

Electronic supplementary information

Carbon Based Ionic Liquid Gels: Alternative adsorbents for Pharmaceutically Active Compounds in Wastewater

Carla Rizzo,^a Salvatore Marullo,^a Francesca D'Anna^{a*}

^aDipartimento di Scienze e Tecnologie Biologiche, Chimiche e Farmaceutiche, Sezione di Chimica,
Viale delle
Scienze, Università degli Studi di Palermo, Palermo 90128, Italy

Corresponding author email: francesca.danna@unipa.it

Figure S1. Strain and frequency sweep.	pag. S2
Figure S2. SEM image of graphite/PF₆ xerogel.	pag. S2
Figure S3. Pictures to check the release of graphite/PF₆ gel components.	pag. S3
Figure S4. Turbidity measurements to check release of graphite from gel.	pag. S3
Figure S5. ¹ H NMR spectra in D ₂ O to check the release of graphite/PF₆ gel components.	pag. S4
Figure S6. ¹ H NMR spectra in D ₂ O to check the release of graphite/NTf₂ gel components.	pag. S4
Figure S7. UV spectra of PhAC solutions as function of contact time with gel.	pag. S5
Figure S8. ¹ H NMR spectra to check the release of graphite/PF₆ gel components in desorption process using 2-Me-THF.	pag. S6
Figure S9. ¹ H NMR spectra to check the release of graphite/PF₆ gel components in desorption process using ethyl-lactate.	pag. S6
Figure S10. Pictures to check stability of graphite/PF₆ gel after desorption process.	pag. S7
Figure S11. UV spectra of PhAC mixtures and single PhACs before and after adsorption on gel.	pag. S7
Table S1. RE of graphite/PF₆ for recycling cycles.	pag. S7
Table S2. Desorption of CBZ from graphite/PF₆ after 9 cycles of adsorption in different solvents.	pag. S8
Table S3. RE of graphite/PF₆ as function of CBZ initial concentration.	pag. S8
Table S4. RE of graphite/PF₆ as function of stirring rate.	pag. S8
Table S5. RE of graphite/PF₆ as function of volume of CBZ.	pag. S8
Table S6. RE of graphite/PF₆ with mixtures of PhACs.	pag. S8

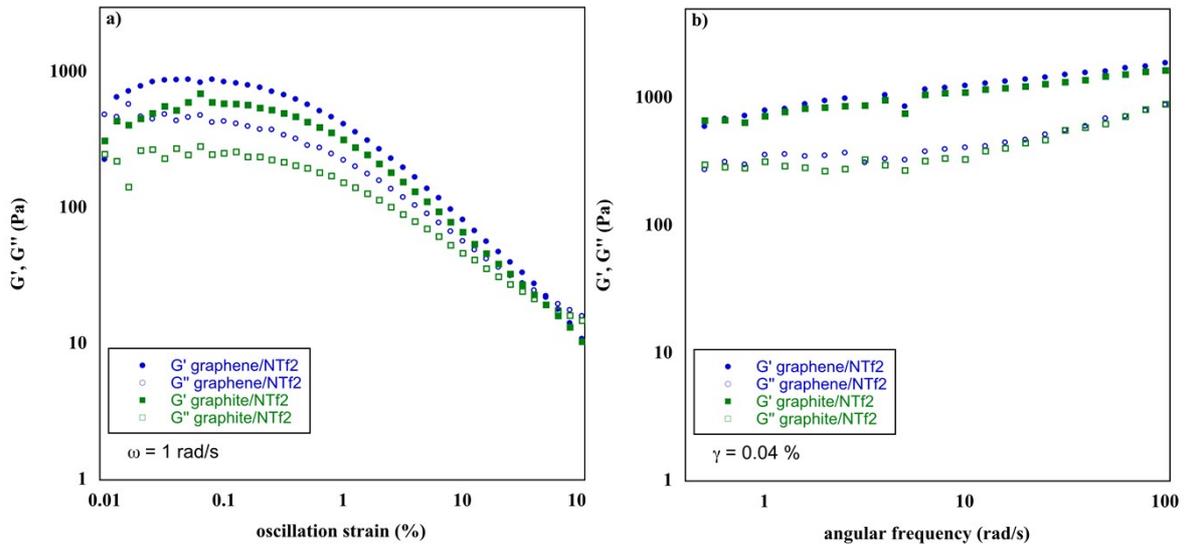


Figure S1. a) Strain and b) frequency sweep.

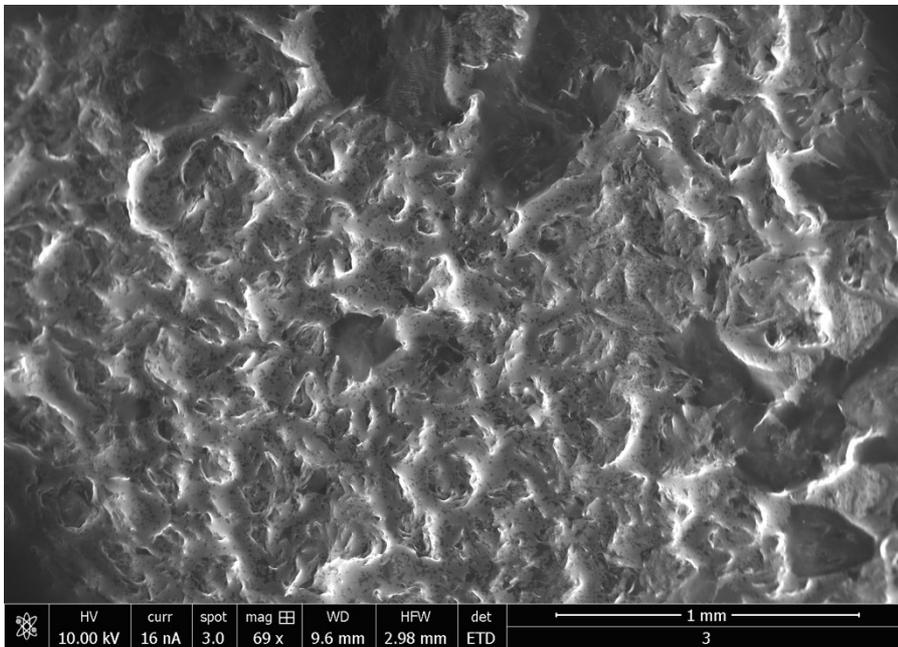


Figure S2. SEM image of graphite/PF₆ xerogel.



Figure S3. Pictures of **a)** water in contact with hybrid gels after adsorption, **b-d)** dispersion of graphite in water, **c-e)** water in contact with **graphite/PF₆** gel after 5h.

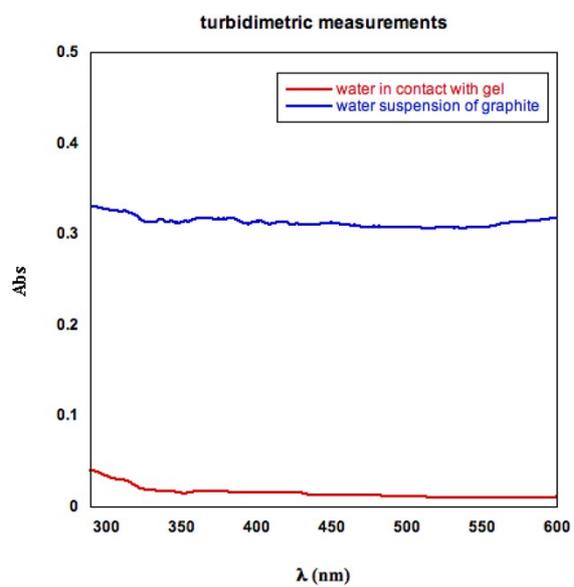


Figure S4. UV spectra of dispersion of graphite in water and water after 5h of contact with **graphite/PF₆** gel.

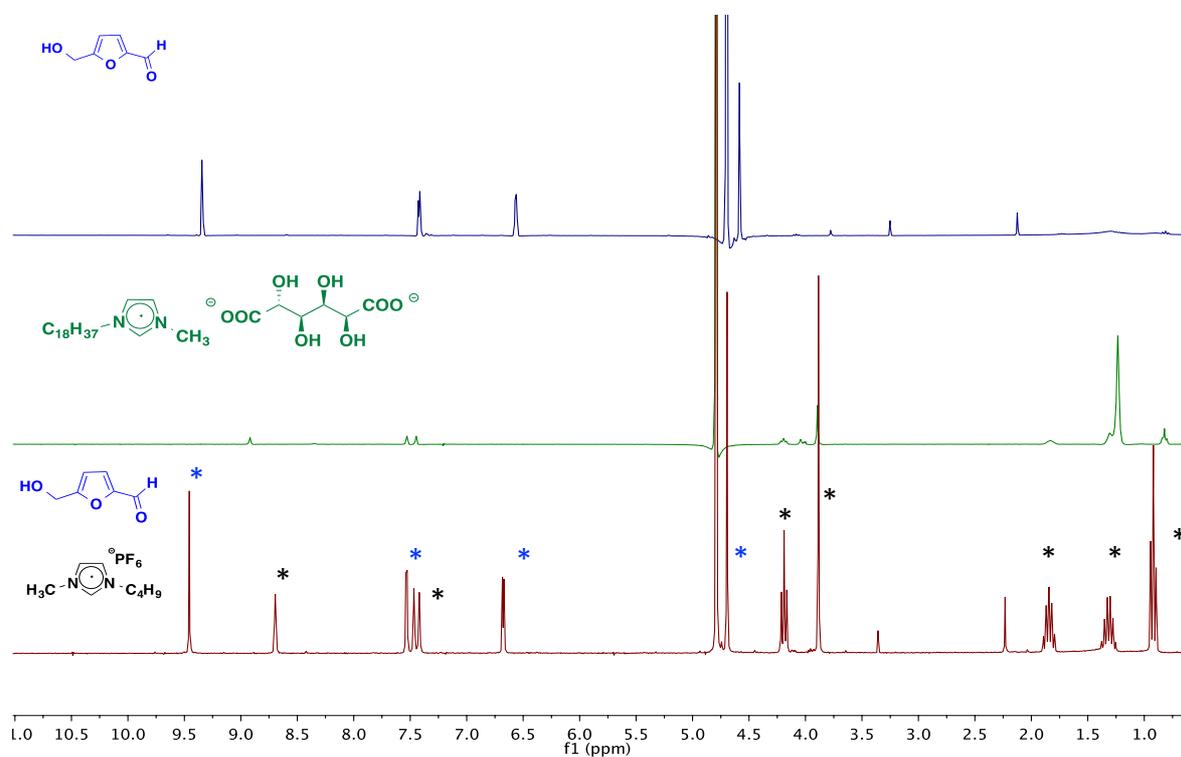


Figure S5. ^1H NMR spectra of: D_2O after 5 h of contact with **graphite/PF₆** with 5-HMF as internal standard (red), colored asterisks identify protons of the components in the mixture; gelator in D_2O (green); 5-HMF in D_2O (blue).

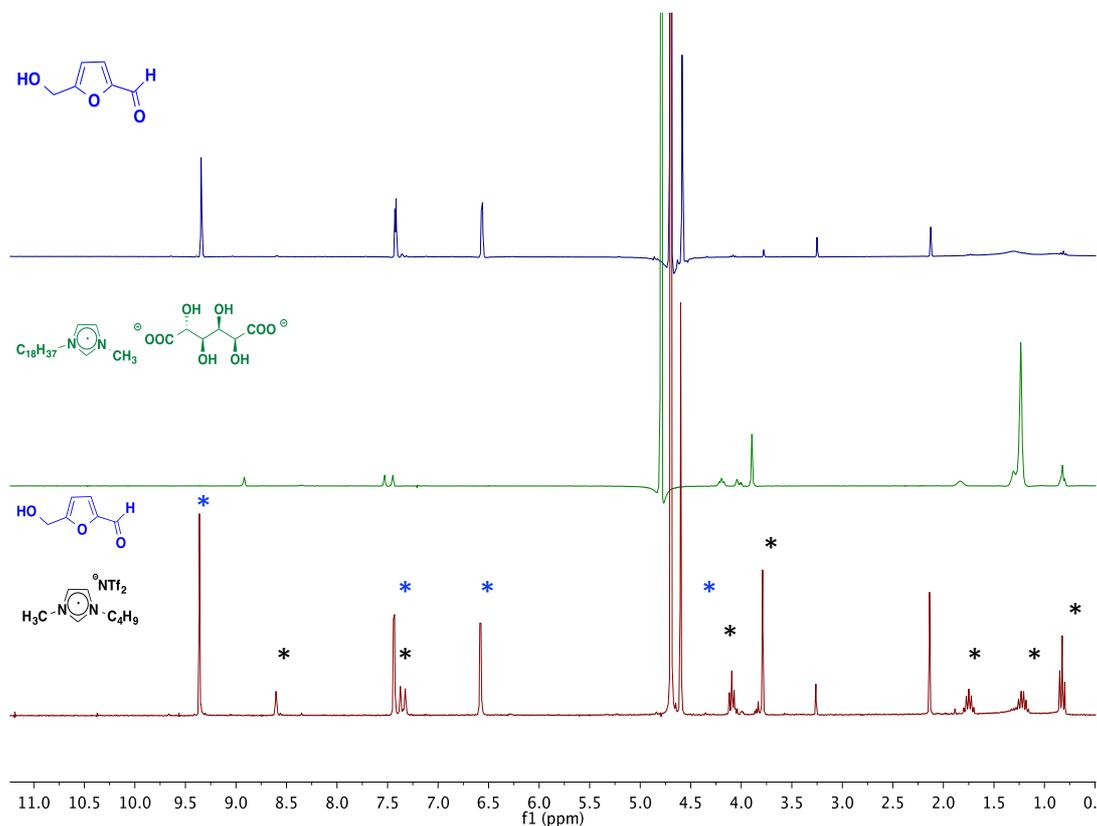


Figure S6. ^1H NMR spectra of: D_2O after 5 h of contact with **graphite/NTf₂** with 5-HMF as internal standard (red), colored asterisks identify protons of the components in the mixture; gelator in D_2O (green); 5-HMF in D_2O (blue).

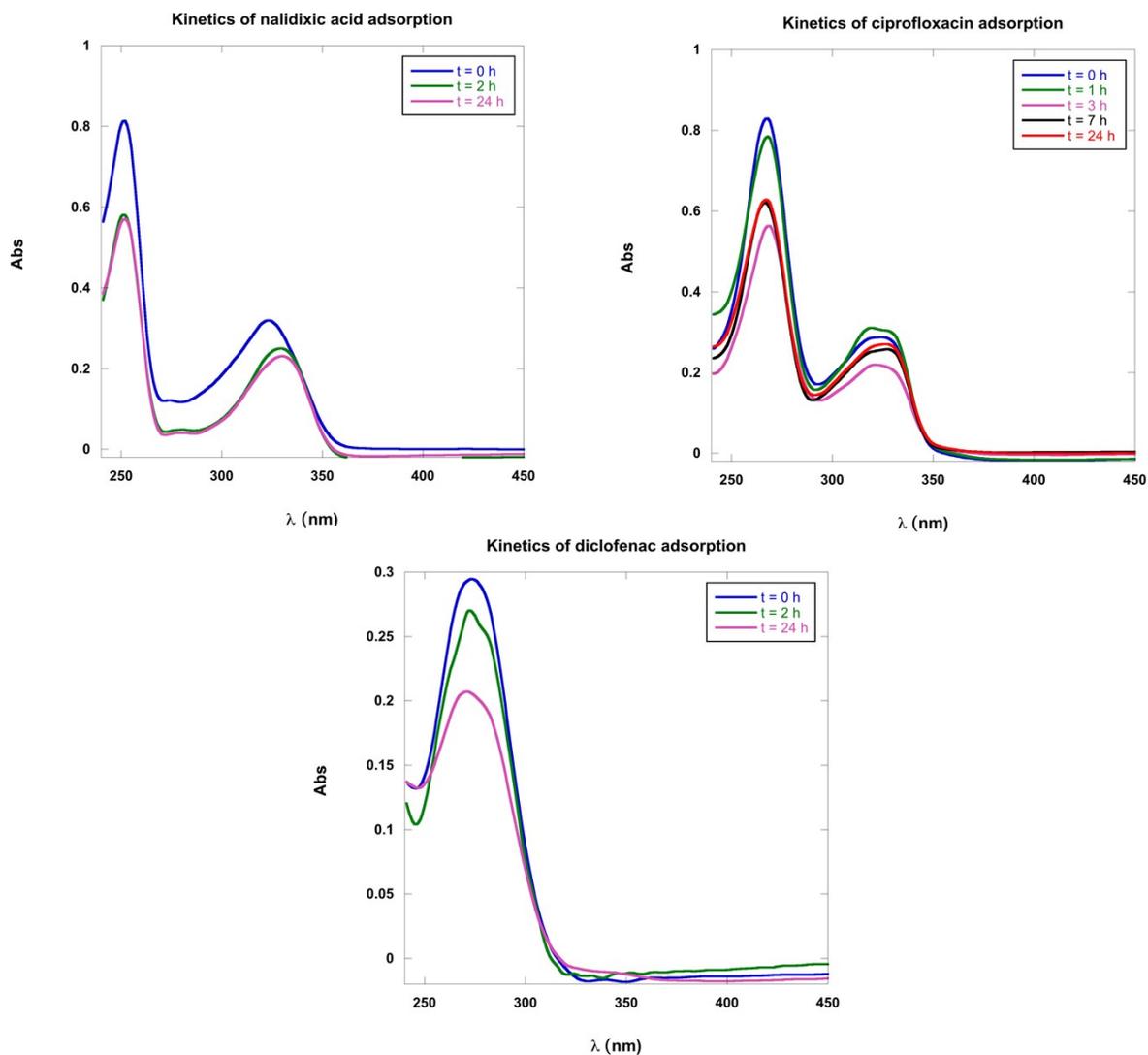


Figure S7. UV spectra of PhAC solutions as function of contact time with gel.

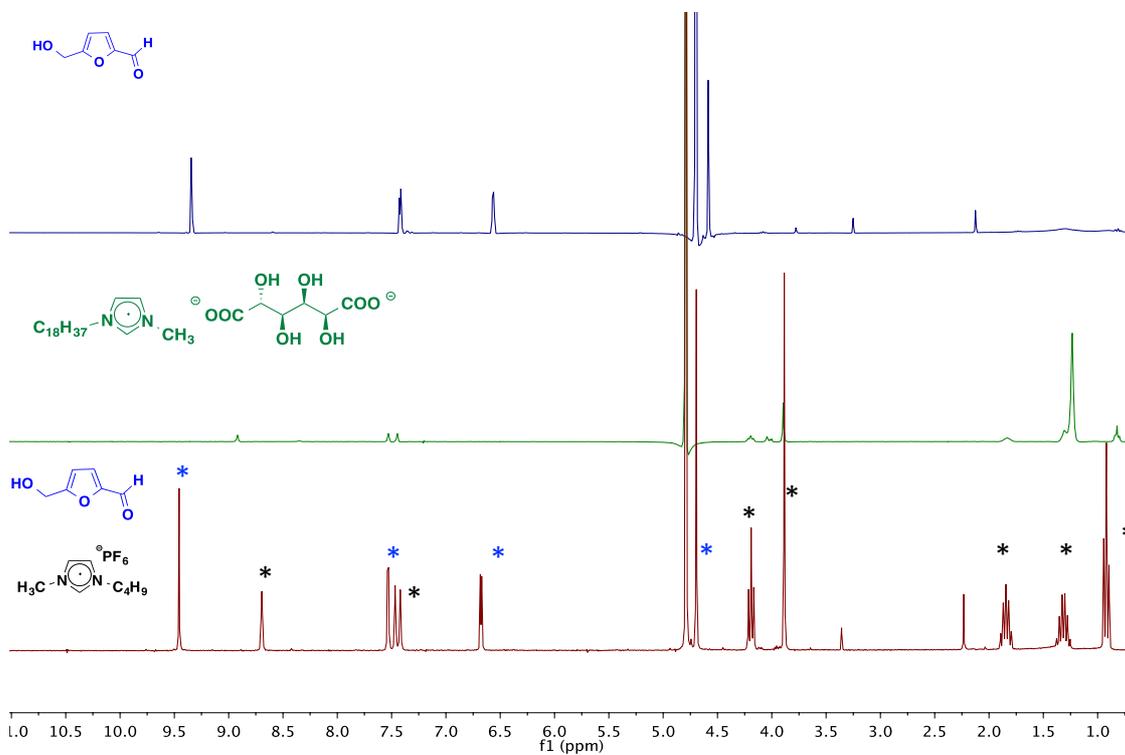


Figure S8. ^1H NMR spectra of: the mixture extracted after the desorption of carbamazepine from **graphite/PF₆** with 2-Me-THF in CD₃OD with 5-HMF as internal standard (red), colored asterisks identify protons of the components in the mixture; gelator in D₂O (green); 5-HMF in D₂O (blue).

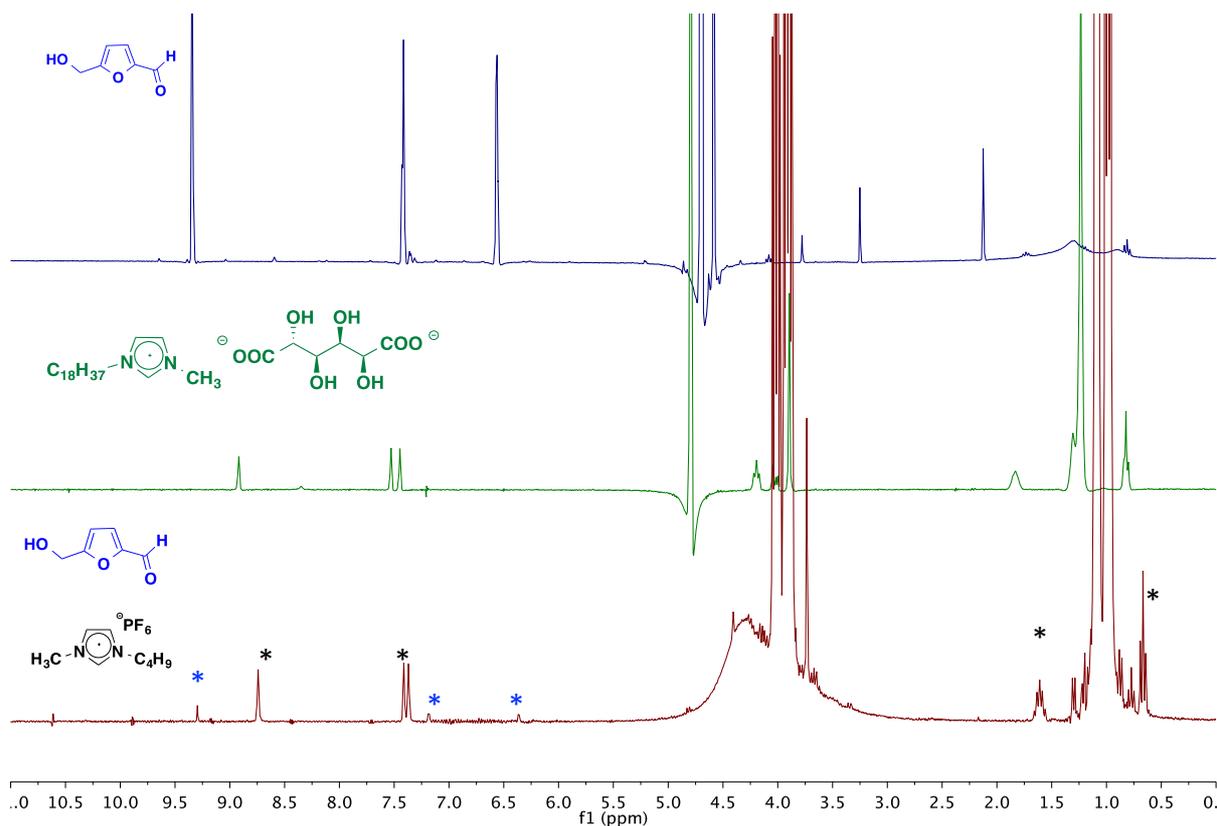


Figure S9. ^1H NMR spectra of: the mixture extracted after the desorption of carbamazepine from **graphite/PF₆** with ethyl-lactate using DMSO-*d*₆ and 5-HMF as internal standards (red), colored asterisks identify protons of the components in the mixture; gelator in D₂O (green); 5-HMF in D₂O (blue).

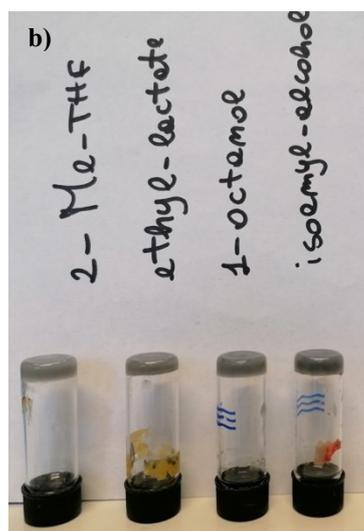


Figure S10. Pictures of **a)** solvents (2-Me-THF, ethyl-lactate, 1-octanol, isoamyl alcohol) in contact with hybrid gels after desorption cycles, **b)** stability of hybrid gels after desorption of carbamazepine.

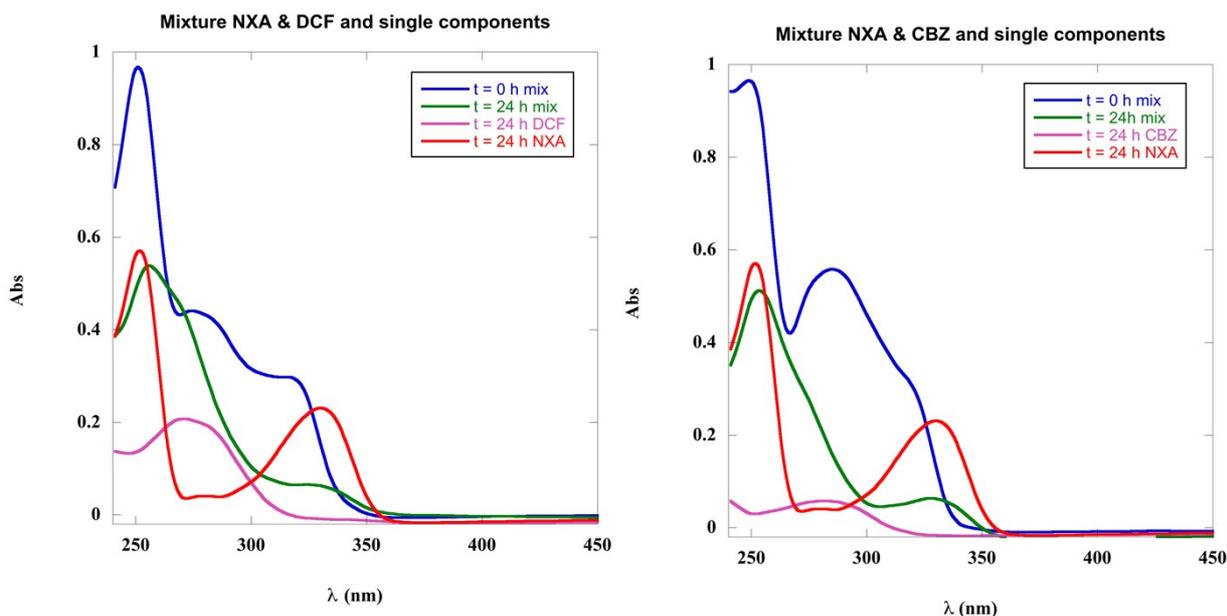


Figure S11. UV spectra of PhAC mixtures and single PhACs before and after adsorption on gel.

Table S1. RE of **graphite/PF₆** for the adsorption of [CBZ] = $1.8 \cdot 10^{-4}$ M after 5 h for several adsorption cycles on the same gel and recycling cycles after desorption of the gel. RE is based on triplicate runs with a reproducibility of 3%.

Recycling cycle	RE %	RE % after desorption
1	83	85
2	74	79
3	74	74
4	59	73
5	52	71
6	62	73
7	57	69
8	59	67
9	40	68
10		61
11		57

Table S2. Desorption of CBZ from **graphite/PF₆** after 9 cycles of adsorption in different solvents.

Solvent of desorption	CBZ extracted (%)	Extraction cycles	Release of [bmim][PF ₆] (%)
2-Me-THF	80	7 x 10 min	17
Ethyl-lactate	58*	3 x 5 min*	20
1-octanol	11	3 x 30 min	
Isoamyl alcohol	25	3 x 30 min	

* reduction of gel volume was observed after the third cycle of desorption.

Table S3. RE of **graphite/PF₆** as function of CBZ initial concentration. RE is based on triplicate runs with a reproducibility of 3%.

Concentration (M)	RE % at 5 h	RE % at 24 h
$6.3 \cdot 10^{-5}$	96	100
$1.3 \cdot 10^{-4}$	89	89
$1.8 \cdot 10^{-4}$	86	90
$3.1 \cdot 10^{-4}$	75	95
$3.4 \cdot 10^{-4}$	78	94
$1.0 \cdot 10^{-3}$	36	86

Table S4. RE of **graphite/PF₆** as function of stirring rate at 5h, 25 °C with an initial concentration of CBZ of $1.8 \cdot 10^{-4}$ M. RE is based on triplicate runs with a reproducibility of 3%.

Rate of stirring (rpm)	RE (%)
0	86
200	85
400	96
800	98

Table S5. RE of **graphite/PF₆** as function of volume of CBZ ($1.8 \cdot 10^{-4}$ M) cast on 0.5 mL of gel after 5 h of contact in static (0 rpm) or dynamic (800 rpm) conditions. RE is based on triplicate runs with a reproducibility of 3%.

Volume (mL)	RE % static condition	RE % dynamic condition
0.6	83	99
1.2	37	86
2.4	19	31
3.6	1	36

Table S6. RE of **graphite/PF₆** in mixtures of PhACs ($1.8 \cdot 10^{-4}$ M) after 24 h of contact. RE is based on triplicate runs with a reproducibility of 3%.

PhAC solution	RE % NXA	RE % CBZ	RE % DCF
NXA	46	-	-
CBZ	-	91	-
DCF	-	-	48
NXA, CBZ, DCF	71	58*	58*
CBZ, DCF	-	75*	75*
NXA, DCF	43	-	-
NXA, CBZ	77	-	-

* RE of total basic PhACs, comprising both CBZ and DCF.