

Supporting Information

Eco-Friendly Innovation: Three-mode Sun-Actuated Thermotropic Devices Integrating κ -Carrageenan-based Film Doped with *Arundo donax* Leaf-Derived Carbon Dots and 1-Butyl-3-methyl-1-H-imidazolium Chloride

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1. Experimental Section

1.1. Materials

Fresh *A. donax* leaves were collected from the Faculty of Health Sciences of the University of Beira Interior and washed by ultrapure water before use. κ -Cg in powder form (Carrageenan CG-130, Genugel, CP Kelco, 3 and 1.3 wt.% of K^+ and Ca^{2+} , respectively,^{1,2} 1-butyl-3-methyl-1H-imidazolium chloride ([Bmim]Cl) (Acros Organics, 98%), silver nitrate (Sigma-Aldrich, 99.8%), sodium hydroxide (eka chemicals), ammonium hydroxide (Alfa Aesar, 28% NH_3), D-glucose anhydrous (Fisher Chemical), (3-aminopropyl)triethoxysilane (APTS, Sigma-Aldrich), glycerol (Gly, Aldrich, 99%), 30% (w/w) aqueous hydrogen peroxide (Carlo Erba), sulfuric acid (95%, VWR Chemicals), ethanol (96%, Valente e Ribeiro) were used as received. High purity deionized water (H_2O) (type II pure water, using Elix Reference Water Purification System 10 from Millipore) was used in all experiments. Glass slides (56 x 76 mm) was purchased from Destalab (Ref. D100001).

1.2. Preparation of CDs

The CDs obtained through the *A. donax* leaves were prepared according to the procedure described in detail elsewhere.³ The *A. donax* leaves were dried in the oven at 100 °C, and then ground in a coffee mill for 30 s. The cluster of *A. donax* leaves was placed in a quartz crucible inside a muffle furnace and the temperature was increased to 400 °C (or 250, 300 or 350 °C) within 150 min (heating 3 °C/min), and the powder was annealed at 400 °C (or 250, 300 or 350 °C) for 180 min. Subsequently, the crucible was allowed to cool down to room temperature. The carbonization of the greenish powder turned into a dark-grey product. 1.0 g of the black powder and 100 mL ultrapure water were added into the beaker and sonicated for 30 min. The supernatant was filtered using paper filter (PALL ACRO DISC, Japan, pore size 0.185 mm) and the black colour suspension resulting of the powder carbonation at 250, 300 and 350 °C was centrifuged at 10000 rpm for 10 min to remove the large un-dissolved particles. Then, the aggregated particles were removed with microfiltration membrane (0.45 and 0.22 μm), and the filtrate was collected. Finally, the solid-state CDs were obtained by freeze-drying the filtrate followed by lyophilization. The CDs obtained at different temperature of carbonization were denoted as CD(T), where T represents the temperature of the carbonization in °C.

1.3. Deposition of Ag islands

The glass slides were submitted to an acid treatment to remove any contaminants. In short, the glass slides were submerged on a solution of 30% (w/w) aqueous hydrogen peroxide and concentrated sulfuric for 1 h. After removing, the slides were rinsed on water 3 times, and 1 time

with ethanol. For the functionalization of the slides with APTS, a solution of 2% APTS (8 mL APTS, 20 mL water, and 372 mL 95% ethanol) was prepared to submerge the slides. After 5 min for the hydrolyzation of the APTS to occur, the slides were submerged from 10 min. After this time, the slides were removed from the APTS solution and rinsed 3 times in ethanol and dried in the oven at 60 °C overnight.

The deposition of silver islands on APTS-glass substrates was adapted from the procedure described in literature⁴. In general, a silver nitrate aqueous solution was prepared (0.5 g silver nitrate in 60 mL water) under stirring, and an aqueous solution NaOH 5 % (w/v) was added drop by drop until the solution turns brown. After added 2 mL of ammonium hydroxide drop by drop, the solution became uncolored. This final solution was cooled until 4 °C and the glass slides were submerged on the solution. A glucose aqueous solution (0.72 g glucose in 15 mL water) was added to the cooled silver solution and the final mixture was allowed to warm up until room temperature. When the solution turns pale yellow, the glass slides were removed, rinsed 3 times in water, and dried at room temperature. For the annealing process, the glass slides with the Si coating were submitted to a thermal treatment at 250 °C in an oven for 3 h.

1.4. Preparation of polymer electrolytes

The polymer electrolytes (PEs) based on κ -Cg and the guest species [Bmim]Cl, Gly and/or CDs were prepared by the solvent casting technique. A mass of approximately 0.30 g of κ -Cg was dissolved in 15 mL of water and heated under magnetic stirring at 60–70 °C during 60 min for complete dissolution. After dissolution, Gly, [Bmim]Cl and CDs were added to the aqueous κ -Cg solution under stirring at 60 °C for 90 min. The relative proportions of Gly: κ -Cg (wt.%) and [Bmim]Cl: κ -Cg (wt.%) were approx. 50 and 25 or 50, respectively (Table S1, Supplementary Information). The relative proportion CD(T): κ -Cg (wt.) was approx. 0.8 or 2.1. All the above solutions were subsequently transferred to Petri plates, cooled down to room temperature, and dried in an oven at 50 °C for at least 4 days. κ -Cg-based composite films doped with [Bmim]Cl and/or CDs are transparent, homogeneous, and luminescent (Figure S2). κ -Cg-based electrolytes samples were denoted as κ -Cg/Gly-CD(T)-[Bmim]Cl, where κ -Cg represents the matrix, Gly glycerol, CD(T) stands for CDs carbonized at the temperature T in °C. Relevant details of the synthesis procedures are collected in Table S1.

Table S1. Relevant details of the synthesis of the κ -Cg based electrolytes.

Ref. PEs	CG_x I_z CD_w electrolytes						
	κ-Cg m (g); v(H ₂ O) (mL)	Gly m (g) % (m/m)		[Bmim]Cl m (g) % (m/m)		CDs m (mg) % (m/m)	
κ -Cg/Gly	0.3065; 15	0.1620	52	-	-	-	-
κ -Cg/Gly -CD(250)	0.3015; 15	0.1598	53			6.1	2.0
κ -Cg/Gly -CD(300)	0.3050; 15	0.1515	50	-	-	2.5	0.82
κ -Cg/Gly -CD(350)	0.3050; 15	0.1508	50	-	-	7.1	2.1
κ -Cg/Gly-CD(400)	0.3130; 15	0.1542	49	-	-	7.5	2.4
κ -Cg/Gly -CD(250)-[Bmim]Cl	0.2998; 15	0.1501	50	0.1176	40	6.3	2.1
κ -Cg/Gly-CD(300)-[Bmim]Cl	0.3200; 15	0.1550	48	0.1232	40	5.2	1.6
κ -Cg/Gly -CD(350)-[Bmim]Cl	0.2986; 15	0.1610	54	0.1374	46	6.2	2.1
κ -Cg/Gly-CD(400)-[Bmim]Cl	0.3007; 15	0.1601	53	0.1452	48	7.2	2.4

Notes: % (m/m) relatively to the κ -Cg.

1.5. TT assembly.

The assembly of the TTD was performed by drop casting the electrolyte solution on a glass slide followed by a short drying period and close with a second glass slide. In the case of the TTD with silver islands, the second glass slide was doped with the silver islands. The final devices were dried at room temperature for 2 days before the performance evaluation.

1.6. Characterization

1.6.1. CDs characterization

The morphology and the nanostructure of the CDs were analyzed by a high-resolution transmission electron microscope, HR-TEM (JEOL, JEMCXII, Transmission Electron Microscope operating voltage at 200 kV). Prior to the analysis the samples were diluted in H₂O (1.7 mg of CDs in 5 mL of H₂O) and then the samples were applied to a 400-mesh copper grid containing a lacey carbon and an ultrathin carbon support film. A drop of the solution on the grid and then left to dried overnight.

X-ray photoelectron spectroscopy (XPS) was performed by a ESCALAB 250Xi (Thermo Fisher Scientific) using a monochromated AlK α (1486.68 eV) radiation, operated at 220 W, 14.6 kV, spot size 650 μ m. XPS spectra were collected at pass energies of 100 and 40 eV, for survey spectra and individual elements, respectively. The energy step for individual elements was 0.1 eV. Samples were mounted on the sample holder using double-sided adhesive carbon tape. XPS spectra were peak-fitted using Avantage data processing software (Thermo Fisher Scientific) and the Shirley-type background was used. All the XPS peaks are referenced to adventitious carbon C1s, C-C peak at 284.8 eV. Quantification was done using sensitivity factors provided by the Avantage

library. Charge neutralization was achieved with both low energy electron and argon ion Flood guns (< 0.1 eV, 120 μ A and 70 μ A current respectively).

The absolute emission quantum yield values (Φ) of CDs dissolved in water were measured at room temperature using a C9920-02 Hamamatsu system. The method is accurate within 10%.

A Rigaku DMAX III/C diffractometer was used to make a 3–60° (2θ) scan in reflection method (operation voltage of 30 kV and current of 15 mA) using monochromated $\text{CuK}\alpha$ radiation ($\lambda = 1.54 \text{ \AA}$). The solid CDs were deposited on a glass substrate.

The FT-IR spectra's were collected on a ThermoScientific Nicolet iS10: smart iTR in the 4000–600 cm^{-1} range by averaging 32 scans at a resolution of 2 cm^{-1} . The sample (2 mg) was finely ground and mixed with ~ 175 mg of pre-dried potassium bromide (KBr, Merck, spectroscopic grade) in an agate mortar and pressed into pellets. Prior to recording the spectra, the pellet was stored under vacuum for 1 week at room temperature to reduce the levels of adsorbed water. A KBr pellet spectrum was used as reference in absorbance calculations.

UV–visible (UV–Vis) absorption spectrum of the CDs in aqueous solution was recorded using a UV–vis spectrophotometer (Thermo Scientific™ OnePlus, Model N.º 840-341500), using a 10 mm path quartz cuvette.

Fluorescence spectra of the CDs dissolved in water were investigated on UV–vis spectrophotometer (U-4100) using a 10 mm path quartz cuvette. The aqueous solutions were prepared by dissolving 4 mg of the sample in 10 mL of distilled water.

The photoluminescence (PL) and time-resolved PL measurements were performed at RT using a FluoTime 300 spectrometer from PicoQuant (Berlin, Germany), with a modular double grating excitation spectrometer (fitted with a 1200 grooves/mm grating blazed at 330 nm) and a single emission monochromator (fitted with a 1200 grooves/mm grating blazed at 500 nm, reciprocal linear density of 1.4 nm mm^{-1}). The excitation source was a 450 W Xe arc lamp. The emission spectra were corrected for detection and optical spectral response of the spectrofluorimeter and the excitation spectra were corrected for the spectral distribution of the lamp intensity using a photodiode reference detector. The absolute emission quantum yield (Φ) values were measured at room temperature using a system (Quantaury-QY Plus C13534, Hamamatsu) with a 150 W xenon lamp coupled to a monochromator for wavelength discrimination, an integrating sphere as the sample chamber, and a multichannel analyzer for signal detection. The method is accurate to within 10%.

1.6.2. κ -Cg-based electrolytes characterization

The analysis of morphology of the κ -Cg-based electrolytes was performed by scanning electron microscopy (SEM)/Energy dispersive spectroscopy (EDS). The SEM images were obtained at 20 kV on a Hitachi S-3400N type II microscope equipped with a Bruker x-flash 5010 at high vacuum. The sample was coated with gold. Elemental mapping of the samples was performed by EDS X-ray analysis. The acquisition time for a satisfactory resolution and noise performance was 60 s.

A Rigaku DMAX III/C diffractometer was used to make a 3–60 ° (2θ) scan with the reflection method (operation voltage of 30 kV and a current of 15 mA). The hybrid samples were deposited on a glass substrate.

The absolute emission quantum yield values (Φ) were measured at room temperature using a C9920-02 Hamamatsu system. The method is accurate within 10%.

ATR/FT-IR spectra were collected on a ThermoScientific Nicolet iS10: smart iTR, equipped with a diamond ATR crystal. For data acquisition, the κ -Cg-based electrolytes were placed onto the crystal and the spectra were recorded. An air spectrum was used as reference in absorbance calculations. All the spectra were collected at room temperature in the 4000–600 cm^{-1} range by averaging 64 scans at a spectral resolution of 4 cm^{-1} .

Bulk ionic conductivities (σ_i) of the membranes were obtained, during heating cycles, from room temperature to 60 °C by means of an Autolab PGSTAT-12 (Eco Chemie), on a cell GE/PE/GE (where GE stands for 10 mm diameter ion-blocking gold electrodes (Goodfellow, > 99.95%)), secured in a suitable constant volume support, over the frequency range from 65 kHz to 0.5 Hz. Prior to characterization, the κ -Cg-based electrolytes were vacuum-dried at 50 °C for about 48 h and stored in an argon-filled glovebox. The electrode–membrane–electrode assembly was secured in a suitable constant-volume support, which was installed in a Buchi TO 51 tube oven. A calibrated type-K thermocouple placed close to the membrane disk was used to measure the sample temperature with a precision of about 0.2 °C. The κ -Cg-based films demonstrated almost ideal semiconductor behaviour up to 70 °C, and bulk conductivities were extracted in the conventional manner from impedance data by using an equivalent circuit composed of Rb in parallel with Gc, where Rb is the bulk electrical resistance of the electrolyte and Gc is its geometric capacity. The circuit element corresponding to the blocking electrode interface was simulated by a series Cdl element, where Cdl is the double layer capacity. The σ_i was calculated using the expression where Rb is the bulk resistance, d is the thickness, and A is the area of the electrolyte sample. Below 70 °C, the Rb values were obtained by the interception of the imaginary part of the impedance (minimum value of Z'') with the slanted line in the real part of the impedance (Z'). Above this

temperature it was impossible to calculate the σ value because the electrolytes exhibited a random behaviour.

Excitation and emission spectra were recorded using a Fluorolog3® Horiba Scientific spectroscopy (Model FL3-2T) with a modular double grating excitation spectrometer and a TRIAX 320 single emission monochromator, coupled to an R928 Hamamatsu photomultiplier using the front face acquisition mode. A 450 W Xe arc lamp was used as the excitation source. The emission spectra were corrected for detection and optical spectral response of the spectrofluorimeter and the excitation spectra were corrected for the spectral distribution of the lamp intensity using a photodiode reference detector.

1.6.3. TTD evaluation methods

AFM images of the glass with and without Ag were obtained using a Nano-Observer AFM microscope from CS Instruments AFM Microscopes-France, in oscillating mode, with a frequency resonance of 60 kHz and a spring constant of 0.3 N m^{-1} and a super sharp Si HQ:NSC19/ACTA probe. All images were treated by the Gwyddion 2.61 program to enhance the quality of the images using tools for flattening and eliminating line noise as well as a low-pass filter.

The optical characterization of the TTDs was made at different temperatures (23, 40 and 60 °C). For the thermal treatment of the devices was made on a hot stirring plate with the desired temperature and the optical spectra was obtained using a DH Mini, UV-Vis-NIR Light Source from Ocean Optics, the optical transmittance was recorded with a miniature spectrometer, Gamry Spectro-115ETM (350–1000 nm) and Ocean Optics FLAME-NIR 950–1650 nm. The spectra were collected and analyzed using the Oceanview 1.6.7

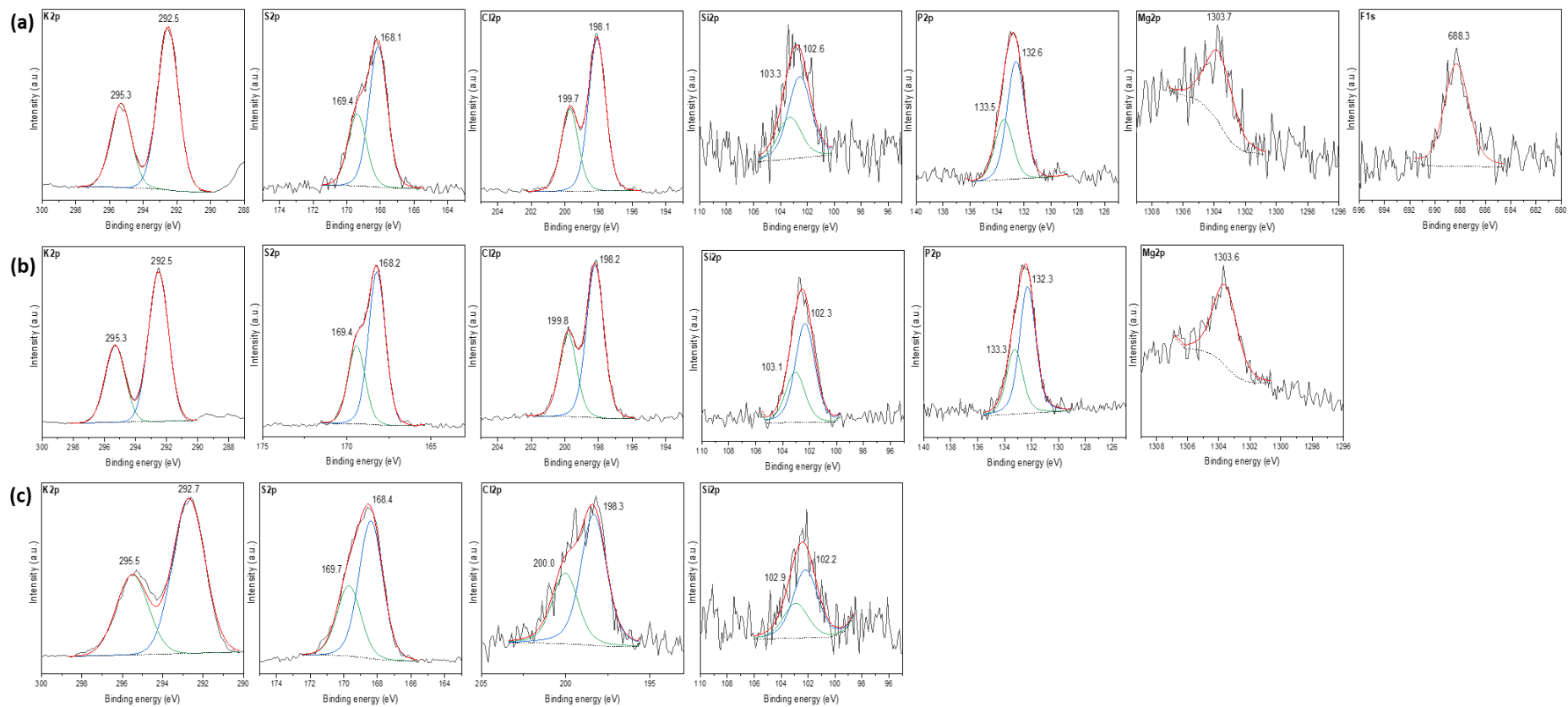


Figure S1. Deconvoluted XPS size distribution of powder CD(T) with T = 250 (a), 300 (b) and 400 (c).

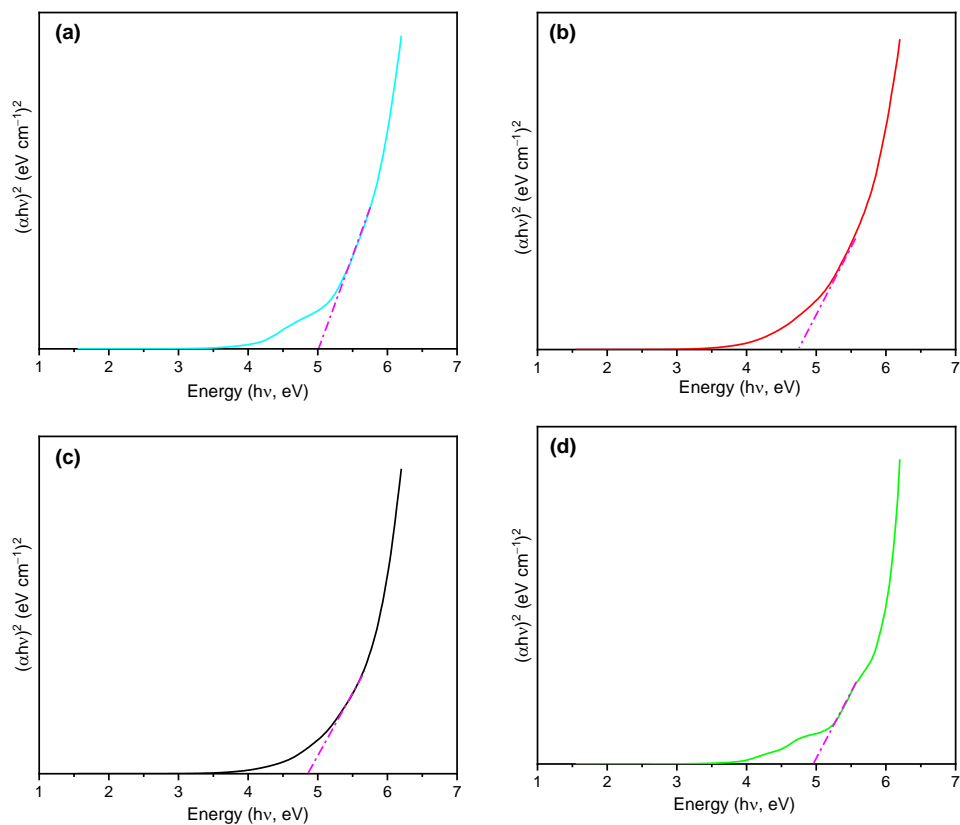


Figure S2. Tauc plots of CD(250) (a), CD(300) (b), CD(350) (c) and CD(400) (d). Here, α stands for the absorption coefficient from the Beer–Lambert equation, h refers to the Plank constant (6.626×10^{-34} J·s) and ν denotes the frequency of the incident photon.

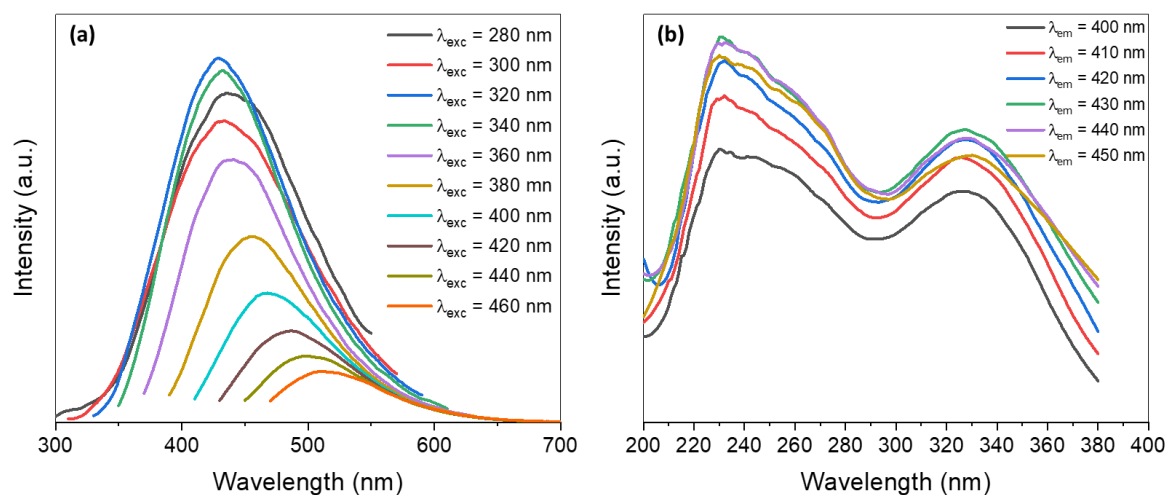


Figure S3. Emission (a) and excitation (b) spectra of CD(350) monitored at different emission wavelengths.

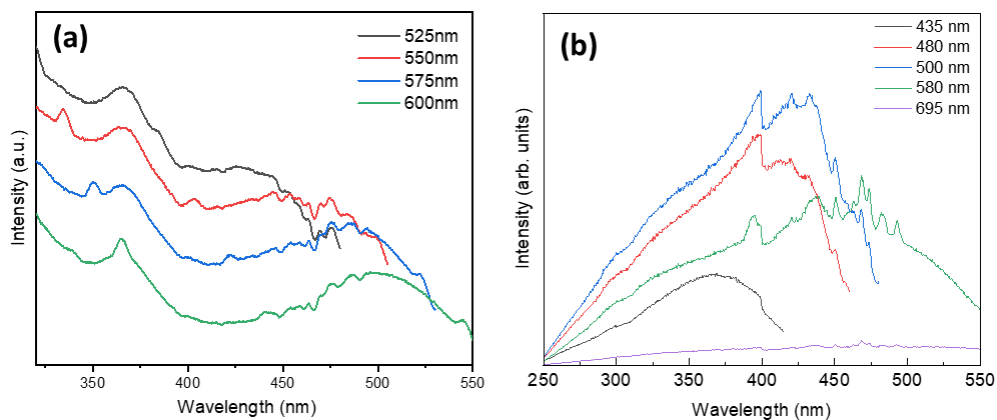


Figure S4. Excitation spectra of CD(300) (a) and CD(400) (b) monitored at different emission wavelengths.

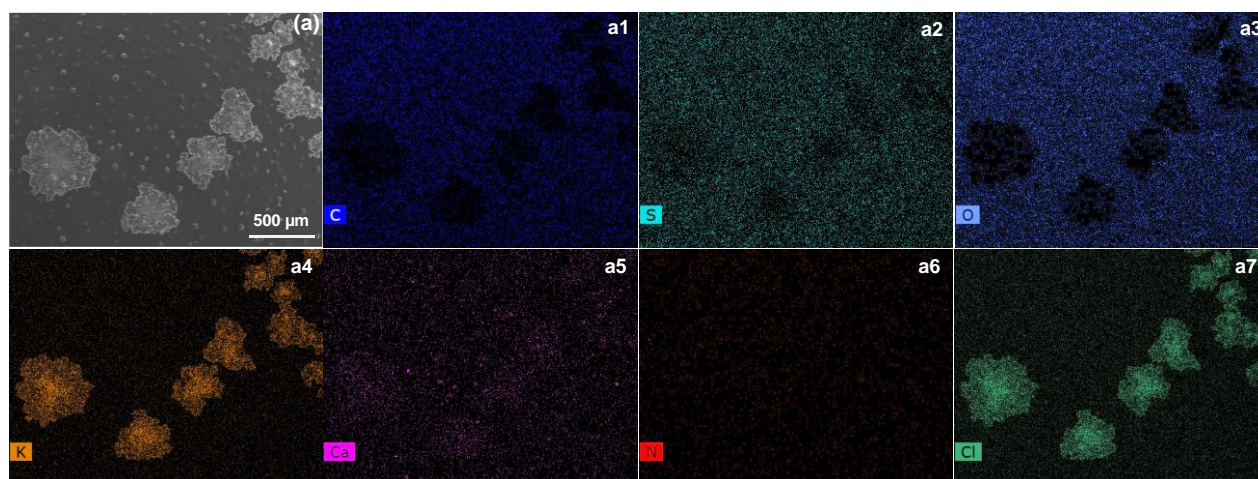


Figure S5. SEM images of κ -Cg/Gly-CD(250) (a) and corresponding EDS mapping images for C (carbon) (a1, b1 and c1), S (sulfur) (a2, b2 and c2), O (oxygen) (a3, b3 and c3), K (potassium) (a4, b4 and c4), Ca (calcium) (a5, b5 and c5), N (nitrogen) (a6, b6 and c6) and Cl atoms (chlorine) (a7, b7 and c7).

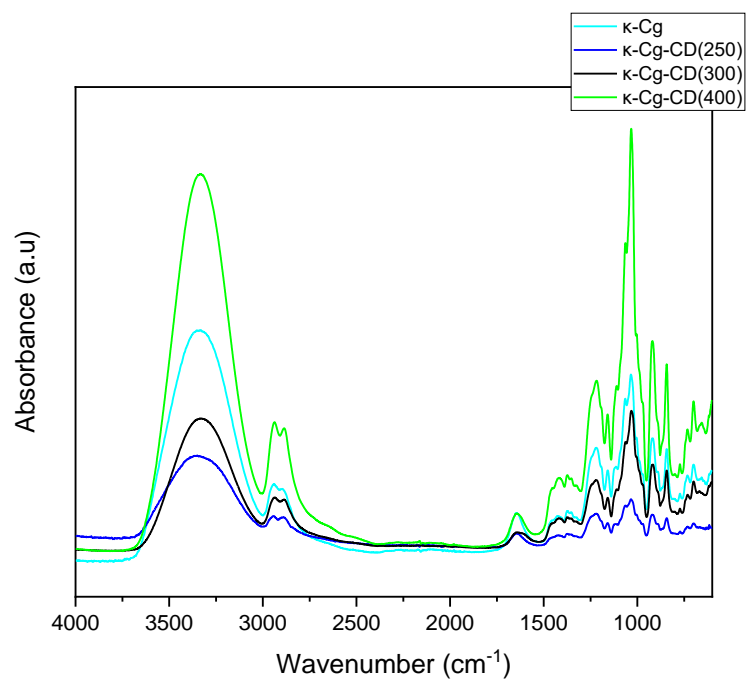


Figure S6. FT-IR spectra of κ -Cg/Gly-CD(T) with T = 250, 300, 350 and 400.

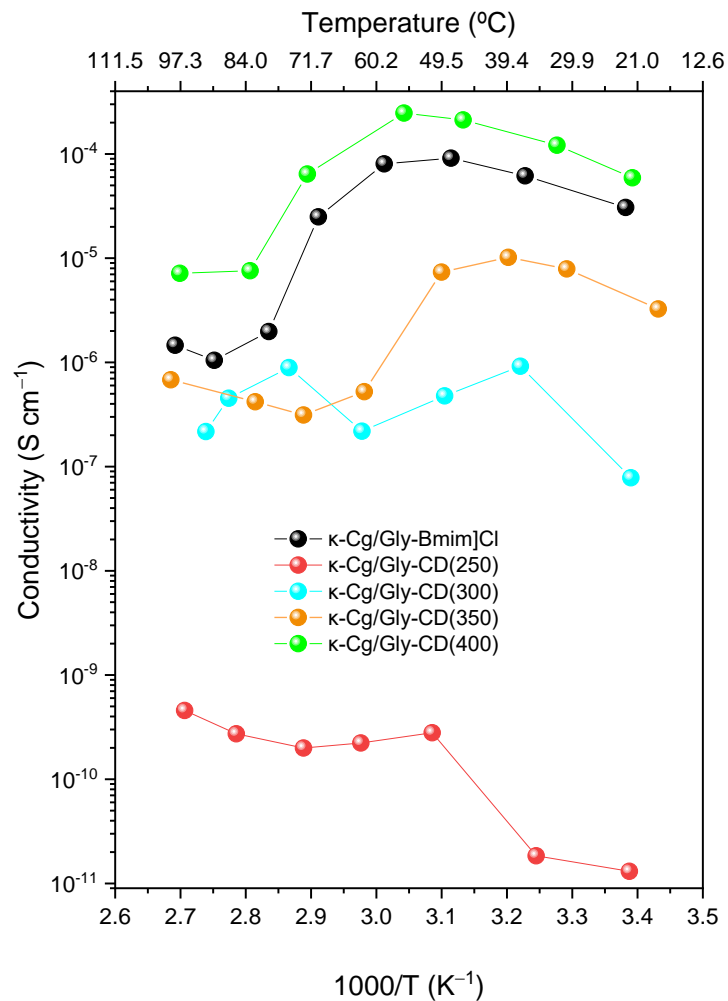


Figure S7. Arrhenius plots of the ionic conductivity.

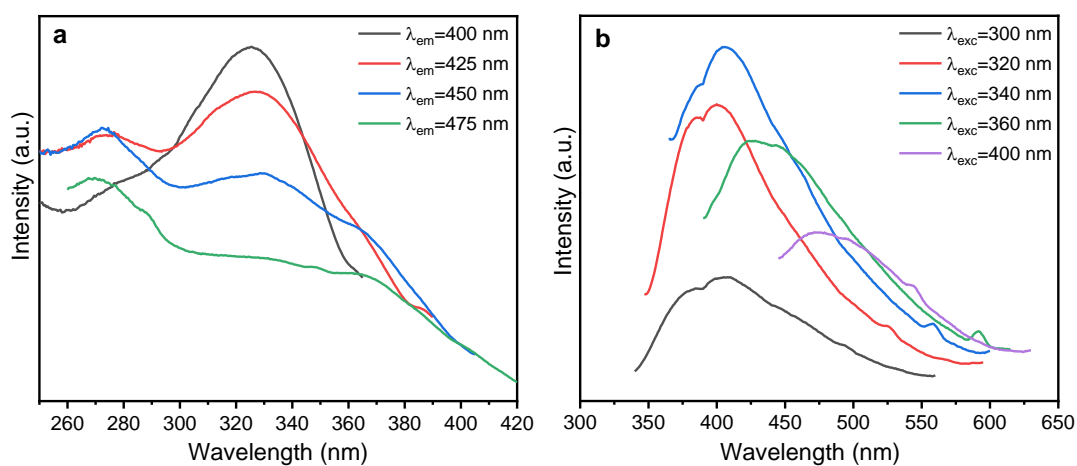


Figure S8. Excitation (a) and emission (b) spectra of the κ -Cg/Gly matrix.

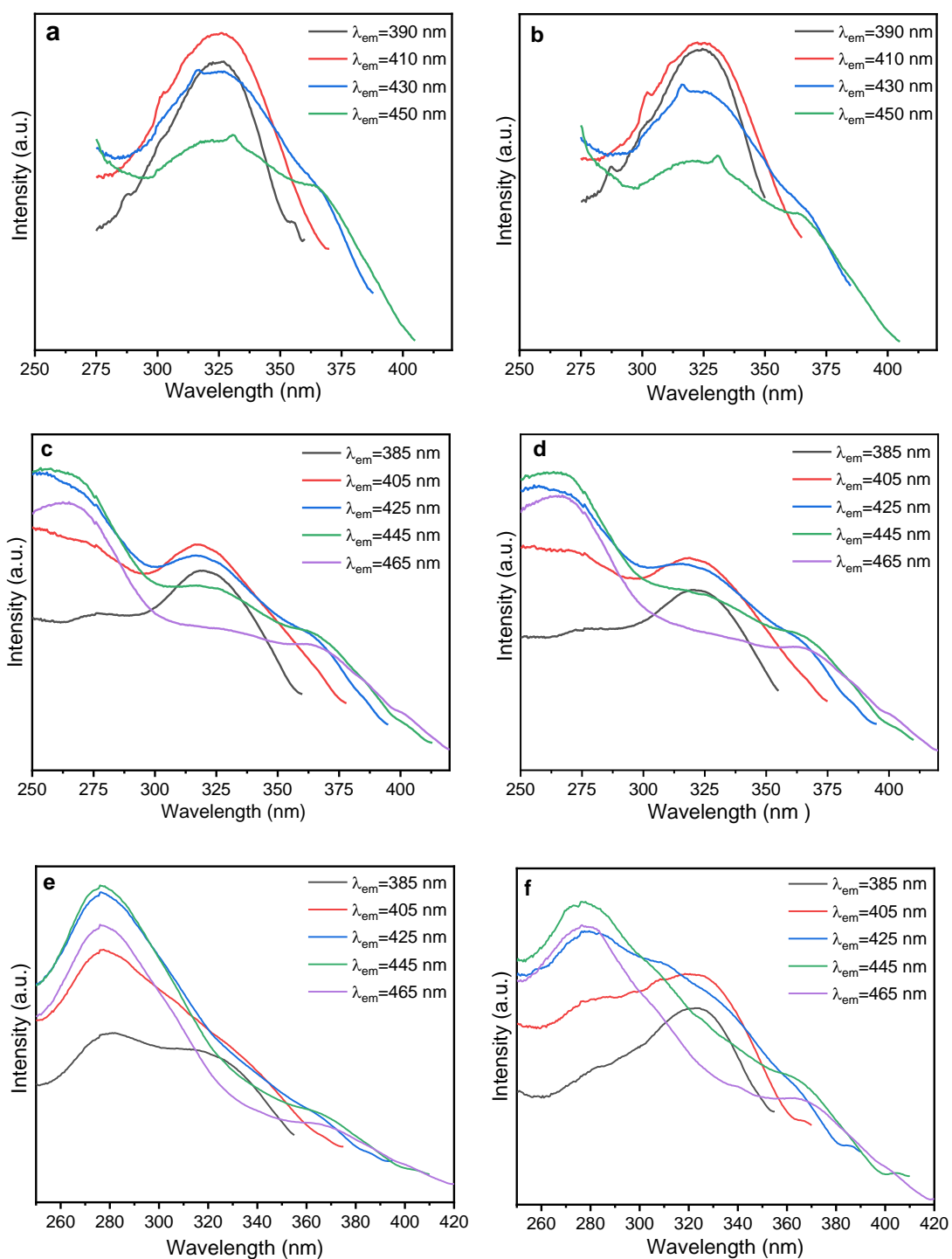


Figure S9. Excitation spectra of κ -Cg/Gly-CD(250) (a), κ -Cg/Gly-CD(250)-[Bmim]Cl (b), κ -Cg/Gly-CD(300) (c), κ -Cg/Gly-CD(300)-[Bmim]Cl (d), κ -Cg/Gly-CD(400) (e) and κ -Cg/Gly-CD(400)-[Bmim]Cl (f) monitored at different emission wavelengths.

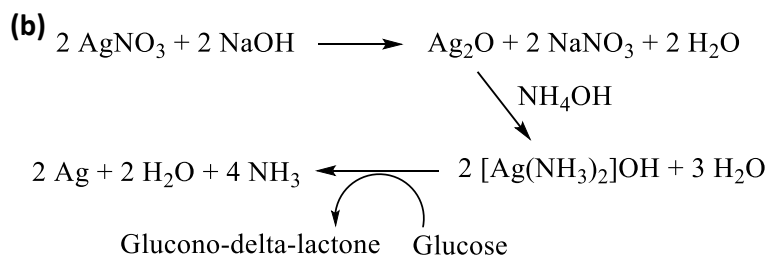
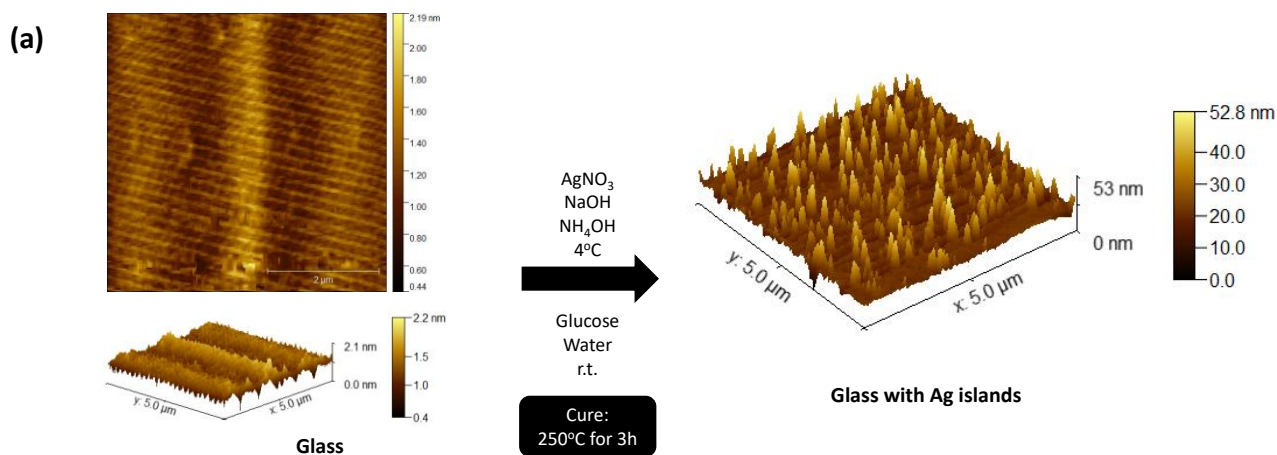


Figure S10. (a) AFM images of the glass slide before (left) and after (right) the chemical deposition of Ag islands. (b) Mechanism representation for the formation of Ag islands.

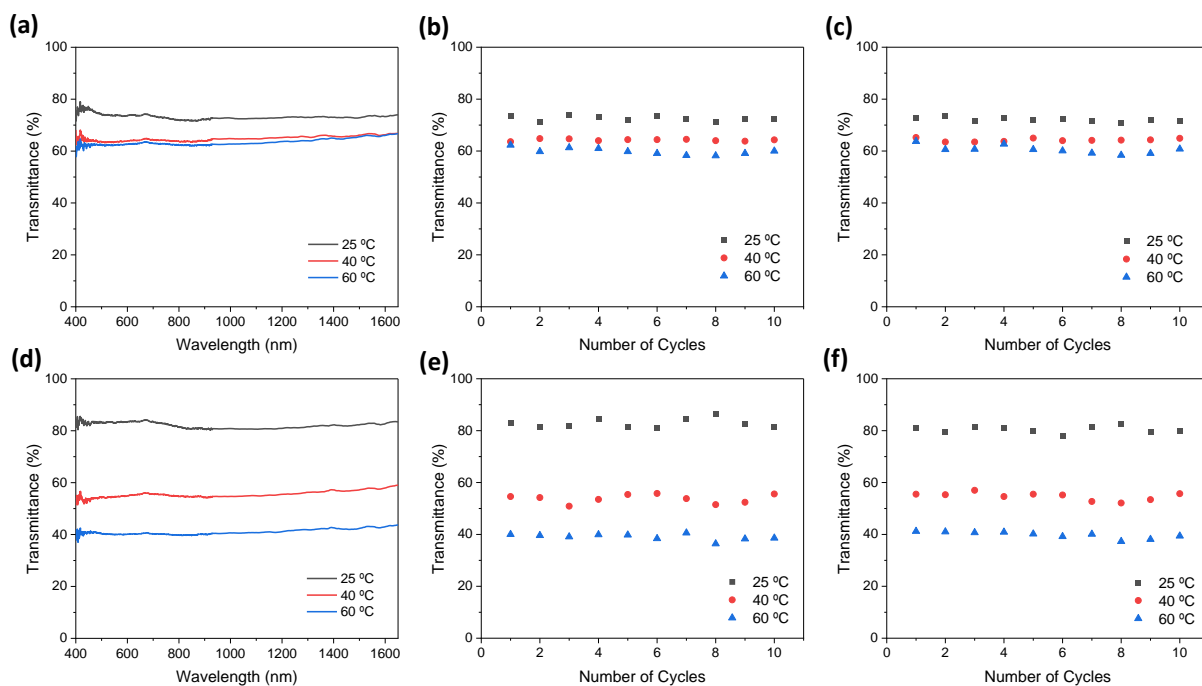


Figure S11. TTDs performance of κ -Cg/Gly-CD(300)-[Bmim]Cl film with glass slides at different temperatures (a), and cycling stability at 550 nm (b) and 1200 nm (c) for the device with simple glass slides. The performance of the device with Ag islands and of κ -Cg/Gly-CD(300)-[Bmim]Cl film at different temperatures (d), and cycling stability at 550 nm (e) and 1200 nm (f).

Table S 2. Peak fitting results obtained after deconvolution of XPS.

Peak	Peak position BE (ev) (fwhm) (Atomic %)				Assignment	Ref.
	CD(250)	CD(300)	CD(350)	CD(400)		
Si2p3	102.57 (2.08) (0.8)	102.34 (1.74) (2.2)	102.33 (1.66) (4.5)	102.19 (2.22) (1.3)	Si–O	5
Si2p1	103.30 (2.07) (0)	103.07 (1.74) (0)	103.06 (1.66) (0)	102.92 (2.23) (0)		
P2p3	132.59 (1.57) (1.44)	132.29 (1.41) (1.76)	132.26 (1.42) (1.5)	-	(P ₂ O ₇) ⁴⁻	6
P2p1	133.49 (1.57) (0)	133.25 (1.41) (0)	133.19 (1.41) (0)	-		
S2p3	168.14 (1.29) (1.4)	168.21 (1.19) (3.7)	168.21 (1.22) (4.45)	168.39 (1.67) (6.5)	SO ₄ ²⁻	7
S2p1	169.38 (1.29) (0)	169.41 (1.19) (0)	169.42 (1.22) (0)	169.69 (1.67) (0)		
Cl2p3	198.09 (1.20) (2.6)	198.20 (1.22) (2.1)	198.25 (1.20) (2.5)	198.33 (1.81) (1.3)	Cl2p _{3/2} of Cl ⁻	8–10
Cl2p1	199.71 (1.2) (0)	199.81 (1.22) (0)	199.89 (1.2) (0)	200.03 (1.82) (0)		
C1s	284.67 (1.37) (31.6)	284.58 (1.30) (19.2)	284.65 (1.44) (16.6)	284.40 (1.22) (8.5)	C=C	7
C1s	285.89 (1.37) (9.2)	285.38 (1.38) (4.4)	285.87 (1.01) (1.4)	285.30 (1.39) (7.8)	C–N (and C–C)	
C1s	-	-	-	286.23 (1.87) (2.6)	C–O	
C1s	287.72 (1.60) (9.3)	287.85 (0.61) (0.6)	287.88 (0.84) (0.87)	288.24 (1.86) (1.8)	O–C=O	
K2p3	292.52 (1.45) (11.7)	292.52 (1.52) (20.1)	292.61 (1.55) (20.3)	292.70 (1.89) (22.6)	K2p _{3/2} of K ⁺	10
K2p1	295.32 (1.45) (0)	295.32 (1.52) (0)	295.41 (1.55) (0)	295.50 (1.89) (0)		
N1s	399.56 (2.03) (3.2)	399.09 (2.47) (2.1)	399.15 (2.56) (1.9)	399.22 (3.57) (1.6)	C–N–C	11
O1s	530.80 (1.85) (18.9)	531.02 (1.91) (35.6)	531.02 (1.94) (33.2)	531.23 (2.10) (38.3)	C–OH/ SO ₄ ²⁻	12
O1s	532.38 (1.88) (8.8)	532.45 (1.91) (7.66)	532.17 (1.94) (12.0)	532.92 (2.12) (7.7)	C=O	
F1s	688.3 (2.3) (0.8)	-	-	-	-CF ₂ CH ₂	
Mg1s	1303.66 (2.16) (0.2)	1303.58 (1.88) (0.41)	1303.58 (2.18) (0.6)	-	Mg metal	

Notes: BE- Binding Energy

Table S 3. Absolute emission quantum yield (Φ) values of CD(*T*) dissolved in water and of κ -Cg/Gly, κ -Cg/Gly-CD(*T*) and κ -Cg/Gly-CD(*T*)-[Bmim]Cl, as a function of the excitation wavelength (λ_{exc}).

λ_{exc} (nm)	CD(250)	CD(300)	CD(350)	CD(400)	
245	<0.01	<0.01	0.01±0.01	0.02±0.01	
330	0.01±0.01	0.02±0.01	0.04±0.01	0.05±0.01	
λ_{exc} (nm)	κ -Cg	κ -Cg/Gly-CD(<i>T</i>)		κ -Cg/Gly-CD(<i>T</i>)-[Bmim]Cl	
		T = 300	T = 400	T = 300	T = 400
280	<0.01	0.01±0.01	0.02±0.01	0.01±0.01	0.02±0.01
330	0.02±0.01	0.03±0.01	0.04±0.01	0.03±0.01	0.04±0.01
380	0.02±0.01	0.02±0.01	-	0.02±0.01	-

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