennifer**; Marks & Clerk LLP, Alpha
 Tower, Suffolk Street Queensway, Birmingham B1 1TT
 (GB).

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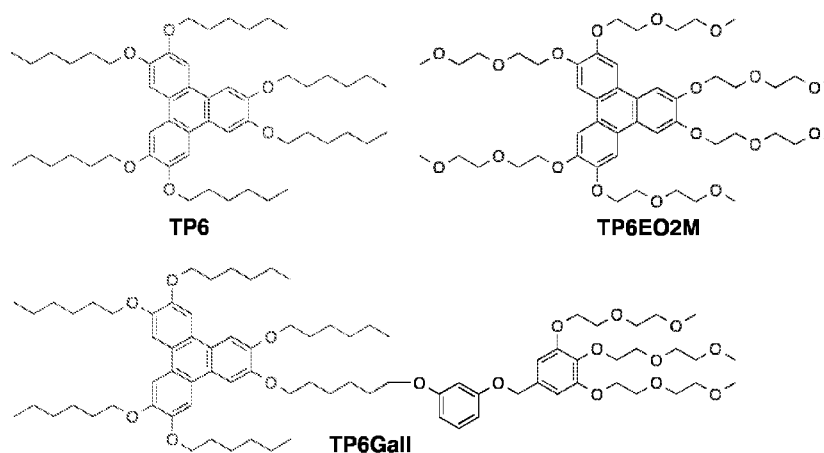
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(54) Title: COMPOSITION

Figure 1



(57) Abstract: Provided is a composition which comprises two immiscible compounds and a compatibiliser, which stabilises the interface between two or more immiscible compounds. The compatibiliser improves the morphology of blends comprising the first and second compounds. The composition may be useful in the context of functional blends for optoelectronic applications, such as solar cells, organic light-emitting diodes (OLEDs) and organic photovoltaics (OPVs). Also provided is a process for the preparation of the composition and compounds for use therein.

Composition

5 The present invention relates to a composition comprising two immiscible compounds and a compatibiliser, a process for the preparation of the composition and compounds for use therein.

Mixing two materials is a successful effective strategy to create new materials that combine the properties of the two components and possibly display additional qualities. However mixing two immiscible compounds generally results in macrophase
10 separation into two thermodynamically different phases leading to poor materials.

Block copolymers have been widely used to compatibilize two immiscible polymers. The approach has also been utilised to control the formation of nanostructures in the bulk of the mixture.
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The phase separation process of blends of immiscible small molecules has been controlled by addition of additives such as octane diiodooctane or by controlling the deposition conditions (solvent and temperature). However additives are non-functional materials and their concentration has to be kept low. Furthermore residuals have a
20 negative impact on the chemical stability of the blend and the phase separation state is only kinetically stable leading to macrophase separation on longer time scales.

Loo et al. have used thiophene-C60 dyads to stabilise mixtures of P3HT (polymer) and PCBM resulting in devices with improve robustness. Systems using only small
25 molecules would be advantageous over polymers because of their well define structure and possibility of extensive purification and reproducibility of synthesised materials.

Date and Bruce have prepared a rod-disk shape amphiphile to suppress the macrophase separation in a blend of rod and disk-like small-molecule mesogens (R. W. Date and D. W. Bruce, J. Am. Chem. Soc., 2003, 125, 9012–9013). A homogenous
30 nematic phase was obtained over a large range of amphiphile concentration demonstrating the compatibilisation of the rod and disklike mesogens. Kouwer and Mehl also developed a series of mesogenic rod-disk shape amphiphiles (P. H. J. Kouwer and G. H. Mehl, J. Mater. Chem., 2009, 19, 1564–1575). Mixing the
35 amphiphile with the rod-like mesogen shows a homogeneous isotropic phase over a

wide range of temperature while mixing with the disklike mesogen shows one monotropic nematic phase. No mixing studies were carried out on the mixture of rods, disks, and amphiphiles.

- 5 It is an object of the present invention to control the morphology of blends of immiscible compounds.

According to a first aspect of the present invention there is provided a composition comprising

- 10 a first compound represented by the formula:

core $W(A)_m$;

a second compound represented by the formula:

core $X(B)_n$; and

a compatibiliser compound represented by the formula:

- 15 $[\text{core } Y(A)_p] - L - [\text{core } Z(B)_q]$;

wherein

each of core W, core X, core Y and core Z comprises an aromatic or a heteroaromatic ring or ring system;

A is an addend of core W and core Y and B is an addend of core X and core Z;

- 20 each of k, m, n, p and q is independently selected from an integer from 1 to 10;

L is a linker covalently linking core Y and core Z; and

wherein the first and second compounds are immiscible.

In some embodiments, k is 1. In other words, the compatibiliser may comprise a single moiety of core Y and its addends A, linked by a linker L to a single moiety of core Z and its addends B. In such embodiments, the composition comprises:

- 25 a first compound represented by the formula:

core $W(A)_m$;

a second compound represented by the formula:

- 30 core $X(B)_n$; and

a compatibiliser compound represented by the formula:

$[\text{core } Y(A)_p] - L - [\text{core } Z(B)_q]$;

wherein

each of core W, core X, core Y and core Z comprises an aromatic or a heteroaromatic ring or ring system;

35

A is an addend of core W and core Y and B is an addend of core X and core Z;
each of k, m, n, p and q is independently selected from an integer from 1 to 10;
L is a linker covalently linking core Y and core Z; and
wherein the first and second compounds are immiscible.

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In some other embodiments, the compatibiliser contains multiple moieties of core Y (with its addend(s) A), each moiety of core Y being covalently linked to core Z via a linker L. In these embodiments, k is from 2 to 10. In some embodiments, k is no greater than 8, no greater than 6 or no greater than 4.

10

By the term "immiscible", as used herein, it will be understood that the first and second compounds do not form a single phase in the absence of further components such as the compatibiliser. Whether two compounds have mixed to form a single phase can be determined using simple optical checks known to the skilled person. The degree of mixing of two compounds can be quantified using known spectroscopic techniques.

15

A "compatibiliser", as used herein, is a compound which stabilises the interface between two or more immiscible compounds. The inventors have developed the compatibiliser to improve the morphology of blends comprising the first and second compounds. The composition may be useful in the context of functional blends for optoelectronic applications, such as solar cells, organic light-emitting diodes (OLEDs) and organic photovoltaics (OPVs). Thus, the invention further provides a solar cell, an OLED or an OPV comprising the composition.

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In some embodiments the first and second compounds melt to form a single isotropic phase. The advantage of a single isotropic melt is that no macrophase separation is likely to occur over time (unless one or both compounds degrade). If multiple phases are present in the melt, macrophase separation can occur over time or heating and cooling. It will be appreciated that formation of a single isotropic phase occurs in the presence of the compatibiliser. Thus, in some embodiments the mixture of the first compound, the second compound and the compatibiliser in the composition form a single isotropic phase.

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In some embodiments, the first compound is hydrophobic and the second compound is hydrophilic. A hydrophilic compound is typically miscible in water.

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In some embodiments, the immiscibility of the first and second compounds is conferred by the addends A and B. By this it will be understood that addends A and B are designed to be incompatible. This is important for controlling the organization of the compounds in the bulk mixture. If the first and second compounds are too compatible (i.e. if they are completely miscible), they create a well-mixed blend which is undesirable. On the other hand, if the first and second compounds are immiscible, macrophase separation occurs. It is desired to achieve a balance between these two states in order to optimize charge transport and the size of the active surface. The composition of the present invention provides this balance by creating immiscibility between the first and second compounds through the provision of incompatible chains, and then controlling that immiscibility through the use of a compatibiliser molecule.

It will be understood that "hydrophobic" and "hydrophilic" are relative terms. Hence the addend A may be more hydrophobic than the addend B and the addend B may be more hydrophilic than the addend A.

Thus, in some embodiments, addends A and B belong to different chemical families. For example, A may be an alkyl chain while B may be an alkoxy or amine group, but A and B will not both be alkyl chains.

The aromatic cores are useful for providing photoactivity in the visible part of the electromagnetic spectrum. In some embodiments each of core W, core X, core Y and core Z is a mesogen. IUPAC defines a "mesogen" as a compound that under suitable conditions of temperature, pressure, and concentration can exist as a mesophase or, in particular, as a liquid-crystal (LC) phase. The compatibiliser may be a mesogenic amphiphile.

In some embodiments one or more of cores W, X, Y and Z are discotic (i.e. disc-shaped). In some embodiments, each of cores W, X, Y and Z is discotic.

The proportion of the compatibiliser can be described relative to the first compound or the second compound as:

compatibiliser (mol) / compatibiliser + first compound (mol); or

compatibiliser (mol) / compatibiliser + second compound (mol).

In one series of embodiments the proportion of compatibiliser relative to the total of compatibiliser + first compound is from 1 to 99%, from 3 to 70%, from 5 to 50% or from 10 to 30 mol%.

5

In one series of embodiments the proportion of compatibiliser relative to the total of compatibiliser + second compound is from 1 to 99%, from 3 to 70%, from 5 to 50% or from 10 to 30 mol%.

10

In one series of embodiments the proportion of compatibiliser relative to the total of compatibiliser + first compound + second compound is from 1 to 99%, from 3 to 70%, from 5 to 50% or from 10 to 30 mol%.

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In one series of embodiments the ratio of the first compound (mol%) to the second compound (mol%) is from 90 : 10 to 10 : 90, from 75 : 25 to 25 : 75, from 60 : 40 to 40 : 60 or approximately 50:50 (equimolar).

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In one series of embodiments the compatibiliser constitutes less than 60, 50, 40, 30, 20, 10 or 5 mol% of the composition. In one series of embodiments the compatibiliser constitutes at least 1, 2, 5, 8, 10, 15, 20 or 30 mol% of the composition. In one embodiment the compatibiliser constitutes from 1 to 60, from 5 to 50, from 20 to 40, from 1 to 10 or from 5 to 8 mol% of the composition.

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In one series of embodiments the first compound constitutes at least 20, 30, 40 or 50 mol% of the composition. In one series of embodiments the first compound constitutes less than 80, 70, 60 or 50 mol% of the composition.

30

In one series of embodiments the second compound constitutes at least 20, 30, 40 or 50 mol% of the composition. In one series of embodiments the second compound constitutes less than 80, 70, 60 or 50 mol% of the composition.

In one embodiment the composition consists of the first compound, the second compound and the compatibiliser.

Examples of compositions in accordance with the invention are listed in the table below:

First compound (mol%)	Second compound (mol%)	Compatibiliser (mol%)
42 to 49	42 to 49	2 to 16
45 to 49	45 to 49	2 to 10
42 to 45	42 to 45	10 to 16
20 to 42	20 to 42	16 to 60
38 to 42	38 to 42	16 to 24
20 to 30	20 to 30	40 to 60

- 5 In one embodiment A is a straight-chain or branched or cyclic alkyl group having from 1 to 50, from 2 to 20 or from 3 to 10 carbon atoms, in which one or more adjacent or non-adjacent hydrocarbon groups may be replaced by -O-, -S-, -CR¹R²-, -S(=O)-, -S(=O)₂-, -SiR¹R²-, -GeR¹R²-, -NR¹-, -BR¹-, -PR¹-, -P(=O)R¹-, -P(=O)OR¹-, -C(=O)-, -C(=S)-, -C(=R¹R²)-, -CR¹=CR²-, -C≡C-, -C(=O)O-, -OC(=O)-, -C(=NR¹)-, -C=NR¹-, -NR¹C(=O)-, -C(=O)NR¹-, -NR¹C(=S)- or -C(=S)NR¹-, and in each of which one or more hydrogen atoms may be replaced by F, -Cl, -Br, -I, -NO₂, -CN, -OH, -C(=O)OR¹, -OC(=O)R¹, a straight-chain or branched or cyclic alkyl, alkoxy, amine, phosphine, phosphite, phosphonite, silane, germane, borane, borate, boronate, sulfane, sulfinyl, sulfonyl group, an aryl, heteroaryl, alkanyl, alkenyl, alkynyl group which may be substituted by one or more non aromatic radicals.

- 20 In one such embodiment A is a straight-chain or branched or cyclic alkyl group having from 1 to 50, from 2 to 20 or from 3 to 10 carbon atoms, in which no more than one hydrocarbon group is replaced by -O-, -S-, -CR¹R²-, -S(=O)-, -S(=O)₂-, -SiR¹R²-, -GeR¹R²-, -NR¹-, -BR¹-, -PR¹-, -P(=O)R¹-, -P(=O)OR¹-, -C(=O)-, -C(=S)-, -C(=R¹R²)-, -CR¹=CR²-, -C≡C-, -C(=O)O-, -OC(=O)-, -C(=NR¹)-, -C=NR¹-, -NR¹C(=O)-, -C(=O)NR¹-, -NR¹C(=S)- or -C(=S)NR¹.

- 25 In one embodiment A is a straight-chain or branched alkyl group having from 1 to 25 carbon atoms, in which at least one hydrocarbon group is replaced by -O-, S-, -CR¹R²-, -S(=O)-, -S(=O)₂-, -SiR¹R²-, in each of which one or more hydrogen atoms may be replaced by F, -Cl, -Br, -OH, -C(=O)OR¹, -OC(=O)R¹, or a straight-chain or branched alkyl, alkoxy, silane or sulfonyl group.

- 30 In one embodiment A is an alkyl or alkoxy group having from 1 to 20 carbon atoms. In one such embodiment A is -O(CH₂)_aCH₃ where a is from 3 to 10 such as OC₆H₁₃.

In one embodiment B is a straight-chain or branched or cyclic alkyl group having from 1 to 50, from 2 to 20 or from 3 to 10 carbon atoms, in which one or more adjacent or non-adjacent hydrocarbon groups may be replaced by -O-, -S-, -CR¹R²-, -S(=O)-, -S(=O)₂-,
 5 -SiR¹R²-, -GeR¹R²-, -NR¹-, -BR¹-, -PR₁-, -P(=O)R₁-, -P(=O)OR₁-, -C(=O)-, -C(=S)-, -C(=R¹R²)-, -CR¹=CR²-, -C≡C-, -C(=O)O-, -OC(=O)-, -C(=NR¹)-, -C=NR¹-, -NR¹C(=O)-, -C(=O)NR¹-, -NR¹C(=S)- or -C(=S)NR¹-, and in each of which one or more hydrogen atoms may be replaced by F, -Cl, -Br, -I, -NO₂, -CN, -OH, -C(=O)OR¹, -OC(=O)R¹, a
 10 straight-chain or branched or cyclic alkyl, alkoxy, amine, phosphine, phosphite, phosphonite, silane, germane, borane, borate, boronate, sulfane, sulfinyl, sulfonyl group, an aryl, heteroaryl, alkanyl, alkenyl, alkynyl group which may be substituted by one or more non aromatic radicals.

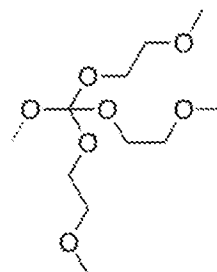
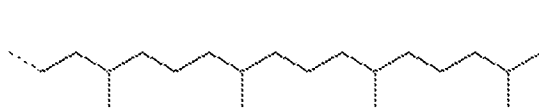
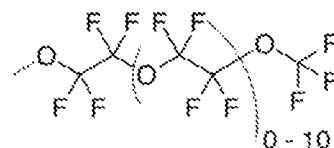
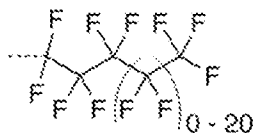
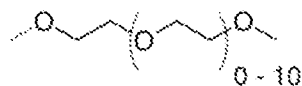
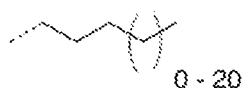
In one embodiment B is a straight-chain or branched or cyclic alkyl group having from 1 to 50, from 2 to 20 or from 3 to 10 carbon atoms, in which at least two adjacent or non-adjacent hydrocarbon groups are replaced by -O-, -S-, -CR¹R²-, -S(=O)-, -S(=O)₂-, -SiR¹R²-, -GeR¹R²-, -NR¹-, -BR¹-, -PR¹-, -P(=O)R¹-, -P(=O)OR¹-, -C(=O)-, -C(=S)-, -C(=R¹R²)-, -CR¹=CR²-, -C≡C-, -C(=O)O-, -OC(=O)-, -C(=NR¹)-, -C=NR¹-, -NR¹C(=O)-, -C(=O)NR¹-, -NR¹C(=S)- or -C(=S)NR¹-, and in each of which one or more hydrogen
 15 atoms may be replaced by F, -Cl, -Br, -I, -NO₂, -CN, -OH, -C(=O)OR¹, -OC(=O)R¹, a straight-chain or branched or cyclic alkyl, alkoxy, amine, phosphine, phosphite, phosphonite, silane, germane, borane, borate, boronate, sulfane, sulfinyl, sulfonyl group, an aryl, heteroaryl, alkanyl, alkenyl, alkynyl group which may be substituted by one or more non aromatic radicals.

25 In one embodiment B is a straight-chain or branched alkyl group having from 1 to 25 carbon atoms, in which at least one hydrocarbon group is replaced by -O-, S-, -CR¹R²-, -S(=O)-, -S(=O)₂-, -SiR¹R²-, in each of which one or more hydrogen atoms may be replaced by F, -Cl, -Br, -OH, -C(=O)OR¹, -OC(=O)R¹, or a straight-chain or branched
 30 alkyl, alkoxy, silane or sulfonyl group.

In one embodiment B is OCH₂CH₂(OCH₂CH₂)_bOCH₃ where b is from 0 to 10 such as OCH₂CH₂OCH₂CH₂OCH₃.

In some embodiments A and/or B is an unsubstituted hydrocarbon chain of from 5 to 20 or from 10 to 15 carbon atoms. For example, A and/or B may be C₁₂H₂₅.

In one embodiment one or both of A and B is independently selected from:

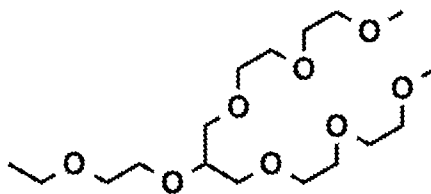


5

wherein x and v are each an integer independently selected from 1 to 10 or from 2 to 5.

In some embodiments A or B is 10-(2-ethoxyethoxy)-2,5,8,12,15,18-hexaoxonadecane, which has, and/or comprises, the following structure:

10



15

In addition to the addends A and B, each of core W, core X, core Y and core Z may be independently and optionally substituted by a further substituent R which is different from A and B.

20

In one embodiment R is -F, -Cl, -Br, -I, -NO₂, -CN, -OH, a straight-chain or branched or cyclic alkyl group having from 1 to 50, from 2 to 20 or from 3 to 10 carbon atoms, in which one or more adjacent or non-adjacent hydrocarbon groups may be replaced by -O-, -S-, -CR¹R²-, -S(=O)-, -S(=O)₂-, -SiR¹R²-, -GeR¹R²-, -NR¹-, -BR¹-, -PR₁-, -P(=O)R₁-, -P(=O)OR₁-, -C(=O)-, -C(=S)-, -C(=R¹R²)-, -CR¹=CR²-, -C≡C-, -C(=O)O-, -OC(=O)-, -C(=NR¹)-, -C=NR¹-, -NR¹C(=O)-, -C(=O)NR¹-, -NR¹C(=S)- or -C(=S)NR¹-, and in each

of which one or more hydrogen atoms may be replaced by F, -Cl, -Br, -I, -NO₂, -CN, -OH, -C(=O)OR¹, -OC(=O)R¹, a straight-chain or branched or cyclic alkyl, alkoxy, amine, phosphine, phosphite, phosphonite, silane, germane, borane, borate, boronate, sulfane, sulfinyl, sulfonyl group, an aryl, heteroaryl, alkanyl, alkenyl, alkynyl group
 5 which may be substituted by one or more non aromatic radicals wherein a plurality of R, either on the same ring or on two different rings, may in turn together form a mono- or polycyclic ring, optionally aromatic, optionally containing one or more heteroatoms.

10 In one embodiment R is -F, -Cl, -Br, -I, -NO₂, -CN, -OH, a straight-chain or branched group having from 1 to 25 carbon atoms, in which at least one hydrocarbon group is replaced by -O-, S-, -CR¹R²-, -S(=O)-, -S(=O)₂-, -SiR¹R²-, in each of which one or more hydrogen atoms may be replaced by F, -Cl, -Br, -OH, -C(=O)OR¹, -OC(=O)R¹, a straight-chain or branched alkyl, alkoxy, silane or sulfonyl group.

15 R¹ and R² are the same or different at each occurrence and are independently selected from hydrogen, halo, alkyl, alkenyl, alkynyl, heteroalkyl, aryl, heteroaryl. In one embodiment R¹ is H and/or R² is H.

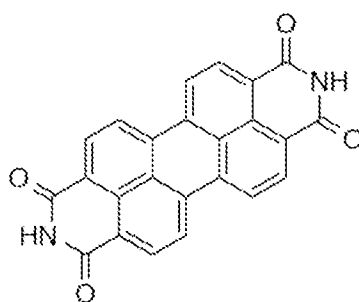
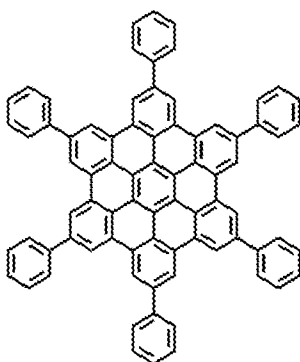
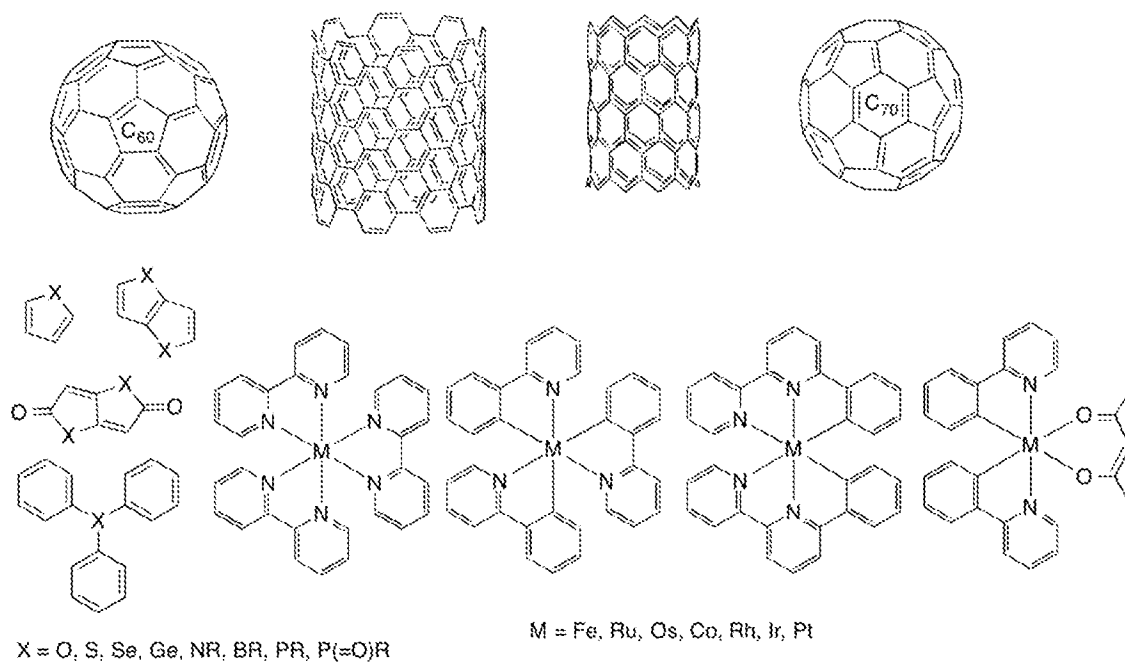
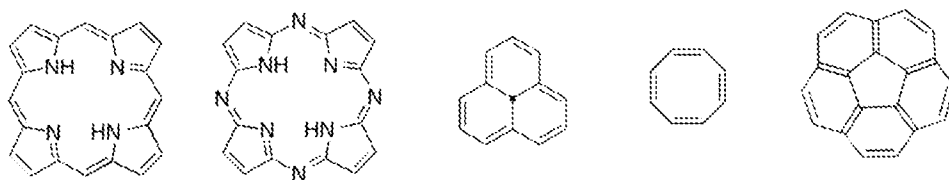
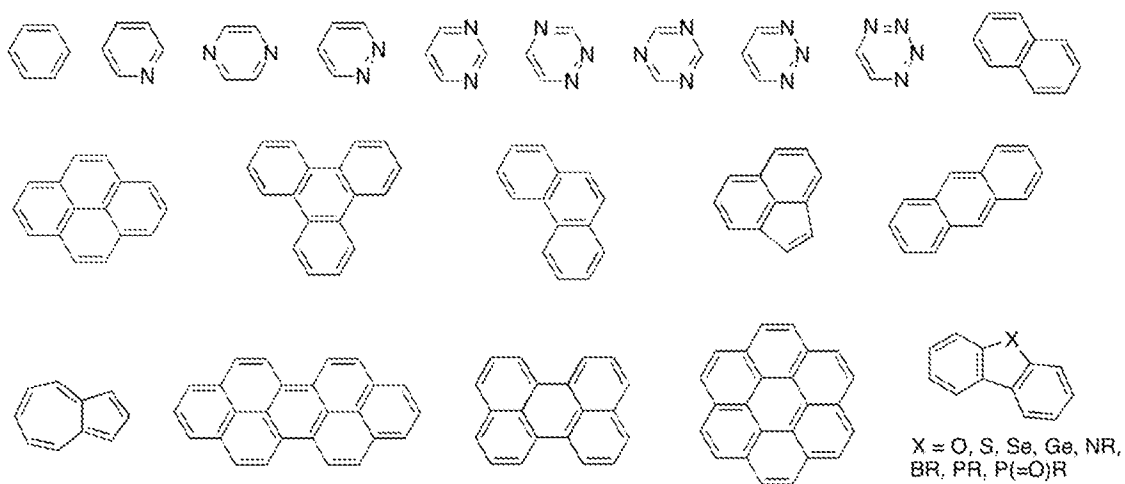
20 In one series of embodiments one or more of m, n, p and q is at least 3, 5 or 8. In one series of embodiments one or more of m, n, p and q is fewer than 10, 8, 6, 4 or 2. In one embodiment one or more of m, n, p and q is from 3 to 6.

In one embodiment m is greater than p.

25 In one embodiment n is greater than q.

In some embodiments each of core W, core X, core Y and core Z is independently selected from an aromatic or a heteroaromatic ring or ring system.

30 In one embodiment one or more of core W, core X, core Y, and core Z is independently selected from, or from derivatives of:



In one embodiment core W and core Y are identical. In one embodiment core X and core Z are identical. In one embodiment all of core W, core X, core Y and core Z are identical. In one embodiment all of core W, core X, core Y and core Z are different from one another.

5

In one embodiment core W, core X and at least one of core Y and core Z are identical. Thus, in this embodiment the compatibiliser comprises a core that is the same as the cores of the first and second compounds.

10

In one embodiment one or more of core W, core X, core Y and core Z is a triphenylene, a perylene or perylene diimide, a coronene, a porphyrin, a thiophene, a triphenyl amine, a carbazole, a benzoimidazole, a ruthenium complex or an iridium complex, and/or a derivative of any one thereof. Where included in an embodiment, a ruthenium complex and/or an iridium complex may be cyclometallated.

15

In one embodiment one or more of core W, core X, core Y and core Z is triphenylene. Triphenylenes are a well-known class of discotic liquid crystals. In one such embodiment core W and/or core Y is triphenylene.

20

In some embodiments one or more of core W, core X, core Y and core Z is or comprises perylene, or is perylene diimide (perylene-3,4,9,10-tetracarboxylic diimide). In some further embodiments core W and/or core Y is or comprises perylene, or is perylene diimide.

25

In some embodiments one or more of core W, core X, core Y and core Z is 2,5,8,11,14,17-hexaphenylhexabenzob[bc,ef,hi,kl,no,qr]coronene. In some further embodiments core W and/or core Y is 2,5,8,11,14,17-hexaphenylhexabenzob[bc,ef,hi,kl,no,qr]coronene.

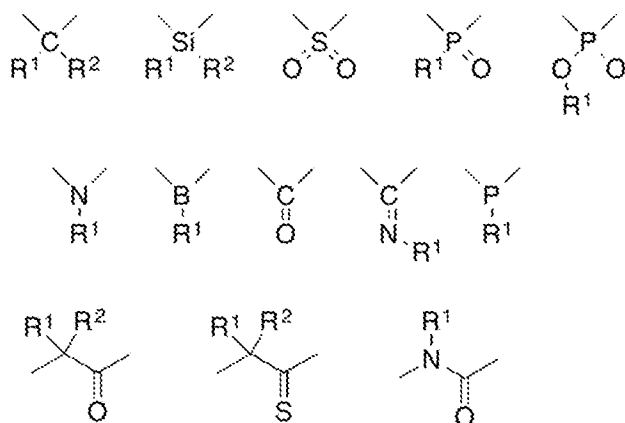
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As is known in the art, discotic liquid crystals are mesophases formed from disc-shaped molecules known as discotic mesogens, which typically comprise an aromatic or heteroaromatic core having flexible chains attached thereto. Such molecules have particular application in OPVs, OLEDs and molecular wires.

Thus, in some embodiments wherein the compatibiliser comprises or is constituted by a discotic mesogen. Such embodiments are advantageous since the compatibiliser is itself functional in that it can be used as an active material in systems such as OPVs and OLEDs. A functional compatibiliser having an 'active' core participates in the system in which the composition is being used. As such, its concentration does not need to be kept low, unlike known additives which are non-functional materials.

L is a linker covalently linking core Y and Z. L comprises at least one atom, which is a carbon atom, a heteroatom, or a metal atom. Suitable heteroatoms include Si, N, S, O, P and B. Suitable metal atoms include, for example Ru, Ir, Pt, Cu, Zn, Ag, Au, Pd, Co, Ni, Fe, Rh, Re, Os and Cr.

In one embodiment L is selected from:



wherein R^1 and R^2 are as defined above.

In some embodiments, the length of the linker is approximately the same, or less than, the combined length of addends A and B. In other embodiments the length of the linker is greater than the combined lengths of addends A and B. In some embodiments the linker has no more than 20, 15, 14, 12, 10 or 9 atoms.

In some embodiments, the linker comprises or consists of addend A, addend B, or addend A covalently linked to addend B.

In one embodiment L is $O-(CH_2)_6-O-Ph-OCH_2$.

In some embodiments the linker L is, and/or comprises, $O-(CH_2)_2-O-(CH_2)_2-$.

In one embodiment the first compound is 2,3,6,7,10,11-hexahexyloxytriphenylene (TP6). In this embodiment core W is triphenylene, addend A is $-\text{OC}_6\text{H}_{13}$ and m is 6.

- 5 In one embodiment the second compound is 2,3,6,7,10,11-hexa(1,4,7-trioxaooctyl)triphenylene (TP6EO2M). In this embodiment core X is triphenylene, addend B is $-\text{OCH}_2\text{CH}_2\text{OCH}_2\text{CH}_2\text{OCH}_3$ and n is 6.

- 10 In one embodiment the compatibiliser is 2,3,6,7,10-pentakis(hexyloxy)-11-((6-(3-((3,4,5-tris(2-(2-methoxyethoxy)ethoxy)benzyl)oxy)phenoxy)hexyl)oxy)triphenylene (TP6Gall). In this embodiment core Y is triphenylene, addend A is $-\text{OC}_6\text{H}_{13}$, p is 5, L is $\text{O}-(\text{CH}_2)_6-\text{O}-\text{Ph}-\text{OCH}_2-$, core Z is benzene, B is $\text{OCH}_2\text{CH}_2\text{OCH}_2\text{CH}_2\text{OCH}_3$ and q is 3.

- 15 In one embodiment the first compound is 2,9-bis(10-((2-(2-methoxyethoxy)ethoxy)methyl)-2,5,8,11,14-pentaoxahexadecan-16-yl)anthra[2,1,9-*def*:6,5,10-*d'e'f'*]diisoquinoline-1,3,8,10(2*H*,9*H*)-tetraone (also referred to herein as BPDI). In this embodiment the core W is perylene-3,4,9,10-tetracarboxylic diimide, addend A is 10-(2-ethoxyethoxy)-2,5,8,12,15,18-hexaoxanonadecane and m is 2.

- 20 In one embodiment the second compound is 2,5,8,11,14,17-hexakis(4-dodecylphenyl)hexabenz[*bc,ef,hi,kl,no,q*r]coronene (also referred to herein as HBC). In this embodiment, the core X is 2,5,8,11,14,17-hexaphenylhexabenz[*bc,ef,hi,kl,no,q*r]coronene, addend B is $\text{C}_{12}\text{H}_{25}$ and n is 6.

- 25 In one embodiment the compatibiliser is 2,5,8,11-tetrakis(4-dodecylphenyl)-14,17-bis(2-(2-ethoxy)ethyl)-9-(10-((2-(2-methoxyethoxy)ethoxy)methyl)-2,5,8,11,14-pentaoxahexadecan-16-yl)anthra[2,1,9-*def*:6,5,10-*d'e'f'*]diisoquinoline-1,3,8,10(2*H*,9*H*)-tetraone)hexabenz[*bc,ef,hi,kl,no,q*r]coronene (also referred to herein as HBC-BPDI).

- 30 In this embodiment core Y is perylene-3,4,9,10-tetracarboxylic diimide, addend A is 10-(2-ethoxyethoxy)-2,5,8,12,15,18-hexaoxanonadecane, p is 1, L is $\text{O}-(\text{CH}_2)_2-\text{O}-(\text{CH}_2)_2-$, the core Z is 2,5,8,11,14,17-hexaphenylhexabenz[*bc,ef,hi,kl,no,q*r]coronene, B is $\text{C}_{12}\text{H}_{25}$ and q is 5.

It will be understood that the compatibiliser of the present invention is not a polymer compatibiliser. In one embodiment the compatibiliser has a molecular weight of less than 20000, 15000, 10000, 8000, 5000, 3000 or 1000g/mol.

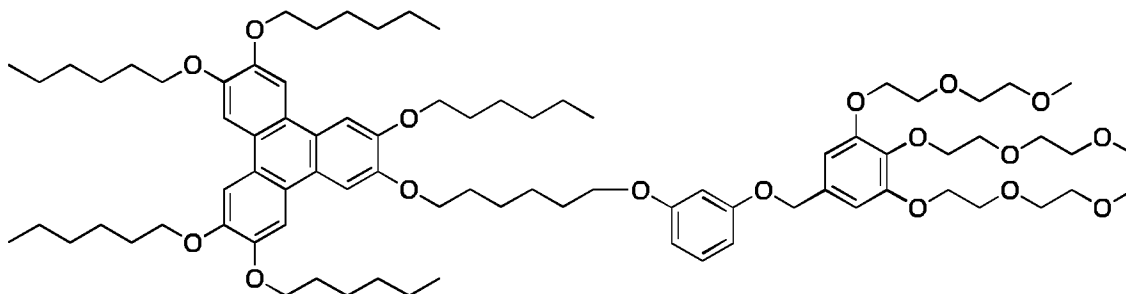
5 According to a second aspect of the present invention there is provided a process for preparing a single isotropic phase by heating the composition of the first aspect.

It will be understood that it is necessary to heat the composition above its clearing point at least once. IUPAC defines clearing point as the temperature at which the transition
10 between the mesophase with the highest temperature range and the isotropic phase occurs.

The invention also resides in compounds for use in embodiments of the first and second aspects of the invention.

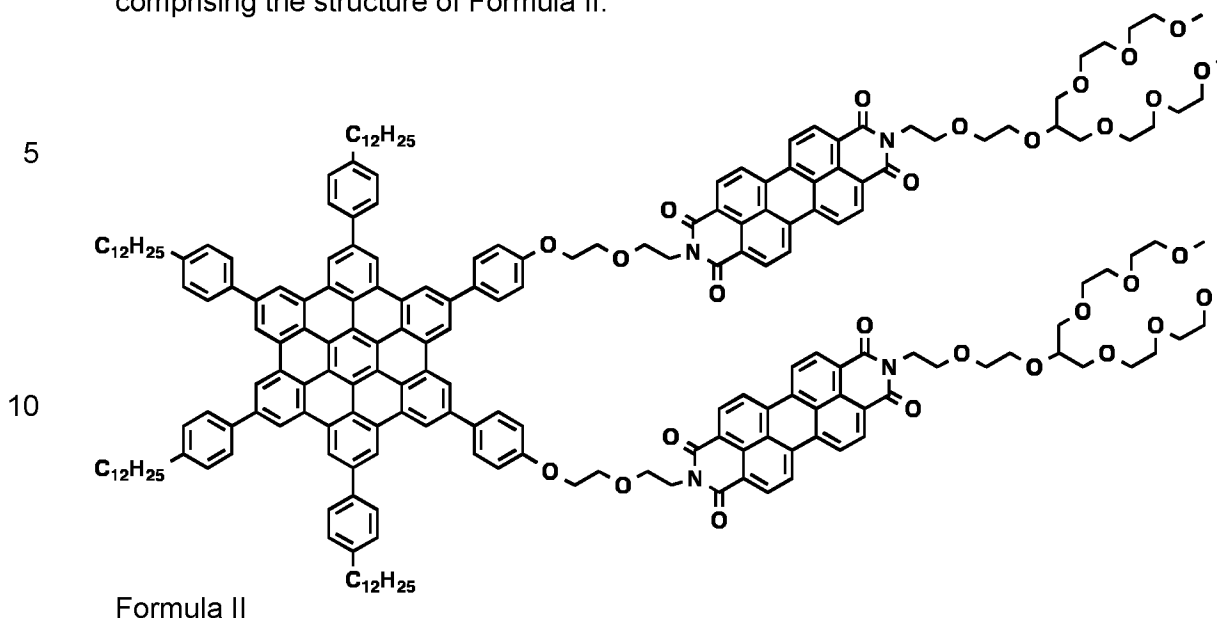
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According to a third aspect of the present invention there is provided a compound comprising the structure of Formula I:



20 Formula I

According to a fourth aspect of the present invention there is provided a compound comprising the structure of Formula II:



Embodiments of the invention are described with reference to the following figures:

Figure 1 shows the structures of the immiscible compounds TP6 and TP6EO2M, and the compatibiliser TP6Gall;

Figure 2 shows polarised optical microscopy (POM) images of equimolar mixtures of TP6 and TP6EO2M in the absence and presence of TP6Gall:

Top: 0mol% TP6Gall a) POM image at 90 °C; b) at 50 °C

Middle: 20mol% TP6Gall c) POM image at 70 °C; d) at 50 °C;

Bottom: 50mol% TP6Gall e) POM image at 70 °C; f) at 50 °C; and

Figure 3 shows the structures of two compounds HBC and BPDI, and the compatibiliser HBC-BPD.

Example 1

Synthetic methods

TP6 and TP6EO2M were obtained according to the procedures of Borner *et al.*, Liq. Cryst., 2006, 33, 1439–1448.

Synthesis of TP6Gall

Synthesis of 3,3',4,4'-tetramethoxybiphenyl (006)

Pd(PPh₃)₄ (428 mg, 0.37 mmol, 1 mol%) was dissolved in a mixture of degassed THF (200 mL) and water (90 mL) and the solution was bubbled with argon for 10 min. 4-Bromo-1,2-dimethoxybenzene (8.00 g, 37.04 mmol, 1 equiv.) and 3,4-dimethoxybenzene boronic acid (8.09 g, 44.45 mmol, 1.2 equiv.) were added and the reaction mixture was stirred at reflux under argon overnight. Solvent was evaporated *in vacuo*, and recrystallisation of the crude from MeOH afforded 006 a lightly brown solid (8.13 g, 80%).

Synthesis of 3,3',4,4'-Tetrahydroxybiphenyl (007)

A solution of 006 (4.00 g, 14.58 mmol, 1 equiv.) in a previously degassed mixture of 48 wt% aqueous hydrobromic acid (120 mL) and glacial acetic acid (120 mL) was stirred overnight at reflux under argon. The reaction mixture was cooled down to room temperature and the product was extracted 5 times with diethyl ether. The etherated phase was washed with a saturated solution of sodium hydrogenocarbonate, brine and the solvent was evaporated *in vacuo*. 007 was obtained as a gray powder and was used in the next step with no further purification.

Synthesis of 3,3'-4,4'-tetrahexyloxybiphenyl (008)

A solution of 007 (3.14 g, 14.39 mmol, 1 equiv.), potassium carbonate (23.8 g, 172.7 mmol, 12 equiv.) and 1-bromohexane (16.2 mL, 115.1 mmol, 8 equiv.) in DMF (8 mL) was stirred overnight at 120 °C. The reaction mixture was cooled down to room temperature, the solvent was evaporated *in vacuo* and the resulting solid was filtered through a pad of silica (eluent: ethyl acetate). The solvent was evaporated and the product was recrystallized in acetonitrile to afford 008 as a light brown solid (4.71 g, 59%).

Synthesis of 2-hexyloxyphenol (009)

To a solution of K₂CO₃ (14.21 g, 103 mmol, 2 equiv.) and catechol (5.670 g, 51.5 mmol, 1 equiv.) in DMF (27 mL), 1-bromohexane (7.3 mL, 51.5 mmol, 1 equiv.) was added dropwise. The mixture was stirred overnight at 120 °C. Solvent was evaporated *in vacuo* and the obtained solid was filtered through a pad of silica (eluent : ethyl

acetate). Purification by chromatography (SiO₂, hexane/ethyl acetate = 20:1) afforded 009 as a colorless oil (4.652 g, 47%).

Synthesis of 1-hexyloxy-2-isopropoxybenzene (010)

5 A solution of K₂CO₃ (1.07 g, 7.7 mmol, 1 equiv.), 009 (1.5 g, 7.7 mmol, 1 equiv.), and 2-bromopropane (2.16 mL, 23.2 mmol, 3 equiv.) in DMF (5.8 mL) was stirred at 55 °C and monitored by TLC. After 44 h stirring, 2-bromopropane (0.72 mL, 7.7 mmol, 1 equiv.) was added and further reacted for 22 h. Water was added and the product was extracted with ethyl acetate. The organic phase was washed with brine, dried over
10 MgSO₄ and the solvent was evaporated *in vacuo*. Purification by column chromatography (SiO₂, hexane/ethyl acetate = 20:1) afforded 010 as a colorless oil (1.240 g, 68%).

Synthesis of 2-hydroxy-3,6,7,10,11-pentahexyloxytriphenylene (011)

15 To a solution of 010 (670 mg, 2.84 mmol, 1.7 equiv.) and 008 (943 mg, 1.70 mmol, 1 equiv.) in DCM (6 mL), FeCl₃ (1.38 g, 8.50 mmol, 5 equiv.) was slowly added. The dark blue solution was stirred overnight at room temperature. The mixture was then poured into cold methanol (40 mL) and stirred for 0.5 h. The solution was filtered and the gray precipitate was washed with cold methanol. Purification by column chromatography
20 (SiO₂, petroleum ether/DCM = 7:3 to 4:6), followed by recrystallization in ethanol afforded 011 as white crystals (360.8 mg, 48%).

Synthesis of ethyl 3,4,5-tris(1,4,7-trioxaoctyl) benzoate (012)

To a solution of diethylene glycol monomethyl ether (11.61 g, 96.6 mmol, 1 equiv.) in
25 THF (40 mL) at 0 °C was added a solution of potassium hydroxide (9.660 g, 214.5 mmol, 2.5 equiv.) in water (40 mL). A solution of tosyl chloride (27.63 g, 144.9 mmol, 1.5 equiv.) in THF (40 mL) was then added dropwise to the solution and stirred for 1 h at 0 °C. The reaction mixture was brought back to room temperature and stirred for another 1 h. The product was extracted with diethyl ether. The etherated
30 phase was then washed with a diluted solution of potassium hydroxide, then water and brine, dried over MgSO₄ and the solvent was evaporated. The resulting oil was used in the next step with no further purification.

A solution of ethyl gallate (293 mg, 1.61 mmol, 1 equiv.), K₂CO₃ (2.23 g, 16.1 mmol, 10 equiv.) and the tosylate derivate previously synthesized in degassed DMF (10 mL)
35

was stirred at 80 °C for 20 h. The solvent was then removed *in vacuo* and the crude filtered through a pad of Celite. DCM was added and the product was extracted with DCM. The organic phase was washed with a solution of HCl, brine, dried over MgSO₄ and the solvent was evaporated. The yellow oil was purified by column chromatography (SiO₂, DCM/MeOH = 97:3) to afford 012 as a colorless oil (486 mg, 49%).

Synthesis of 3,4,5-tris(1,4,7-trioxaoctyl) benzyl alcohol (013)

To a stirred suspension of LiAlH₄ (200 mg, 5.15 mmol, 1.3 equiv.) in dry THF (4 mL) was added a solution of 011 (2,00 g, 3.96 mmol, 1 equiv.) in dry THF (8 mL) at 0 °C under argon atmosphere. The mixture was brought back to room temperature and stirred for 3 h and was then quenched by addition of successively isopropyl alcohol (1 mL), cold water (3 mL) and a 30% aqueous solution of sodium hydroxide (1 mL). After filtration of the crude, the product was extracted with ethyl acetate, washed with brine, dried over MgSO₄, and the solvent was evaporated *in vacuo* to afford 013 as a light yellow oil (1.137 g, 62%).

3,4,5-Tris(1,4,7-trioxaoctyl) benzyl chloride (014)

To a solution of 013 (200 mg, 0.43 mmol, 1 equiv.) in dry DCM (25 mL) was added dropwise a solution of SOCl₂ (320 µL, 4.3 mmol, 10 equiv.) in dry DCM (10 mL) at room temperature. The solution was stirred at room temperature for 3 h. The solvent was evaporated *in vacuo*, affording 014 in quantitative yield.

Synthesis of 2-(6-bromohexyloxy)-3,6,7,10,11-pentahexyloxytriphenylene (015)

A solution of 011 (192 mg, 0.27 mmol, 1 equiv.), K₂CO₃ (75 mg, 0.54 mmol, 2 equiv.) and 1,6-dibromohexane (200 µL, 1.34 mmol, 5 equiv.) in degassed DMF (1 mL) was stirred at 120 °C for 21 h under argon. DMF was then evaporated *in vacuo* and the resulting dough was filtrated through a pad of Celite and washed with DCM. Solvent was evaporated and the resulting solid was recrystallized in ethanol to afford 015 as an off-white solid (140.3 mg, 57%).

2-(6-(3-hydroxyphenyloxy)hexyloxy)-3,6,7,10,11-pentahexyloxytriphenylene (016)

A solution of 2-(6-bromohexyloxy)-3,6,7,10,11-pentahexyloxytriphenylene (246.1 mg, 0.27 mmol, 1 equiv.), resorcinol (149 mg, 1.35 mmol, 5 equiv.) and K₂CO₃ (75 mg, 0.54 mmol, 2 equiv.) in DMF (4 mL) was heated at 120 °C and monitored by TLC

(Hexane/DCM = 3:6). After 20 h heating, the reaction mixture was cooled down to room temperature and acidified with a diluted solution of HCl. Extraction with ethyl acetate followed by evaporation *in vacuo* of the solvent afford an orange solid which was purified by column chromatography (SiO₂, DCM). 016 was obtained as a white solid (105.9 mg, 42%).

Synthesis of TP6Gall (017)

A solution of 014 (163 mg, 0.339 mmol, 3 equiv.), 016 (105.9 mg, 0.113 mmol, 1 equiv.) and K₂CO₃ (47 mg, 0.339 mmol, 3 equiv.) in degassed DMF (5 mL) was stirred under argon at 120 °C for 24 h. The solvent was evaporated *in vacuo*. The product was extracted three times with ethyl acetate, dried over MgSO₄ and the solvent was evaporated *in vacuo*. Purification by column chromatography (SiO₂, DCM → DCM/MeOH = 97.5 : 2.5) affords TP6Gall as an off-white solid in a 87% yield.

15 Analysis

The mesomorphic and thermal properties of TP6, TP6EO2M, TP6Gall (structures shown in Fig. 1), and mixtures were examined by polarised optical microscopy (POM). All blends were prepared by dissolving the components in dichloromethane at the required ratio followed by solvent evaporation and overnight drying under vacuum. Data discussed are obtained after heating at least once the mixtures above the clearing point.

When TP6 and TP6EO2M are mixed in an equimolar ratio, macrophase separation is observed both above (Fig. 2a) and below (Fig. 2b) the clearing point with the formation of large domains of TP6 and TP6EO2M (>10's micrometers scale). Upon addition of TP6Gall to the TP6:TP6EO2M mix, the morphology of the blend is altered. Upon addition of 20mol% TP6Gall, smaller domains (≈micrometer scale) are observed in addition to large domains both above (Fig. 2c) and below (Fig. 2d) the clearing point. Upon addition of 50mol% TP6Gall, only one phase is observed above the clearing point (Fig. 2e). Upon cooling, unexpectedly, TP6EO2M phase segregates from a phase composed of TP6/TP6Gall and form a dense crystalline network (Fig. 2f).

Importantly, this particular morphology is well reproducible upon heating-cooling cycles, most likely because the liquid phase is a single isotropic phase, even if mechanically challenged during the process.

5 Example 2

Synthetic methods

(4-Dodecylphenyl)magnesium bromide

10 In a flame-dried round bottom flask equipped with a condenser, to a solution of magnesium turnings (320 mg, 13.2 mmol, 1.5 equiv) and iodine in THF (6 mL), solution of 4-bromododecylbenzene (2.93 g, 9 mmol, 1 equiv) in THF (15 mL) was added dropwise. The mixture was stirred at reflux under argon for 6 h. A greenish solution was obtained.

15

Di(4-n-dodecylbiphenyl)acetylene (044)

To a solution of di(4-bromophenyl)acetylene (719 mg, 2.14 mmol, 1 equiv) in dry THF (60 mL) was added the fresh solution (ca. 0.4 mol/L) of (4-dodecylphenyl)magnesium bromide (20 mL, 8.57 mmol, 4 equiv) in a flame-dried apparatus. PdCl₂(dppf) (138 mg, 20 0.17 mmol, 8 mol%) was then added and the reaction mixture was stirred at reflux under argon overnight. The solution was then cooled down to room temperature. The obtained white precipitate was filtered and washed with methanol and petroleum ether. 044 was obtained as an off-white solid (1.1672 mg, 82 %).

25 Hexa(4-n-dodecylbiphenyl)benzene (045)

To a flame-dried apparatus and under argon atmosphere, a suspension of 044 (600 mg, 0.9 mmol, 1 equiv), Co(CO)₈ (31 mg, 0.09 mmol, 10 mol%) in dioxane (degassed, dried on 3 Å molecular sieve) was refluxed for 3 h. The solvent was evaporated and purification by column chromatography (SiO₂, DCM/PE = 2:8) afforded 045 in a 55 % 30 yield (990 mg).

Synthesis of HBC (046)

To a solution of 045 (612 mg, 0.31 mmol, 1 equiv) in anhydrous DCM (400 mL) and under constant bubbling of argon was added a solution of anhydrous FeCl₃ (2.23 g, 35 13.8 mmol, 45 equiv) in CH₃NO₂ (50 mL). The reaction mixture was stirred under

constant bubbling or argon at 21 °C for 2 h. After a reductive work-up by adding methanol (400 mL) the precipitate was filtered *in vacuo* and washed with methanol. The dark brown precipitate was dissolved in the minimum amount of DCM and precipitated out again by adding methanol to remove any residual iron chloride. 046
5 was obtained as a yellow solid (405 mg, 74 %). MS (LD+, retinoic acid): $m/z[M+H]^+ = 1988.1$.

2,5,8,12,15,18-hexaoxonadecan-10-ol (079)

In a flame-dried apparatus and under argon atmosphere, a solution of sodium hydride
10 (60 % in mineral oil, 12.24 g, 510 mmol, 10 equiv) in dry THF (80 mL) was stirred at reflux. A solution of diethylene glycol monomethylether (30.6 g, 255 mmol, 5 equiv) in THF (20 mL) was then added dropwise over a period of 30 min. The reaction mixture was further stirred at reflux for 1 h. Epichlorhydrin (4.72 g, 51 mmol, 1 equiv) was then added dropwise. After stirring at reflux for 24 h, the mixture was cooled down to room
15 temperature. DCM and water were added. Extraction of the organic compounds with DCM followed by high vacuum distillation (150 °C) afforded 079 as a light yellow oil (11.07 g, 74 %).

10-(2-(2-bromoethoxy)ethoxy)-2,5,8,12,15,18-hexaoxonadecane (084)

In a flame-dried apparatus and under argon, a solution of sodium hydride (60% in mineral oil, 186 mg, 7.76 mmol, 2 equiv) in dry THF (70 mL) was stirred at room temperature for 15 min. 079 (1.15 g, 3.88 mmol, 1 equiv) was then added. After 30 min stirring, bis(2-bromoethyl)ether (4.5 g, 19.4 mmol, 5 equiv) was added dropwise and the reaction mixture was stirred overnight at room temperature. Isopropanol (3 mL) was
25 added. Filtration through a pad of Celite (Eluent : DCM) followed by the evaporation of the solvent afforded a light brown oil which was purified by column chromatography (SiO₂, neat ACN) to obtain the pure 084 as a light brown oil (891 mg, 51 %).

2-(10-((2-(2-methoxyethoxy)ethoxy)methyl)-2,5,8,11,14-pentaoxahexadecan-16-yl)isoindoline-1,3-dione (086)

A solution of 084 (149 mg, 0.33 mmol, 1 equiv) and potassium phthalimide (122 mg, 0.66 mmol, 2 equiv) in DMF (2 mL) was stirred overnight at 85 °C. After cooling to room temperature, the mixture was filtrated through a pad of Celite (Solvent : DCM) and the filtrate was evaporate *in vacuo*. Purification by column chromatography (SiO₂,
35 EtOAc/MeoH = 95:5) afforded 086 as a light yellow oil (114mg, 67 %).

10-((2-(2-methoxyethoxy)ethoxy)methyl)-2,5,8,11,14-pentaoxahehexadecan-16-amine (087)

A solution of 086 (353 mg, 0.687 mmol, 1 equiv) and hydrazine hydrate (0.1 mL) in ethanol (12 mL) was stirred overnight at reflux. The solvent was evaporated *in vacuo* and the resulting dough was filtrated though a pad of Celite (Eluent : MeOH). The solvent was evaporated. Diethyl ether was added and the solution was sonicated for 10 min. Filtration though a pad of Celite (eluent : diethyl ether) afforded 087 as a yellow oil (220 mg, 84 %).

BPDI

Under argon, 3,4,9,10-Perylenetetracarboxylic dianhydride (75 mg, 0.192 mmol, 1 equiv), zinc acetate (35 mg, 0.192 mmol, 1 equiv) and 087 (220 mg, 0.575 mmol, 3 equiv) in quinoline (3 mL) were stirred at 180 °C for 6 h. After cooling down to room temperature chloroform was added and the organic phase was washed with a 1M aqueous solution of HCl (x4). The solvent was evaporated. BPDI was further purified by preparative HPLC (C₁₈, Isocratic ACN/H₂O = 1:1) and obtained as a red solid in a 51 % yield.

1,3-bis(4-bromophenyl)propan-2-one (056)

To a flame-dried apparatus and under argon, a solution of *p*-bromophenylacetic acid (5.00 g, 23.3 mmol, 2 equiv) in dry DCM (50 mL) was added to a solution of DCC (4.8 g, 23.3 mmol, 2 equiv) and DMAP (709 mg, 5.8 mmol, 0.5 equiv) in dry DCM (50 mL). The reaction mixture was stirred at room temperature for 24 h. The precipitate was removed by filtration and the filtrate was evaporated *in vacuo*. The solid was recrystallized from methanol to afford 056 as a light orange solid (1.94 g, 45 %).

4,4'-dibromobenzil (101)

A solution of bis(4-bromophenyl)acetylene (1.500 g, 4.46 mmol, 1 equiv), and iodine (566 mg, 2.23 mmol, 0.5 equiv) in DMSO (7.5 mL) was stirred at 155 °C for 6 h. The reaction mixture was then cooled down to room temperature and a 1% aqueous solution of Na₂S₂O₄ (100 mL) was added. The resulting yellow crystals were filtrated and washed with water. Recrystallisation from dioxane afforded 101 as yellow crystals (1.06 g, 65 %).

Tetra(4-bromophenyl)cyclopentadienone (102)

To a solution of 056 (310 mg, 0.84 mmol, 1 equiv) and 101 (310 mg, 0.84 mmol, 1 equiv) in ethanol (3 mL) was added a solution of KOH (47 mg, 0.84 mmol, 1 equiv) in ethanol (0.5 mL). The reaction mixture was stirred at reflux for 40 min. The dark red solution was then cooled down to 0 °C. The resulting dark red precipitate was filtered and washed with cold ethanol, affording 102 as dark red crystals (365 mg, 62 %).

Bis(4-methoxybiphen-4'-yl)acetylene (094)

In a flame-dried apparatus and under argon, to a solution of bis(4-bromophenyl)acetylene (567 mg, 1.69 mmol, 1 equiv) in dry THF (40 mL) was added a fresh solution of 4-methoxyphenylmagnesium bromide (4 equiv) in THF (15 mL). PdCl₂(dppf) (110 mg, 0.14 mmol, 8 mol%) was then added. The reaction mixture was stirred at reflux overnight under argon. The solution was then cooled to room temperature. The resulting white precipitate was then filtered *in vacuo* and washed with methanol and petroleum ether. 094 was obtained as an off-white solid (528 mg, 80 %).

1,2,3,4-tetra(4-bromophenyl)-5,6-di(4'-methoxybiphenyl-4-yl)benzene (103)

Under argon, a solution of 102 (179 mg, 0.256 mmol, 1 equiv) and 094 (127 mg, 0.325 mmol, 1.3 equiv) in degassed diphenyl ether (0.75 mL) was stirred at 250 °C for 24 h. The crude was purified by column chromatography (SiO₂, dry load, Hexane/EtOAc = 8:2) to afford 103 as a pale yellow solid (193 mg, 71 %).

1,2,3,4-tetra(4-dodecylphenyl)-5,6-di(4'-methoxybiphenyl-4-yl)benzene (104)

In a flame-dried apparatus, to a solution of 103 (400 mg, 0.376 mmol, 1 equiv) and *trans*-PdCl₂(PPh₃)₂ (11 mg, 15 μmol, 4 mol%) in THF (7 mL) was added a fresh solution of 4-dodecylbenzene magnesium bromide (10 equiv) in THF (8 mL). The reaction mixture was stirred at reflux overnight under argon. After evaporation of the solvent *in vacuo*, the crude was dissolved in the minimum amount of DCM and methanol was added. The resulting precipitate was filtered and washed with methanol. Purification by column chromatography (SiO₂, dry load, Hexane : EtOAc = 9/1) afforded 104 as a waxy yellow solid (281 mg, 43 %).

2,5,8,11-tetrakis(4-dodecylphenyl)-14,17-bis(4-methoxyphenyl)hexabenz[bc,ef,hi,kl,no,qr]coronene (105)

To a solution of 104 (175 mg, 101 μ mol, 1 equiv) in anhydrous DCM (140 mL) and under a constant flux of argon was added a solution of anhydrous FeCl_3 (737 mg, 4.5 mmol, 4.5 equiv) in CH_3NO_2 (18 mL). The reaction mixture was stirred at 21 °C under a constant flux of argon for 2 h. Methanol (150 mL) was then added to quench the reaction. The white precipitate was filtrated *in vacuo* and washed with methanol. 105 was obtained as a yellow solid in a 96 % yield (166 mg) and was used in the next step without further purification.

4,4'-(8,11,14,17-tetrakis(4-dodecylphenyl)hexabenz[bc,ef,hi,kl,no,qr]coronene-2,5-diyl)diphenol (106)

To a solution of 105 (50 mg, 29 μ mol, 1 equiv) in anhydrous DCM (2 mL) was added 2 drops of BBr_3 at 0 °C. The reaction mixture was stirred for 1 h at 0 °C. The black solution was brought back to room temperature and stirred overnight. Water was added to quench the residual BBr_3 . Methanol was added and the resulting precipitate was filtrated *in vacuo* and washed with methanol. 106 was obtained as a yellow solid in a quantitative yield and was used in the next step with no further purification.

Perylene-3,4,9,10-tetracarboxylic acid monoanhydride monopotassium carboxylate (097)

A solution of 3,4,9,10-perylenetetracarboxylic anhydride (3.00 g, 7.65 mmol, 1 equiv) in a 5 % aqueous KOH solution (35 mL) was heated at 90 °C for 4 h. After cooling down the reaction mixture to room temperature, a 10 % aqueous H_3PO_4 solution (12.5 mL) was added and stirred for 1 h at 90 °C. The resulting precipitate was filtrated and washed with water. The obtained bordeaux solid was dried at 80 °C overnight. 097 was obtained in a 96 % yield (3.29 g) and was used in the next step without further purification.

3,4,9,10-Perylenetetracarboxylic-3,4-anhydride-9,10-imide (098)

A solution of 097 (2.00 g, 4.46 mmol, 1 equiv) and a 35 % ammonium hydroxide solution (2.5 mL) in water (100 mL) was stirred at 0-5 °C for 4 h. The reaction mixture was then stirred at 90 °C for another 2 h. A 25 % aqueous potassium carbonate solution (25 mL) was then added and the solution was further stirred for 1 h at 90 °C. After cooling it down to room temperature, the obtained precipitate was filtrated and

washed with a 2 % aqueous potassium carbonate solution until the filtrate was colourless (ca. 150 mL). The residue was dissolved in an hot 3.5 % aqueous KOH solution and the resulting solution was heated at 70 °C for 5 min. After filtration, the filtrate was acidified with a 10 % aqueous HCl solution until pH = 1. After filtration and washing with water, the dark red residue was dried in the oven (T = 80 °C) overnight and 098 was obtained as a dark red powder in a 18 % yield (308 mg).

2-(2-(2-hydroxyethoxy)ethyl)anthra[2,1,9-def:6,5,10-d'e'f']diisoquinoline-1,3,8,10(2H,9H)-tetraone (099)

A solution of 098 (250 mg, 0.639 mmol, 1 equiv), zinc diacetate monohydrate (59 mg, 0.320 mmol, 0.5 equiv) and 2-(2-aminoethoxy)ethanol (95 µL, 0.959 mmol, 1.5 equiv) in quinoline (4 mL) was heated at 180 °C for 6 h. After cooling down to room temperature, a 1 M solution of HCl was added and the solution was filtered *in vacuo*. The residue was washed with a 3.5 % KOH solution (to remove any residual starting material) and with water. After drying the residue overnight in an oven (T= 100 °C) 099 was afforded as a dark red powder (236 mg, 77 %).

2-(2-(2-hydroxyethoxy)ethyl)-9-(10-((2-(2-methoxyethoxy)ethoxy)methyl)-2,5,8,11,14-pentaoxahexadecan-16-yl)anthra[2,1,9-def:6,5,10-d'e'f']diisoquinoline-1,3,8,10(2H,9H)-tetraone (100)

Under an argon atmosphere, a solution of 099 (200 mg, 0.42 mmol, 1 equiv), 084 (374 mg, 0.84 mmol, 2 equiv), K₂CO₃ (116 mg, 0.84 mmol, 2 equiv) and KI (14 mg, 84 µmol, 20 mol%) in dry DMF (10 mL) was stirred at 80 °C for 3 days. Filtration through a pad of Celite (Eluent : DCM) followed by precipitation from hexane affords 100 as a dark red solid (185 mg, 58 %). 100 was further purified by preparative HPLC

HBC-BPDI (0108)

To a solution of 100 (101 mg, 119 µmol, 1 equiv) and triethylemaine (120 mg, 1.19 mmol, 10 equiv) in DCM (4 mL) at 0 °C was added mesyl chloride (96 mg, 838 µmol, 7 equiv). The solution was stirred at 0 °C for 3 h. the obtained orange solution was wash with water, brine and dried over MgSO₄ and the solvent was evaporated. The obtained 100-OMs was used in the next step with no further purification.

- To a solution of 0106 (50 mg, 30 μ mol, 1 equiv), the freshly prepared 100-OMs (4 equiv), and Cs_2CO_3 (39 mg, 119 μ mol, 4 equiv) in a 1:4 mixture of DMF/THF was stirred at 90 °C for 24 h under an argon atmosphere. The crude was then dissolved in DCM and the organic phase was washed with water, brine and dried over MgSO_4 .
- 5 Evaporation of the solvent *in vacuo* afforded a bright red dough which was purified by recrystallisation from acetonitrile (to remove the residual 100-OMs). 108 was obtained as a dark red solid in a 40 % yield (40 mg).

Claims

1. A composition comprising
- 5 a first compound represented by the formula:
core $W(A)_m$;
a second compound represented by the formula:
core $X(B)_n$; and
a compatibiliser compound represented by the formula:
10 $([\text{core } Y(A)_p] - L)_k - [\text{core } Z(B)_q]$;
wherein
each of core W, core X, core Y and core Z comprises an aromatic or a
heteroaromatic ring or ring system;
A is an addend of core W and core Y and B is an addend of core X and core Z;
each of k, m, n, p and q is independently selected from an integer from 1 to 10;
15 L is a linker covalently linking core Y and core Z; and
wherein the first and second compounds are immiscible.
2. The composition of claim 1, wherein k is equal to 1.
- 20 3. The composition of claim 1 or claim 2, wherein the first and second compounds form a single isotropic phase.
4. The composition of any one of claims 1 to 3, wherein each of core W, core X, core Y and core Z is a mesogen.
- 25 5. The composition of any preceding claim, wherein core W and core Y are identical.
6. The composition of any preceding claim, wherein core X and core Z are identical.
- 30 7. The composition of any one of claims 1 to 3, wherein core W, core X and at least one of core Y and core Z are identical.
8. The composition of any preceding claim, wherein one or more of core W, core X, core Y and core Z is, or comprises, a triphenylene, a perylene, a coronene, a

porphyrin, a thiophene, a triphenyl amine, a carbazole, a benzoimidazole, a ruthenium complex or an iridium complex, or a derivative of any one thereof.

9. The composition of claim 8, wherein one or more of core W, core X, core Y and core Z is triphenylene, or comprises or is constituted by perylene, or is perylene diimide.

10. The composition of any one of claims 1 to 7, wherein one or more of core W, core X, core Y and core Z is 2,5,8,11,14,17-hexaphenylhexabenzob[bc,ef,hi,kl,no,qr]coronene.

11. The composition of any preceding claim, wherein the first compound is hydrophobic and the second compound is hydrophilic.

12. The composition of any preceding claim, wherein the compatibiliser is an amphiphile.

13. The composition of any preceding claim, wherein the proportion of compatibiliser relative to the total of compatibiliser + first compound + second compound is from 3 to 70 mol%.

14. The composition of any preceding claim, wherein the ratio of the first compound (mol %) to the second compound (mol %) is from 90 : 10 to 10 : 90.

15. The composition of any preceding claim, wherein the composition consists of the first compound, the second compound and the compatibiliser.

16. The composition of any preceding claim, wherein A and/or B is a straight-chain or branched or cyclic alkyl group having from 1 to 50 carbon atoms, in which one or more adjacent or non-adjacent hydrocarbon groups may be replaced by -O-, -S-, -CR¹R²-, -S(=O)-, -S(=O)₂-, -SiR¹R²-, -GeR¹R²-, -NR¹-, -BR¹-, -PR₁-, -P(=O)R₁-, -P(=O)OR₁-, -C(=O)-, -C(=S)-, -C(=R¹R²)-, -CR¹=CR²-, -C≡C-, -C(=O)O-, -OC(=O)-, -C(=NR¹)-, -C=NR¹-, -NR¹C(=O)-, -C(=O)NR¹-, -NR¹C(=S)- or -C(=S)NR¹-, and in each of which one or more hydrogen atoms may be replaced by F, -Cl, -Br, -I, -NO₂, -CN, -OH, -C(=O)OR¹, -OC(=O)R¹, a straight-chain or branched or cyclic alkyl, alkoxy, amine, phosphine, phosphite, phosphonite, silane, germane, borane, borate, boronate,

sulfane, sulfinyl, sulfonyl group, an aryl, heteroaryl, alkanyl, alkenyl, alkynyl group which may be substituted by one or more non aromatic radicals, and wherein R^1 and R^2 are independently selected from hydrogen, halo, alkyl, alkenyl, alkynyl, heteroalkyl, aryl or heteroaryl.

5

17. The composition of claim 16, wherein A is an alkyl or alkoxy group having from 1 to 20 carbon atoms.

18. The composition of claim 17, wherein A is $-O(CH_2)_aCH_3$ where a is from 3 to 10.

10

19. The composition of claim 16, wherein B is $OCH_2CH_2(OCH_2CH_2)_bOCH_3$ where b is from 0 to 10.

20. The composition of claim 16, wherein A is or comprises 10-(2-ethoxyethoxy)-2,5,8,12,15,18-hexaoxanonadecane.

15

21. The composition of any preceding claim, wherein one or more of m, n, p and q is at least 3.

20

22. The composition of any preceding claim, wherein L is $O-(CH_2)_6-O-Ph-OCH_2-$ or $O-(CH_2)_2-O-(CH_2)_2-$.

23. The composition of any one of claims 1 to 9, 11 to 19, 21 or 22, wherein the compatibiliser is 2,3,6,7,10-pentakis(hexyloxy)-11-((6-(3-((3,4,5-tris(2-(2-methoxyethoxy)ethoxy)benzyl)oxy)phenoxy)hexyl)oxy)triphenylene (TP6Gall) (Formula I).

25

24. The composition of any one of claims 1 to 9, 11 to 19, or 21 to 23, wherein the first compound is 2,3,6,7,10,11-hexahexyloxytriphenylene (TP6).

30

25. The composition of any one of claims 1 to 9, 11 to 19, or 21 to 24, wherein the second compound is 2,3,6,7,10,11-hexa(1,4,7-trioxaoctyl)triphenylene (TP6EO2M).

26. The composition of any one of claims 1 to 16 or 20 to 22, wherein the compatibiliser is 2,5,8,11-tetrakis(4-dodecylphenyl)-14,17-bis(2-(2-ethoxy)ethyl)-9-(10-

35

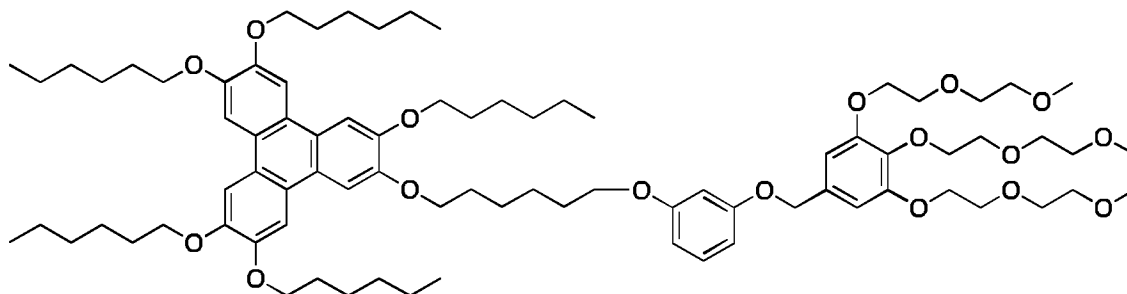
((2-(2-methoxyethoxy)ethoxy)methyl)-2,5,8,11,14-pentaoxahexadecan-16-yl)anthra[2,1,9-*def*:6,5,10-*d'e'f'*]diisoquinoline-1,3,8,10(2*H*,9*H*)-tetraone)hexabenzo[*bc,ef,hi,kl,no,qr*]coronene (HBC-BPDI)(Formula II).

27. The composition of any one of claims 1 to 16, 20 to 22 or 26 wherein the first compound is 2,9-bis(10-((2-(2-methoxyethoxy)ethoxy)methyl)-2,5,8,11,14-pentaoxahexadecan-16-yl)anthra[2,1,9-*def*:6,5,10-*d'e'f'*]diisoquinoline-1,3,8,10(2*H*,9*H*)-tetraone (BPDI).

28. The composition of any one of claims 1 to 16, 20 to 22, 26 or 27 wherein the second compound is 2,5,8,11,14,17-hexakis(4-dodecylphenyl)hexabenzo[*bc,ef,hi,kl,no,qr*]coronene.

29. A process for preparing a single isotropic phase by heating the composition of any one of claims 1 to 28.

30. A compound comprising the structure of Formula I:



Formula I

31. A compound comprising the structure of Formula II:

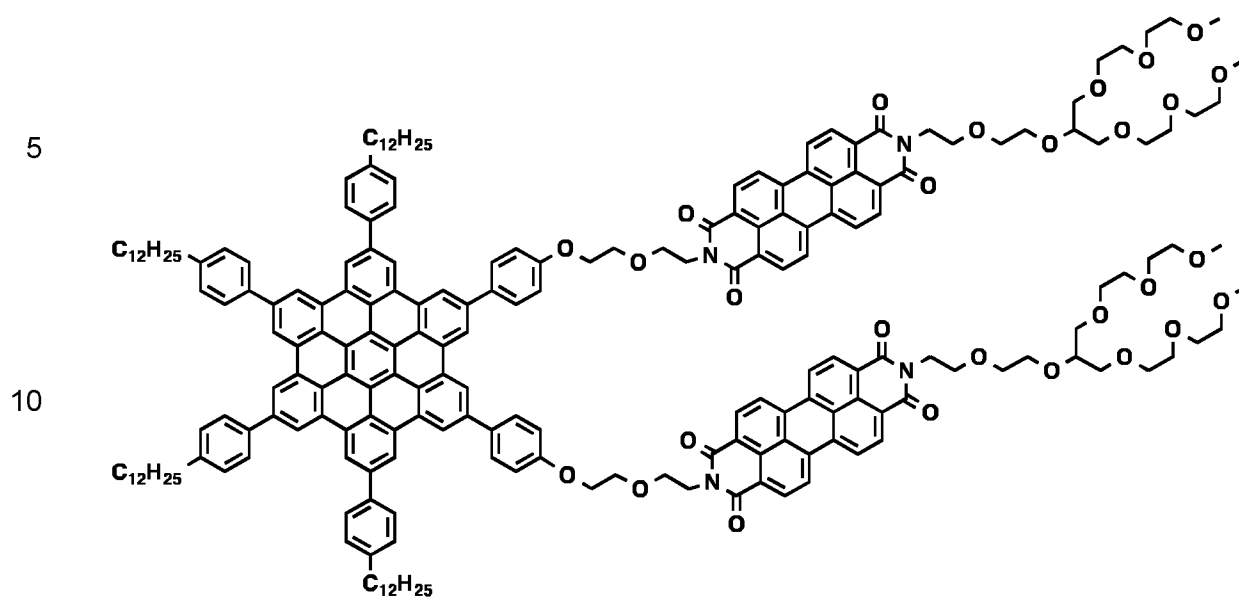


Figure 1

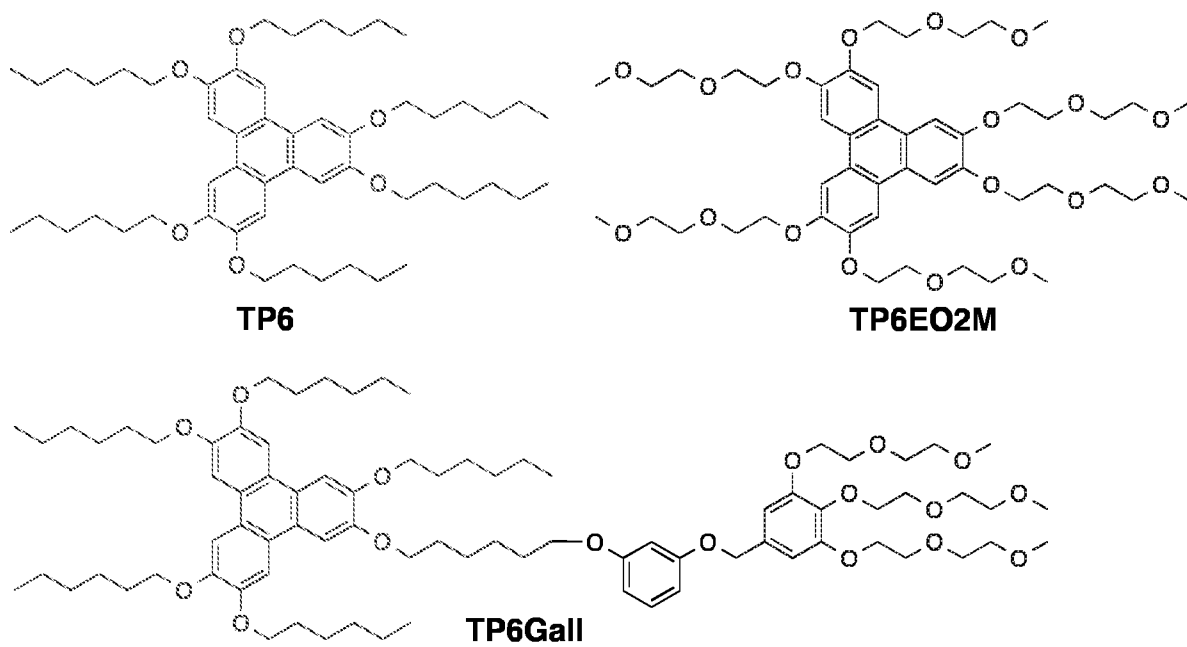


Figure 2

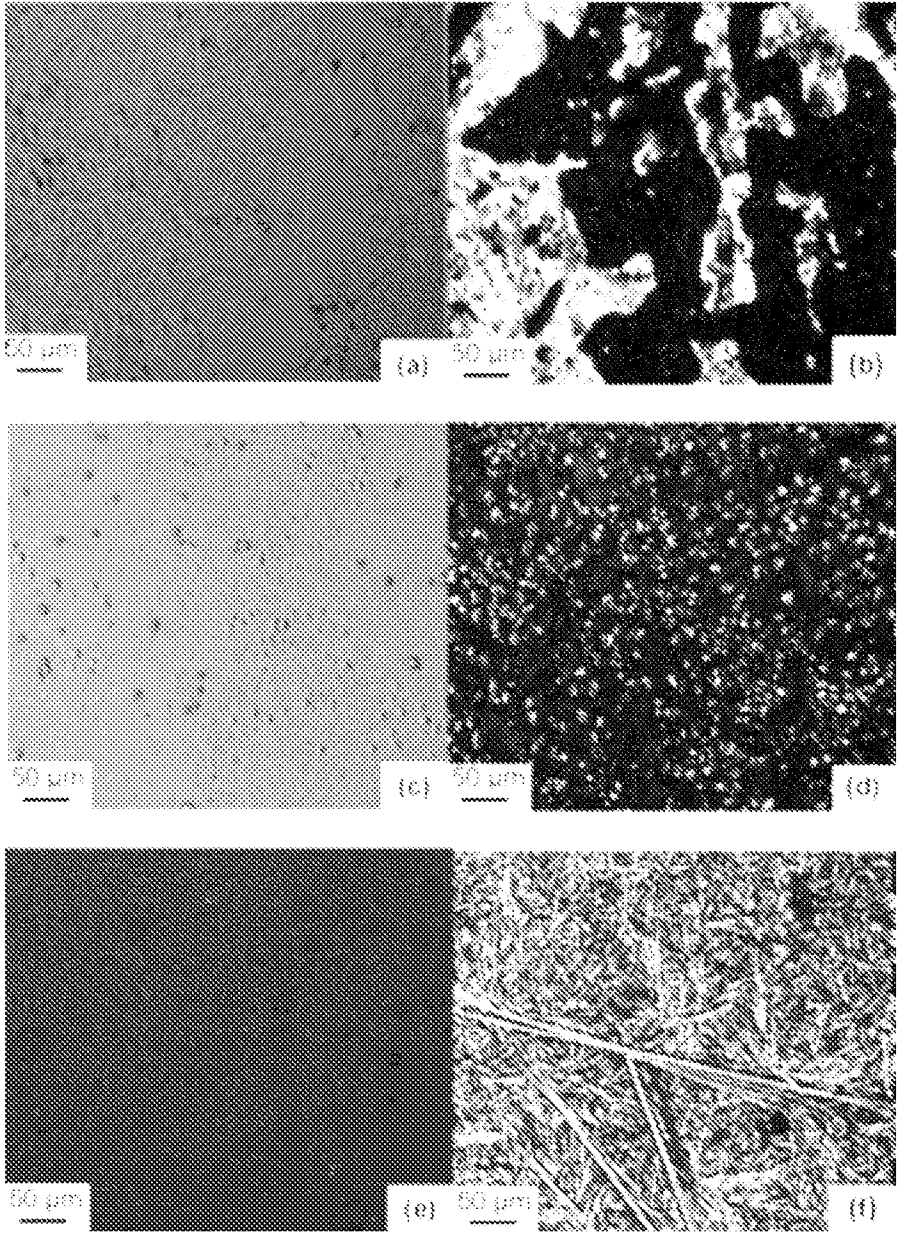
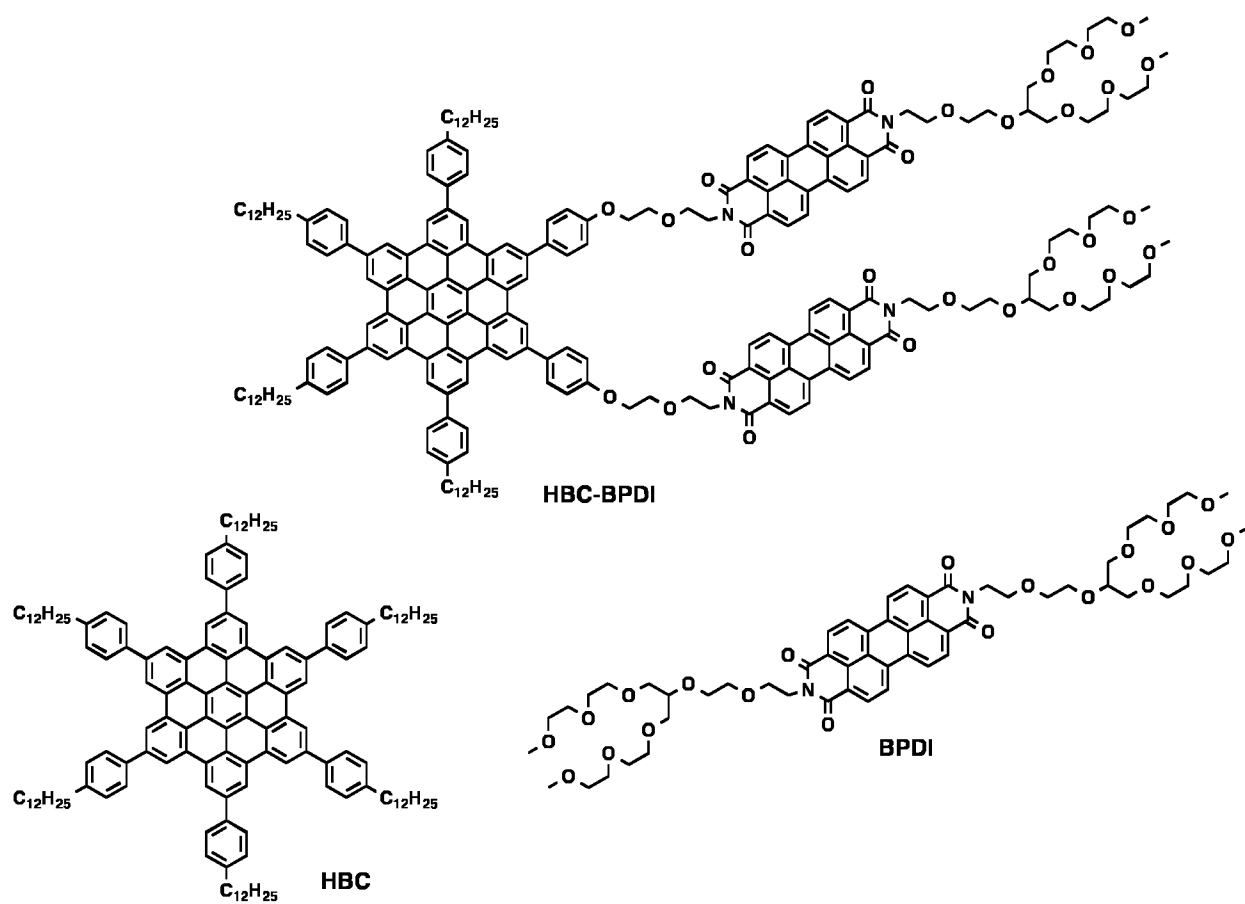


Figure 3



INTERNATIONAL SEARCH REPORT

International application No
PCT/GB2016/051644

A. CLASSIFICATION OF SUBJECT MATTER		
INV.	C09K11/06 C09K19/54	C07C43/20 C09K19/34 C09K19/44 C09K19/52
ADD.		
According to International Patent Classification (IPC) or to both national classification and IPC		
B. FIELDS SEARCHED		
Minimum documentation searched (classification system followed by classification symbols) C09K C07C		
Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched		
Electronic data base consulted during the international search (name of data base and, where practicable, search terms used) EPO-Internal		
C. DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	RICHARD W DATE AND DUNCAN W BRUCE: "Shape Amphiphiles: Mixing Rods and Discs in Liquid Crystals", JOURNAL OF THE AMERICAN CHEMICAL SOCIETY, AMERICAN CHEMICAL SOCIETY, US, vol. 125, no. 30, 30 July 2003 (2003-07-30), pages 9012-9013, XP002674882, ISSN: 0002-7863, DOI: 10.1021/JA0357947 [retrieved on 2003-07-02]	1-19,21, 24,25, 28,29
A	chart 2; page 9012, lines 46-50 page 9013, lines 19-27 ----- -/-	20,22, 23,26, 27,30,31
☒ Further documents are listed in the continuation of Box C. ☐ See patent family annex.		
* Special categories of cited documents : "A" document defining the general state of the art which is not considered to be of particular relevance "E" earlier application or patent but published on or after the international filing date "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified) "O" document referring to an oral disclosure, use, exhibition or other means "P" document published prior to the international filing date but later than the priority date claimed "T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art "&" document member of the same patent family		
Date of the actual completion of the international search 18 August 2016		Date of mailing of the international search report 29/08/2016
Name and mailing address of the ISA/ European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Fax: (+31-70) 340-3016		Authorized officer Pöttsch, Robert

INTERNATIONAL SEARCH REPORT

International application No

PCT/GB2016/051644

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	Holger Bengs: "Induction and Variation of Discotic Columnar Phases through Doping with Electron Acceptors", 1 June 1990 (1990-06-01), XP055296376, Retrieved from the Internet: URL: http://onlinelibrary.wiley.com/store/10.1002/adma.19900020306/asset/19900020306ftp.pdf?v=1&t=is0cwruc&s=af7e92100fdec10ba8fdc168220055543a39c55e [retrieved on 2016-08-18] figure 1; compounds 1,5 -----	1-31
A	DEBIJE M G ET AL: "Dramatic increase in charge carrier lifetime in a liquid crystalline perylene bisimide derivative upon bay substitution with chlorine", JOURNAL OF MATERIALS CHEMISTRY, ROYAL SOCIETY OF CHEMISTRY, GB, vol. 15, 25 January 2005 (2005-01-25), pages 1270-1276, XP002526946, ISSN: 0959-9428, DOI: 10.1039/B416407E scheme 1; compounds HBC-PhC12 -----	1-31
A	SANTANU KUMAR PAL ET AL: "Triphenylene-based discotic liquid crystals: recent advances", LIQUID CRYSTALS, TAYLOR & FRANCIS, vol. 40, no. 12, 9 November 2013 (2013-11-09), pages 1769-1816, XP001586827, ISSN: 0267-8292, DOI: 10.1080/02678292.2013.854418 [retrieved on 2013-11-09] page 1798 -----	1-31