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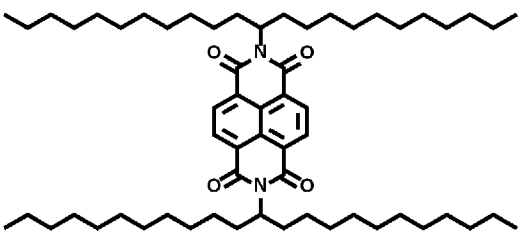
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(54) Title: STABLE SOLVENT-FREE CHARGE TRANSFER LIQUID

(57) Abstract: The present invention provides a solvent-free charge transfer liquid formed by dialkoxynaphthalene as a donor and a new derivative molecule of naphthalenediimide of formula (2) as an acceptor.



(2)

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STABLE SOLVENT-FREE CHARGE TRANSFER LIQUID

FIELD OF THE INVENTION:

The present invention relates to a stable solvent-free charge transfer liquid. More particularly, the present invention provides a solvent-free charge transfer liquid formed by dialkoxynaphthalene as a donor and a new derivative molecule of naphthalenediimide as an acceptor.

BACKGROUND AND PRIOR ART OF THE INVENTION:

Charge transfer (CT) complexes have usually been the solution or gel-based assemblies, where the polarity of the solvent had a dominant role to play. Among the various combinations investigated, dialkoxynaphthalene (DAN) as donor and naphthalenediimide (NDI) as acceptor remain as one of the most successful CT pairs. Mostly, CT interaction between DAN and NDI is exploited in solutions, gels, and crystals and found that 1:1 complex is relatively stable. It has to be noted that the stability of the D-A (1:1) complex is variable with solvent polarity. In addition, the characteristic signal arising from CT interaction is visualized only at relatively high concentrations. Moreover, the processability of these solution-based assemblies without altering the functional features raises major concerns. Hence, a reliable, alternate way of CT complex formation is highly desired.

In molecular systems, studying charge transfer, energy/electron transfer, etc., solvent molecules and thereby polarity of the medium creates a drastic difference in efficiency. In addition, the preferred interaction of solvent with the active molecules through various noncovalent interactions dictates the functional properties to a certain extent. This eventually imposes conditions for the formation of thin films and cocrystals out of D-A combinations from solution. Hence it is meaningful to study D-A interaction in a solvent-free condition that supports CT and provides improved processability without compromising the functional properties for further development.

Recently, organic solvent-free room temperature (RT) liquids have been successfully applied in various areas such as dye-sensitized solar cells, light-emitting diodes, optical lasers, sensors, and as photon upconversion liquids, porous liquids, luminescent/phosphorescent liquids, etc.

It is evident from the literature available to date that an aromatic donor-acceptor charge-transfer interaction in a room temperature solvent-free liquid medium has never been attempted, though the concept of charge transfer liquid will be very much beneficial for processing of optoelectronically active materials in device fabrication.

In order to address these issues, the inventors studied the D-A CT interaction in the solvent-free liquid state.

OBJECTS OF THE INVENTION:

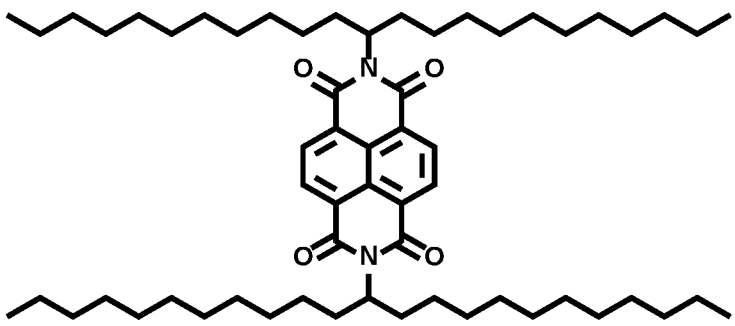
The main objective of the present invention is to provide a solvent-free charge transfer liquid by using a new acceptor molecule.

Another objective of the present invention is to provide a new acceptor molecule and a process for the preparation thereof.

SUMMARY OF THE INVENTION:

Accordingly, the present invention provides a solvent-free charge transfer liquid. More, particularly, an efficient and stable charge transfer liquid formed by a solvent-free liquid dialkoxynaphthalene donor and naphthalenediimide acceptor is provided, wherein the donor: acceptor ratio varies from 1:0.001 to 1:1.

In yet another embodiment, the present invention provides a new acceptor compound of Formula (2) and process for the preparation thereof; wherein the process comprises of reacting Naphthalene-1,4,5,8-tetracarboxylic dianhydride of formula (A) and 2.5 equivalents of tricosan-12-amine in DMF and stirring for 6 h at 140°C to afford compound of formula 2;



(2)

The present invention provides a charge transfer complex, which exhibits remarkable stability and retains the characteristic features even at elevated temperatures.

In an embodiment of the present invention, the process for the preparation of CT liquid is provided, wherein the process comprises heating the donor compound of formula **1** and acceptor compound of formula **2** with different equivalents in glass vials (1.5 ml) gently to become a homogeneous mixture and stirring well with spatula wherever needed to afford CT liquid.

ACRONYMS USED TO DESCRIBE THE INVENTION:

CT: charge transfer

RT: room temperature (30°C)

BRIEF DESCRIPTION OF THE DRAWINGS:

Figure 1: a) Photographs of compound **1** (liquid at 30°C) and compound **2** (molten at 50 °C) under visible light. b) Absorption spectra of neat samples of **1** and **2**.

Figure 2: Absorption spectral variation of compounds **1**+**2** CT liquid with an increasing amount of compound **2** from 0.001 to 0.01 equivalents; inset shows the corresponding photographs of CT liquids of compound **1** and compound **2**.

Figure 3: Absorption spectral variation of compounds **1**+**2** CT liquid with an increasing amount of compound **2** from 0. 2 to 1.0 equivalents in the solvent-free condition.

Figure 4: Photographs of the compounds **1**+**2** CT liquids with varying D-A ratio stored at three different temperatures, a) -20 °C (freezer), b) 30 °C (RT) and c) 90 °C (oven) for two months.

Figure 5: Photographs of the compounds **1**+**2** CT liquids with D:A a) ratio of 1:1 (left) and 1:0.001 (right) upon heating from 30 to 150 °C in air. b) Eluting as a single band in column chromatography separation.

Figure 6: Comparison of CT complex within solvent-free (neat) and solvent present conditions and at the higher temperature

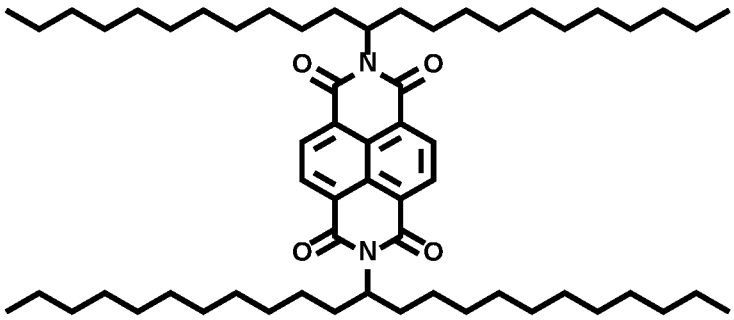
Figure 7: a) DSC thermograms in the heating trace of compounds **1**+**2** CT liquids with varying amounts of compound **2** at a scanning rate of 10 °C/min. b) TGA analysis of compounds **1**,**2** and compounds **1**+**2** (1:1) CT liquid.

Figure 8: Chemical structures and photographs of the CT liquids of compound **3**, compound **4**, compounds **1**+**4** and compounds **2**+**3** with varying D-A ratio

DETAILED DESCRIPTION OF THE INVENTION:

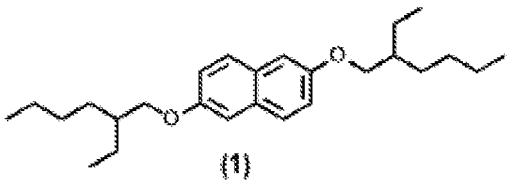
The invention will now be described in detail in connection with certain preferred and optional embodiments, so that various aspects thereof may be more fully understood and appreciated.

Accordingly, to accomplish the objective of providing a stable, solvent-free CT liquid, the present invention provides a new compound, 2,7-di(tricosan-12-yl)benzo[lmn][3,8]phenanthroline-1,3,6,8(2H,7H)-tetraone of formula 2, which serves as an acceptor in the CT liquid of the present invention.



(2)

The compound of formula **2** forms a solution in the donor compound of formula **1**, wherein the donor compound of formula **1** is 2,6-bis((2-ethylhexyl)oxy)naphthalene.



(1)

In an embodiment, the charge transfer liquid comprising a donor and an acceptor, wherein the donor: acceptor ratio varies from 1:0.001 to 1:1, is solvent-free.

In an embodiment, an acceptor compound of Formula (**2**) is prepared by the process comprising the steps of:

a) 1,2-Tricosanone, ammonium acetate (NH_4OAc) and sodium cyanoborohydride ($NaBH_3CN$) are dissolved in 40 ml methanol (HPLC grade) and stirred at 30°C for 56 h (3 days) to obtain a clear solution;

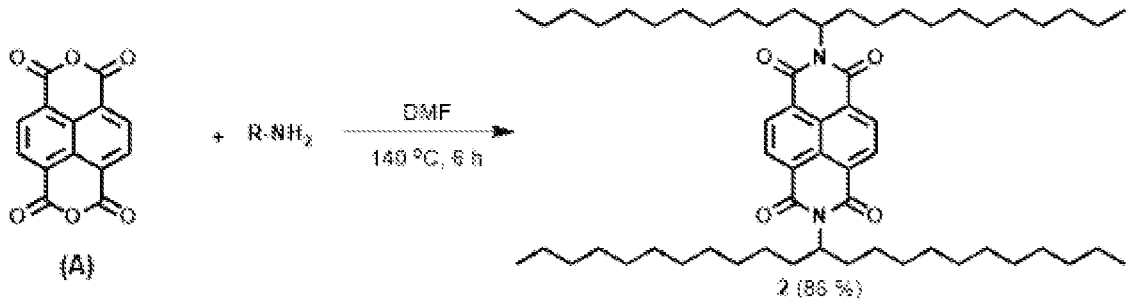
b) quenching the solution of step (a) by adding concentrated hydrochloric acid dropwise, concentrating with the rotatory evaporator, and dispersing the solid obtained in water and adjusting to pH = 10 with KOH;

c) extracting the obtained latex solution using dichloromethane two times, combining the organic layers, drying to remove the solvent under reduced pressure to give pale yellow liquid i.e. the amine in quantitative yield.;

d) suspending 1 equivalent Naphthalene-1,4,5,8-tetracarboxylic dianhydride of formula (A) and 2.5 equivalent of tricosan-12-amine in DMF and stirring for 6 h at 140°C;

e) cooling the reaction mixture after the reaction is complete, quenching by addition of hydrochloric acid, extracting with dichloromethane, drying the organic layer and purifying the product obtained to yield a light brown color solid of the compound of Formula 2. (~ 86%).

The process for the preparation of a compound of formula (2) is depicted below in scheme-1:



Scheme-1

In an embodiment, the donor compound of formula **1** is synthesized by reported processes. [ref. *Chem. Commun.* **2015**, *51*, 8265-8268]

In an embodiment of the present invention, the process for the preparation of CT liquid is provided, wherein the process comprises heating the donor compound of formula **1** and acceptor compound of formula **2** with different equivalents in glass vials (1.5 ml) gently to become a homogeneous mixture and stirring well with spatula wherever needed to afford CT liquid.

The compound of formula **1** or donor compound is a colorless free-flowing liquid at 30°C and the physical nature of **1** is investigated by differential scanning calorimetry (DSC). The glass transition temperature ($T_{g,\ offset}$) around -60°C is observed in the heating cycle, as shown in figure 7a. The RT liquid property of **1** is further characterized by rheology and powder XRD experiments, showing that **1** lacks crystalline ordering and remains as a stable liquid at RT.

Acceptor compound i.e. a compound of formula 2 or an acceptor compound is a low melting solid with melting point (T_m) around at 46 °C. Absorption spectra of the compounds **1** and **2** in DCM (1 X 10^{-5} M) and as neat thin film are recorded. It can be seen that both the compounds absorb in the range of 300 to 400 nm and there is no absorption peak between

400 to 600 nm for both the molecules, as seen in Figure 1. Both **1** and **2** exhibited almost similar absorption features in solution and thin film.

In an embodiment, the donor molecule, compound 1 and acceptor molecule, compound 2 are mixed in ratios ranging from 0.001:1 to 1: 1 of compound 2: compound 1 in neat conditions to form stable CT liquid, as shown in Figures 2 and 3 and example 3. A strong red-brown color of a solvent-free CT complex is formed when molecule 1 is mixed with 2 and the CT formation is evident over the entire range of the ratio of 2:1 specified. The CT complex is a liquid at 30 °C for the range of 0.001:1 to 1:1 of compounds 2:1.

In another aspect of an embodiment, the CT complex formed is evaluated for stability at elevated temperatures. The CT liquid is found to be extremely stable even upon storing at various temperatures. The **1**+**2** CT liquids are stored in three different temperatures; 1) -20 ^{o}C, 2) 30 ^{o}C and 3) 90 ^{o}C.They retain the CT band intensity even after prolonged storage, preferably for two months, as shown in Figure 4.

In one more aspect of an embodiment, the CT liquid is found to be stable at a very high temperature of 150 ^{o}C, as shown in Figure 5a.

The stability of the CT liquid is tested under chromatographic conditions. Separation of the compounds by column chromatography using dichloromethane as the solvent is attempted and it is found that the CT complex is ultra-stable and elute as a single band, as seen in Figure 5b.

Referring to Figure 8, wherein compounds 3 and 4 are listed and a comparison of charge transfer between two liquids at RT is conducted. As observed in compounds **1**+**2** CT liquid, combinations of compounds **1**+**4** and **2**+**3** also form a homogeneous mixture. However, only a very weak CT is observed in all these cases.

The CT is characterized by DSC analysis and the CT remains as a uniform liquid composite with a slight increase in T_m with increasing equivalents of compound **2,** as shown in Figure 7a. TGA shows that CT liquid is very stable and 5% mass loss occurred at 256 ^{o}C, as depicted in Figure 7b.

We have found that solvent-free conditions are ideal to study D–A interactions due to the absence of competitive solvent-solute interaction, thereby delivering a strong D–A interaction. This type of ‘‘green’’ (solvent-free) CT system affords better processability and is expected to deliver optoelectronically active materials suitable for various applications such as tunable emission, organic field-effect transistors, organic light-emitting diodes for flexible electronics, etc. The concept of charge transfer liquid will be highly beneficial for the development of processable optoelectronically active materials.

EXAMPLES:

Following examples are given by way of illustration and therefore should not be construed to limit the scope of the invention.

Example 1: Synthesis of a compound, 2,6-bis((2-ethylhexyl)oxy)naphthalene, of Formula (1):

Potassium carbonate 8.6 g (5 eq.) was suspended in 50 ml of acetonitrile and sonicated for 30 min followed by degassing N_2 for 30 minutes. 12 g (5 eq.) of 2-ethylhexyl bromide was added into the above reaction mixture. Separately 2 g (1 eq.) of 1,5-dihydroxynaphthalene suspended in acetonitrile and added via a dropping funnel to the reaction mixture. After completion of the addition, the reaction mixture was refluxed (90 ^{o}C) under N_2 for 24h. The progress of the reaction was monitored by TLC. After completion of the reaction, the reaction mixture was filtered, the filtrate collected and the solvent removed under reduced pressure. The residue obtained was dissolved in chloroform and washed with 1M hydrochloric acid, water, and brine. The organic layer was dried over sodium sulfate and the solvent was removed under reduced pressure. The crude product was purified by silica gel column chromatography by using 10% dichloromethane in petroleum ether as eluent. The combined organic layer was concentrated under reduced pressure and dried under vacuum to yield a colorless liquid (3.94 g, 82%).

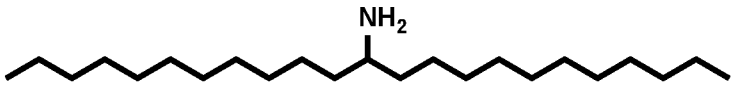
^{1}H-NMR (400 MHz, $CDCl_3$, 25 ^{o}C): δ = 7.63 (2H, d, J = 8.7 Hz), 7.14-7.12 (2H, dd, J = 8.7 and 2.26 Hz), 7.10 (2H, d, J = 2.26 Hz), 3.95-3.93 (4H, m), 1.79 (2H, m), 1.55-1.38 (8H, m), 1.37-1.34 (8H, m), 0.98-0.91 (12H, m) ppm.

^{13}C-NMR (100 MHz, $CDCl_3$, 25 ^{o}C): δ = 155.76, 129.66, 127.91, 119.24, 106.88, 70.55, 39.41, 30.60, 29.11, 23.94, 23.06, 22.9, 14.09, 11.14 ppm.

MALDI-TOF MS: calcd. for $C_{26}H_{40}O_2$ = 384.6040, found 384.2239.

Example 2: Synthesis of compound, 2,7-di(tricosan-12-yl)benzo[lmn][3,8]phenanthroline-1,3,6,8(2H,7H)-tetraone of formula 2:

A) Synthesis of Tricosan-12-amine:



1,2-Tricosanone, ammonium acetate (NH_4OAc) and sodium cyanoborohydride ($NaBH_3CN$) were dissolved in 40 ml methanol (HPLC grade) and stirred at RT for 56 h (3 days). After a clear solution obtained to the reaction mixture, it was quenched by adding conc. hydrochloric acid dropwise. The solution was then concentrated with a rotatory evaporator. The solid thus obtained was dispersed in 250 ml of water and adjusted to pH = 10 with KOH. The obtained latex solution was extracted by using 150 ml of dichloromethane two times. The combined organic layer was dried over sodium sulfate and the solvent was removed under reduced pressure to give pale yellow liquid in quantitative yield. The amine obtained was used for further steps without purification.

B) Synthesis of compound, 2,7-di(tricosan-12-yl)benzo[lmn][3,8]phenanthroline-1,3,6,8(2H,7H)-tetraone, of formula 2:

Naphthalene-1,4,5,8-tetracarboxylic dianhydride (200 mg, 1 eq.) and tricosan-12-amine (633 mg, 2.5 eq.) were suspended in DMF (20 mL) in 100 ml round bottom flask. The reaction mixture was stirred for 6 h at 140°C. The progress of the reaction was monitored by TLC. After completion of the reaction, the reaction mixture was allowed to cool to RT and it was quenched by the addition of hydrochloric acid (2N, 40 mL). The reaction mixture was extracted with dichloromethane (3 × 200 mL). The organic layer was dried over sodium sulfate and the solvent was removed under reduced pressure. The crude product was purified by silica gel column chromatography by using 50% dichloromethane in petroleum ether as eluent. The combined organic layer was concentrated under reduced pressure and dried under vacuum to yield a light brown color solid (584 mg, 86%).

^{1}H-NMR (500 MHz, $CDCl_3$, 25 ^{o}C): δ = 8.73 (4H, s), 5.21-5.12 (2H, m), 2.24-2.17 (4H, m), 1.87-1.78 (4H, m), 1.33-1.19 (72H, m), 0.85 (12H, t, J = 6.8 Hz) ppm.

^{13}C-NMR (125 MHz, $CDCl_3$, 25 ^{o}C): δ = 164.1, 162.9, 131.3, 130.6, 126.8, 55.2, 32.3, 31.9, 29.6, 29.5, 29.3, 26.9, 22.7, 14.1 ppm.

MALDI-TOF MS: calcd. for $C_{60}H_{98}N_2O_4$ = 911.4540, found 911.5922.

Example 3: **Preparation of liquid CT complex**

The donor compound of formula **1** and acceptor compound of formula **2** with different equivalents in glass vials (1.5 ml) was heated gently to become a homogeneous mixture and stirred well with spatula wherever needed.

CT complex liquid was made as follows:

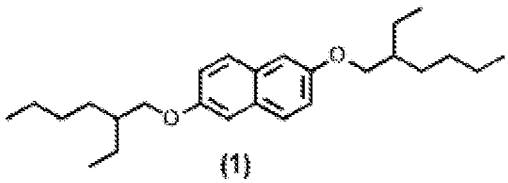
Equivalence of Acceptor compound 2	Weight of Donor compound 1 in mg	Weight of Acceptor compound 2 in mg	Total Weight of CT liquid in mg
0.001	500	1.19	501.19
0.005	495	5.87	500.87
0.01	490	11.61	501.61
0.05	450	53.32	503.32
0.1	405	95.98	500.98
0.2	340	161.15	501.15
0.3	295	209.73	504.73
0.4	260	246.47	506.47
0.5	230	272.54	502.54
0.6	210	298.60	508.60
0.7	190	315.19	505.19
0.8	175	331.78	506.78
0.9	160	341.26	501.26
1.0	150	355.48	505.48

ADVANTAGES OF THE INVENTION

- Stable up to a very high temperature
- Charge transfer efficiency over a large concentration range

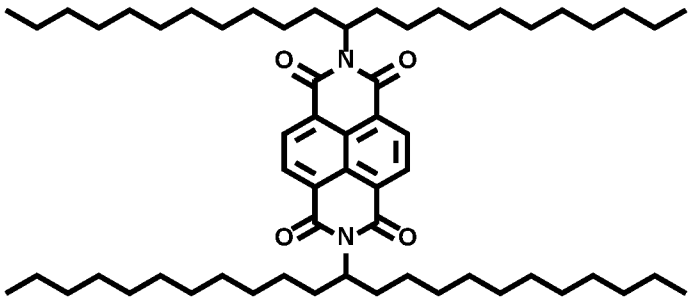
We claim:

1. A charge transfer liquid comprising a donor and an acceptor, wherein the donor: acceptor ratio varies from 1:0.001 to 1:1, and wherein, said charge transfer liquid is solvent-free.
2. The charge transfer liquid as claimed in claim 1, wherein the donor is compound 2,6-bis((2-ethylhexyl)oxy)naphthalene of formula (1);



(1)

3. A compound, 2,7-di(tricosan-12-yl)benzo[lmn][3,8]phenanthroline-1,3,6,8(2H,7H)-tetraone of formula 2; wherein the compound of formula (2) is used as an acceptor in a charge transfer liquid.



(2)

4. The charge transfer liquid as claimed in claim 1, wherein the charge transfer liquid is stable up to 150°C.

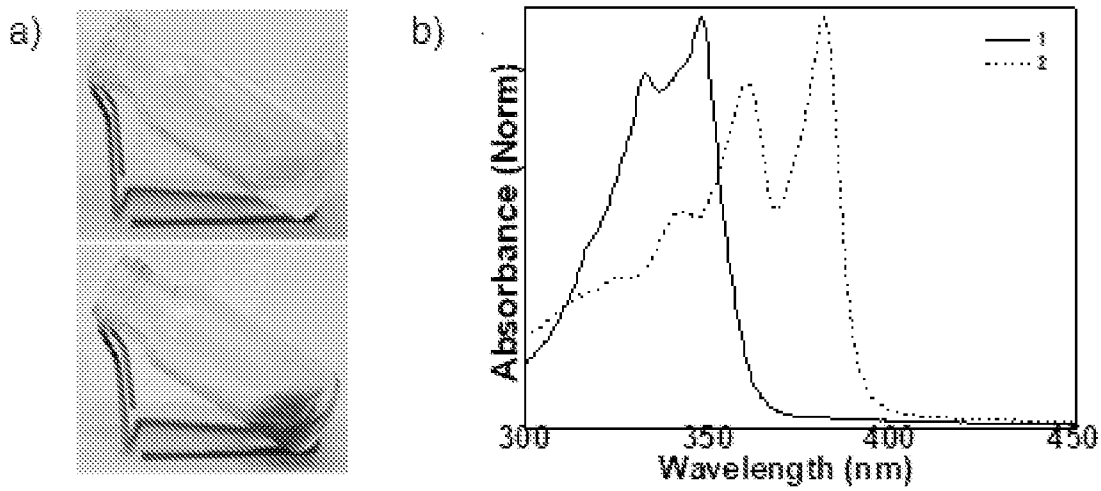


Figure 1

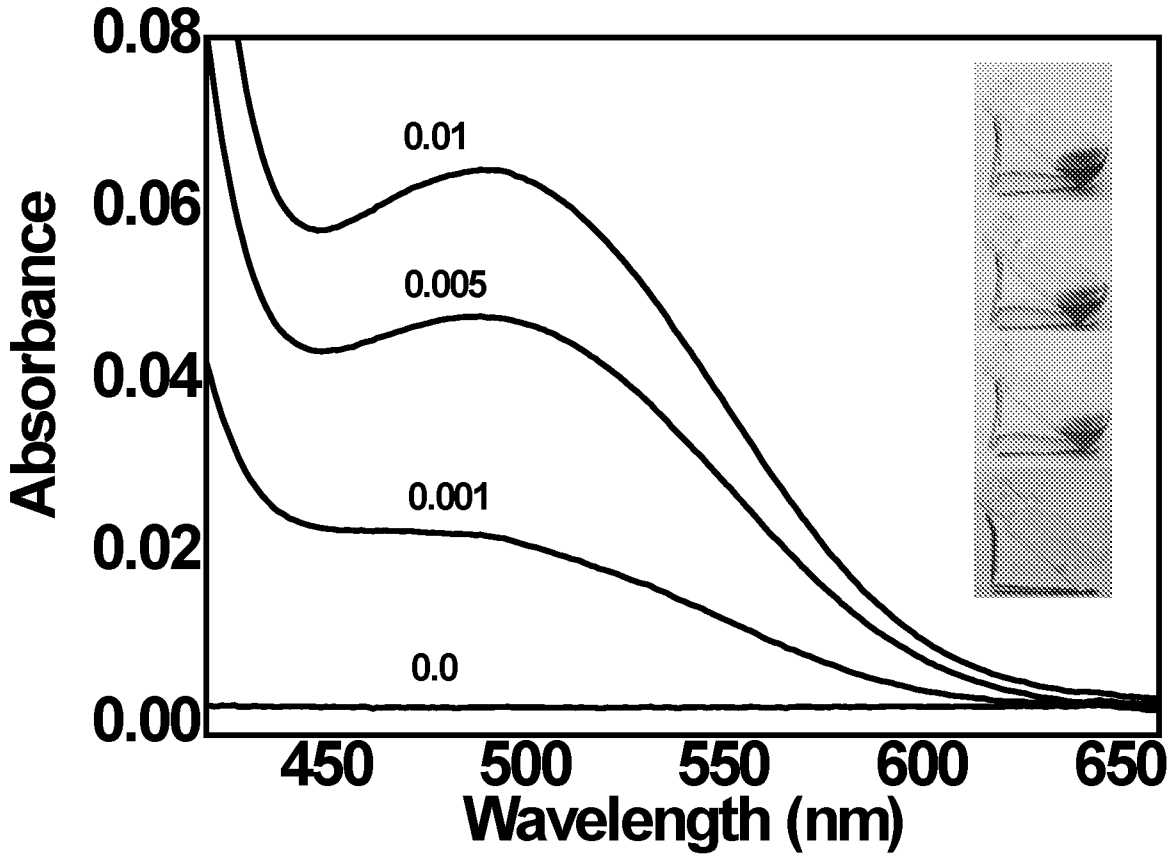

0.08
0.06
0.04
0.02
0.00
Absorbance
0.01
0.005
0.001
0.0
450
500
550
600
650
Wavelength (nm)

Figure 2

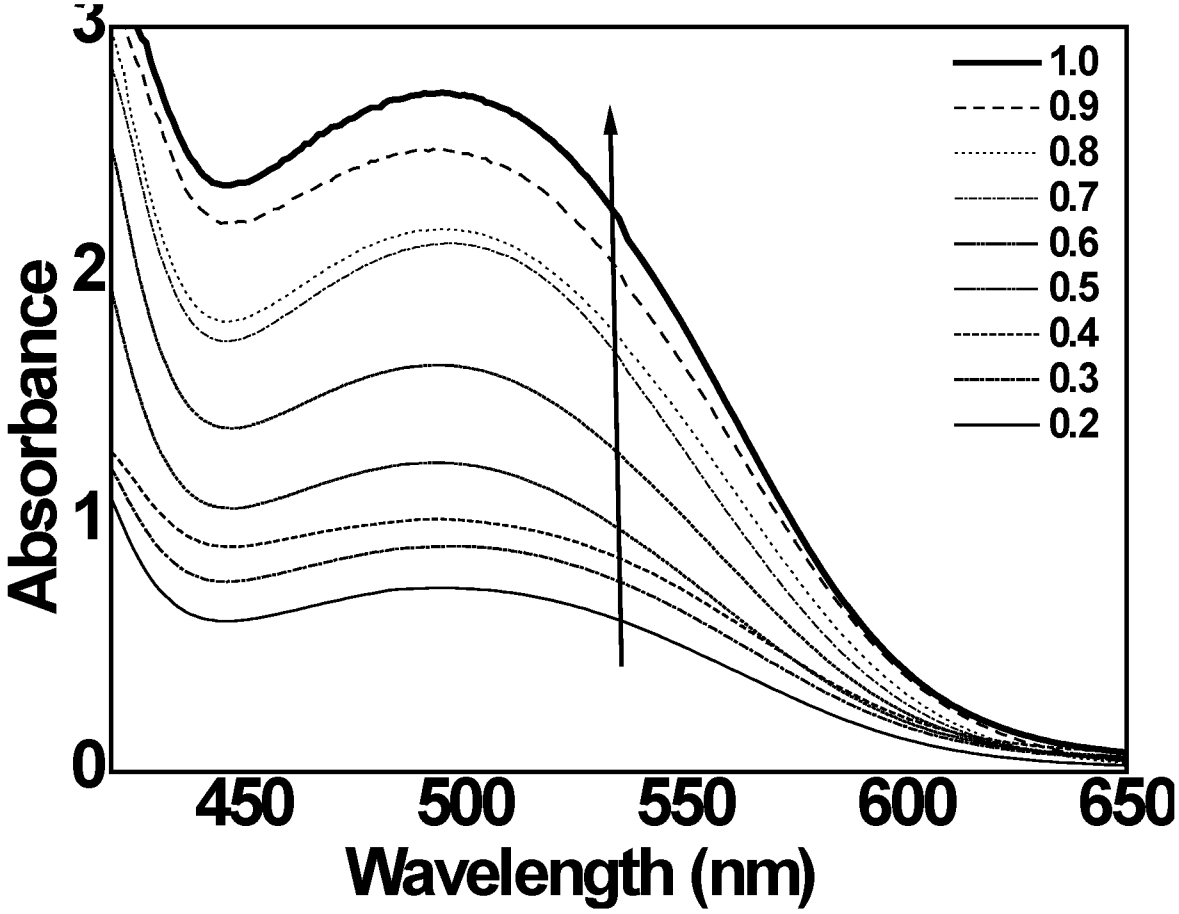

Absorbance
Wavelength (nm)
0
1
2
3
450
500
550
600
650
1.0
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2

Figure 3

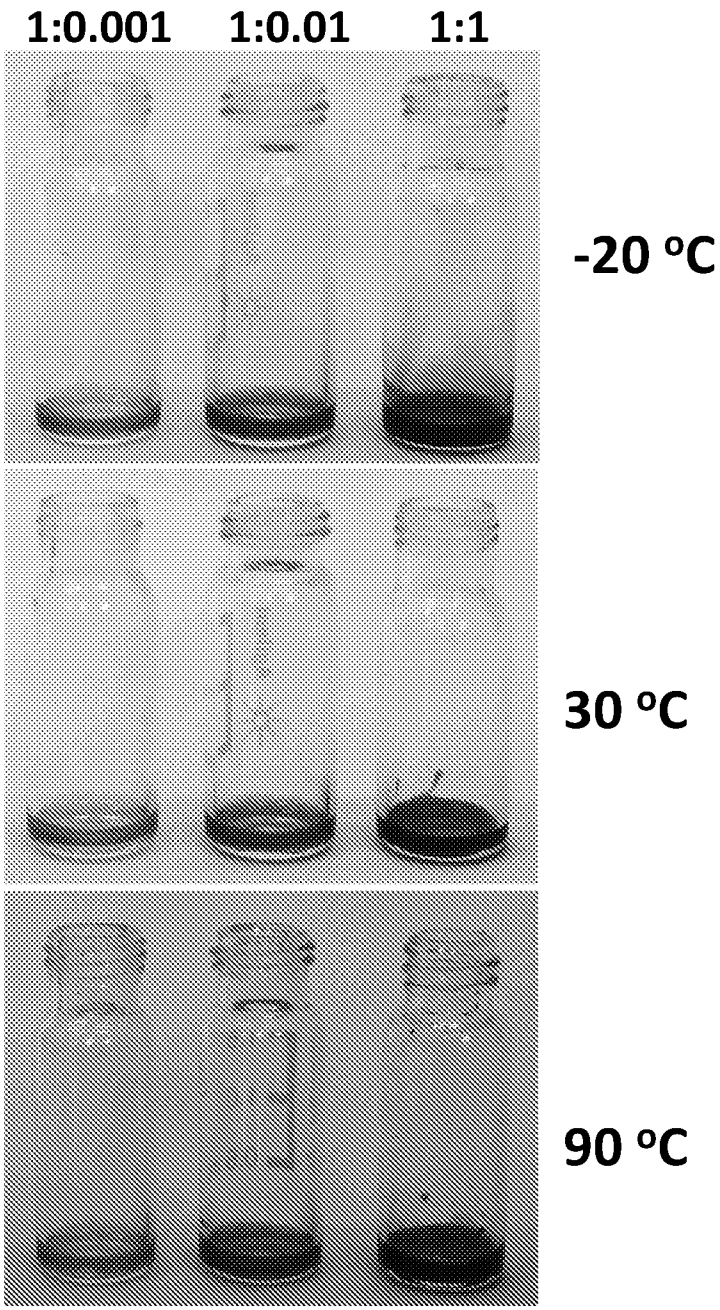


Figure 4

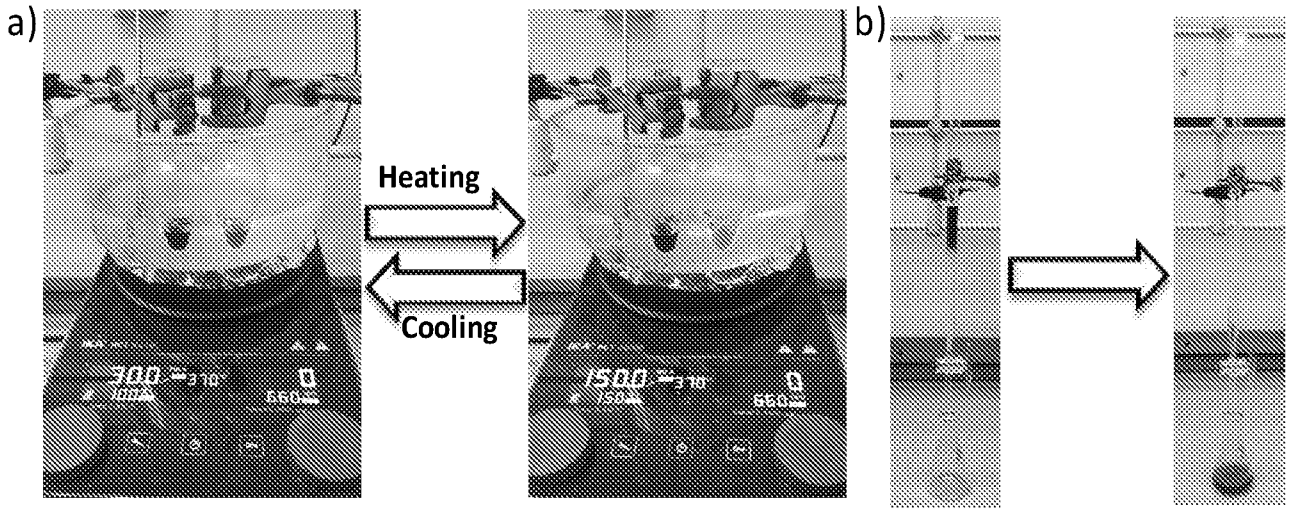


Figure 5

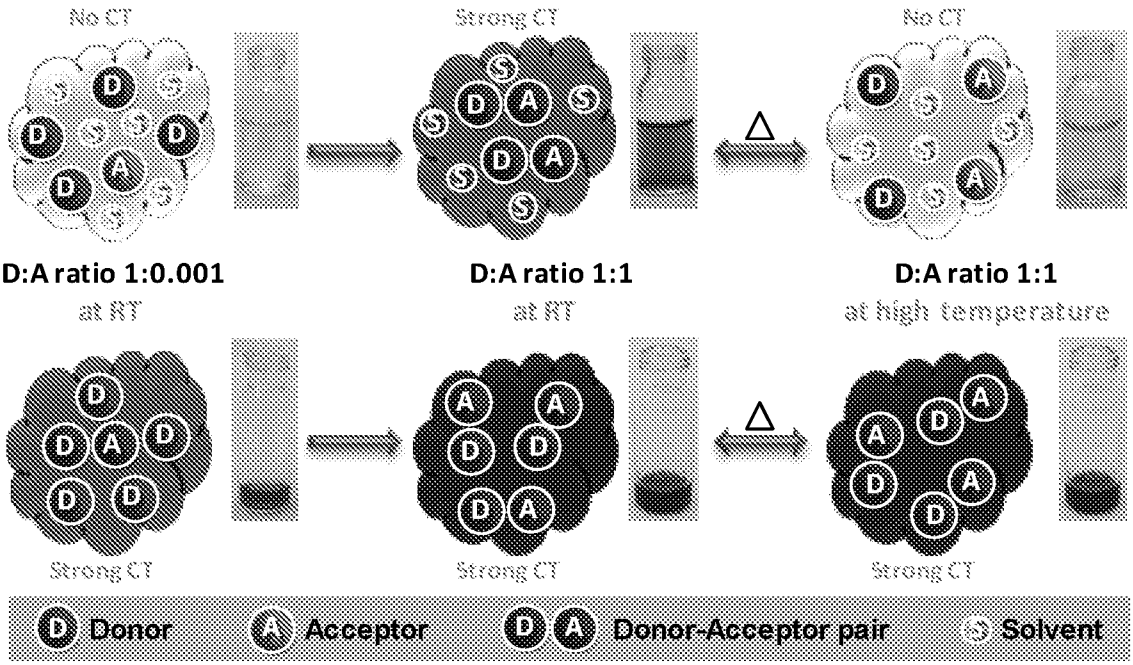

No CT
Strong CT
No CT
D:A ratio 1:0.001
at RT
D:A ratio 1:1
at RT
D:A ratio 1:1
at high temperature
Strong CT
Strong CT
Strong CT
Donor
Acceptor
Donor-Acceptor pair
Solvent

Figure 6

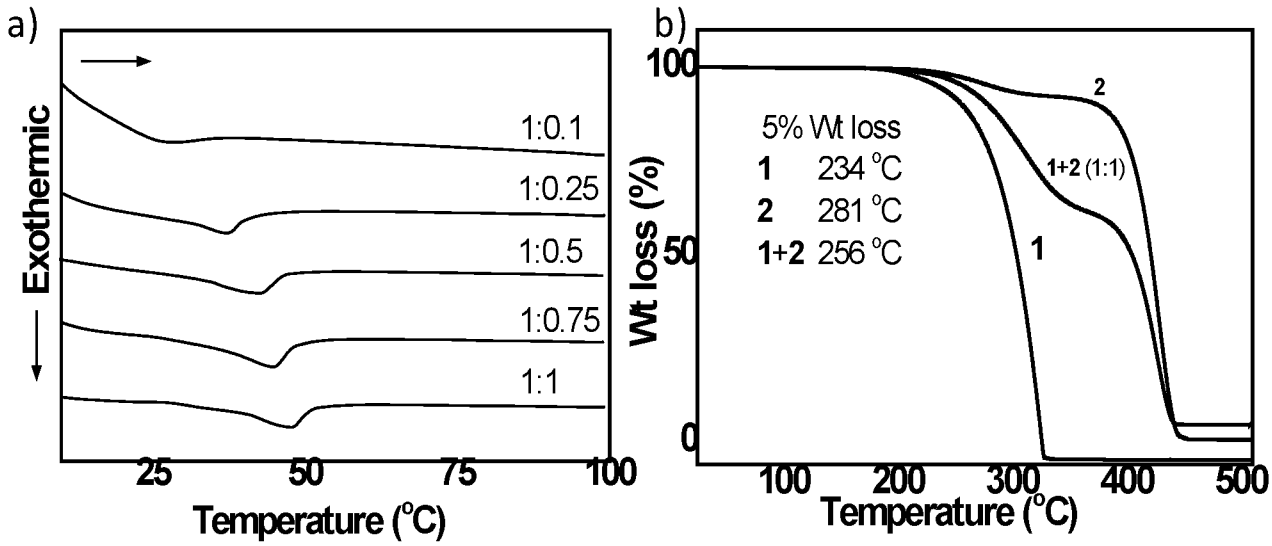

a)
Exothermic
1:0.1
1:0.25
1:0.5
1:0.75
1:1
25
50
75
100
Temperature (°C)
b)
100
50
0
Wt loss (%)
5% Wt loss
1 234 °C
2 281 °C
1+2 256 °C
2
1+2 (1:1)
1
100
200
300
400
500
Temperature (°C)

Figure 7

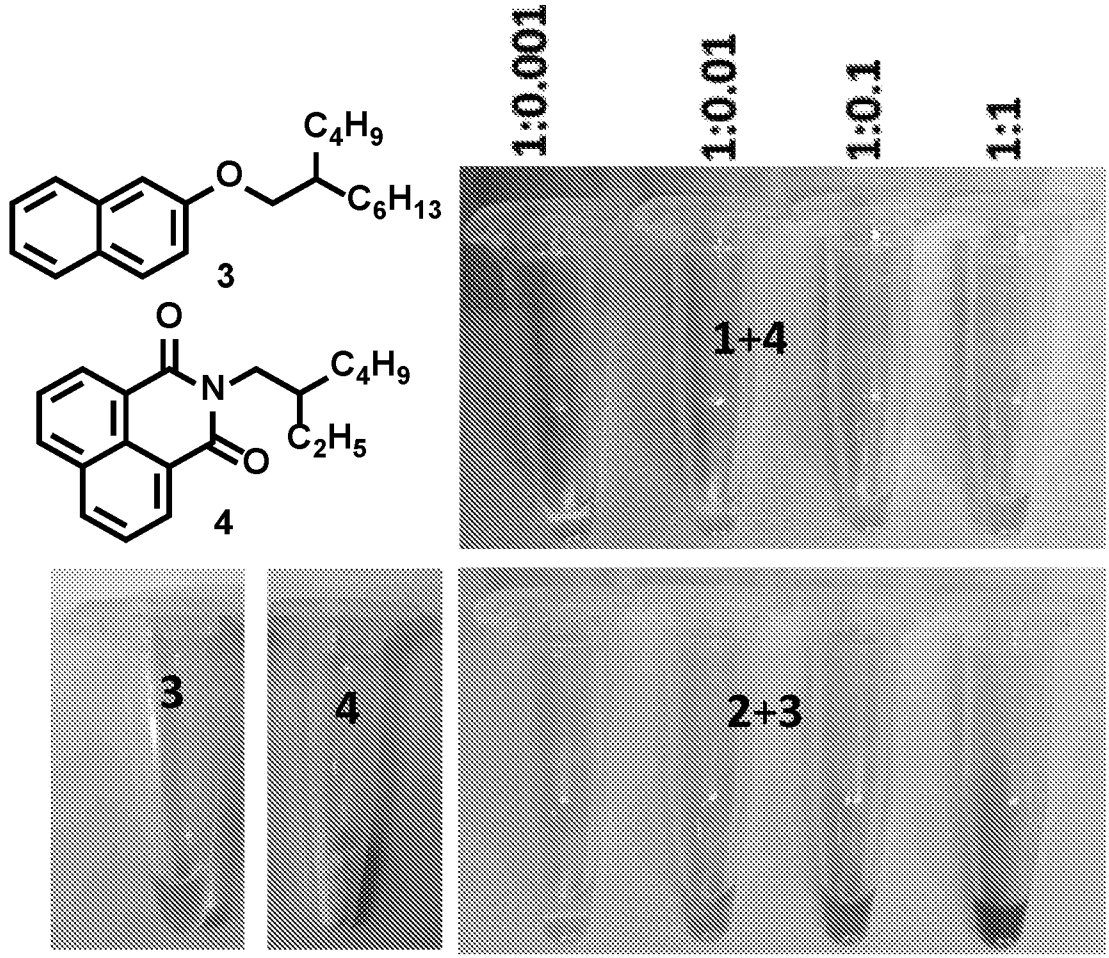


Figure 8

INTERNATIONAL SEARCH REPORT

International application No.
PCT/IN2020/050266

A. CLASSIFICATION OF SUBJECT MATTER
C07D333/22 Version=2020.01

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)

C07D

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)

TotalPatent One, IPO Internal Database

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	Alvey, P. M et al. (2010). A Systematic Study of Thermochromic Aromatic DonorAcceptor Materials. The Journal of Organic Chemistry, 75(22), 7682-7690. doi:10.1021/jo101498b. Whole document	1-4
A	Kobaisi, M. A et al. (2017). The sensitivity of donor-acceptor charge transfer to molecular geometry in DAN - NDI based supramolecular flower-like self-assemblies. Scientific Reports, 7(1). doi:10.1038/s41598-017-15599-9. Whole document	1-4
A	Kemnitz, K et al. (1988). Electron transfer by isolated rhodamine B molecules adsorbed on organic single crystals: a solvent-free model system. The Journal of Physical Chemistry, 92(13), 3915-3925. doi:10.1021/j100324a046. Whole document	1-4

☐ 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
"D" document cited by the applicant in the international application
"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	Date of mailing of the international search report
07-09-2020	07-09-2020
Name and mailing address of the ISA/ Indian Patent Office Plot No.32, Sector 14,Dwarka,New Delhi-110075 Facsimile No.	Authorized officer Arun Kumar Yelshetty Telephone No. +91-1125300200