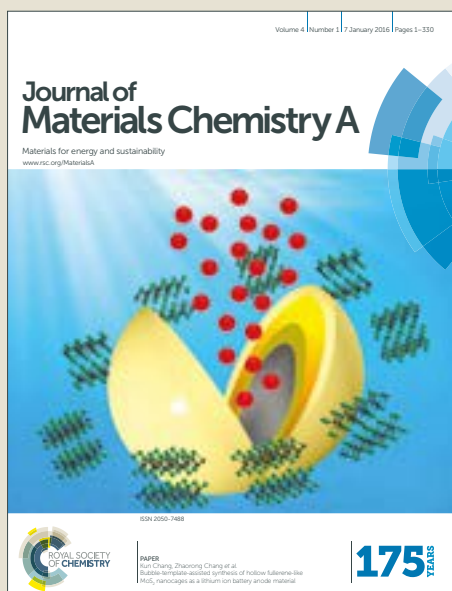


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COMMUNICATION

Combinatorial identification of a highly soluble phase-selective organogelator with high gelling capacity for crude oil gelation†

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Excellent solution-based gelators are generally advantaged by a short gelling time and low gelator loading when compared to powder gelators having a structurally similar backbone. However, high solubility in essentially non-toxic carrier solvents such as ethanol and ethyl acetate still presents a significant bottleneck before their commercial use in large-scale treatment of marine oil spill. In this work, we present a molecular strategy for rapid evolution of a highly soluble POSG (6a-Phe-C4) with high gelling ability toward seven types of (un)weathered crude oils at room temperature. Discovered from a total of 116 PSOGs, gelator 6a-Phe-C4 possesses a record-high solubility of 380 mg/mL in mixed carrier solvent (ethyl acetate:ethanol = 3:2, v:v). This value is even higher than the solubility of 359 mg/mL for NaCl in water. Moreover, its gelling abilities toward two types of heavy crude oils and three types of highly weathered crude oils at room temperature are much better than the best solution-based gelator F-Leu-C4 recently developed by us. As such, the amounts of carrier solvent required now can be reduced markedly to 1.8 - 5.7 vol% of oil to be gelled, which are 0.4 - 5.8 times smaller than those needed for F-Leu-C4.

To help minimize environmental pollution and damages caused by the frequently occurring marine oil spills,¹ phase-selective organogelators (PSOGs), which are capable of phase-selectively gel oil in the presence of water, have been proposed and actively investigated as a unique type of oil-controlling material.²⁻³¹ One noteworthy advantage associated with PSOGs is that the gelled oil stays afloat in water and in principle can be near-quantitatively separated from water by such as simple filtration, enabling near-complete elimination of oil-caused toxicity and possible reclamation of treated oil.

Despite that recent developments on new types of PSOGs have made rapid oil gelation possible with room-temperature operations,¹⁹⁻²³ there still exist significant challenges. For instance, powder gelators often come with low gelling ability and, particularly

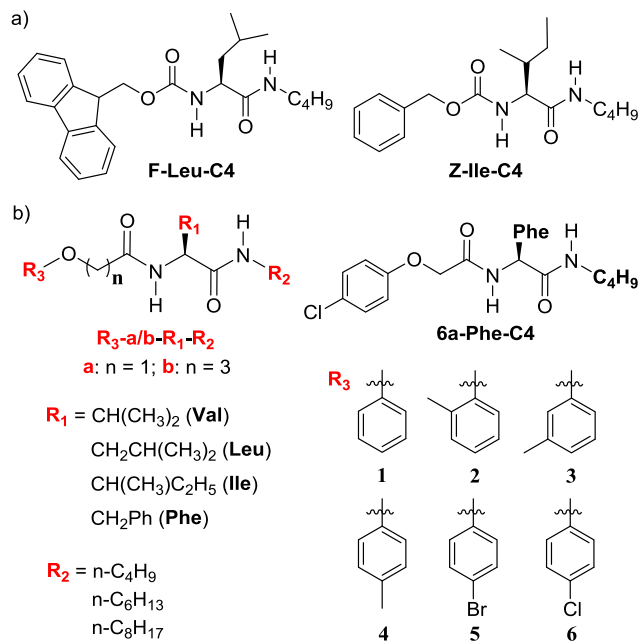


Fig 1 a) Structures of mono-peptide-based phase-selective organogelators **Z-Ile-C4**²⁰ and **F-Leu-C4**.²¹ b) Molecular design for combinatorial evolution of a highly soluble organogelator (**6a-Phe-C4**) with high gelling capacity.

slow gelling actions. Without constant stirring, gelation of crude oil at room temperature could take hours or even days.^{19,20} Although Chaudhuri and his co-workers demonstrated near instantaneous gelation of one type of crude oil using naphthalene diimide-based powder gelator,²³ the generality of the gelator toward different types of crude oils has not been verified. Further, the ability of the hitherto developed powder gelators to rapidly gel heavy crude oil with a viscosity of ≥ 500 mPa•s has not been demonstrated.²⁰ As for solution-based gelators, the foremost challenge is their generally

Table 1. MGC^a and BMGC^b values (% w/v, mg/100 μ L) determined for 14 gelators in five oils using ethyl acetate/ethanol as the carrier solvent.

Gelators	Solubility (mg/mL)	Diesel		Mineral oil		Silicone oil		Pump oil		Paraffin oil	
		MGC	BMGC	MGC	BMGC	MGC	BMGC	MGC	BMGC	MGC	BMGC
1a-Ile-C4	360	0.48	0.92	0.68	0.68	2.69	2.69	0.71	0.83	0.62	0.87
1b-Ile-C4	330	0.87	0.73	0.66	0.70	0.61	0.59	1.23	1.82	1.42	1.65
3a-Ile-C8	150	0.39	0.53	0.81	0.42	1.22	0.89	0.52	1.31	0.82	0.83
5a-Ile-C4	365	0.85	0.88	0.73	1.15	0.48	0.72	0.51	0.86	0.54	0.66
5b-Ile-C6	360	0.62	0.55	0.48	0.48	0.73	0.73	1.20	0.86	1.31	0.88
1a-Phe-C8	110	0.58	0.43	0.47	0.68	0.51	0.67	0.74	0.75	0.78	0.83
5a-Phe-C8	120	0.53	0.66	0.47	0.63	0.44	1.26	0.93	1.77	0.68	0.94
6a-Phe-C4	380	0.72	0.65	0.56	0.57	0.51	0.64	0.63	0.75	0.58	0.78
3a-Val-C6	150	0.71	0.63	0.66	0.53	0.83	0.67	0.92	1.62	0.64	0.84
3a-Val-C8	360	0.66	0.86	0.44	0.64	0.41	0.71	0.89	1.47	0.54	0.78
4a-Val-C4	340	0.56	0.64	0.53	0.66	0.52	0.57	0.43	0.94	0.45	1.13
5a-Val-C6	150	0.78	0.67	0.63	0.70	0.55	0.63	0.52	1.45	0.90	0.85
5a-Val-C8	140	0.88	0.64	0.47	0.66	0.73	0.68	0.53	1.42	0.50	0.75
6a-Val-C8	150	0.46	0.59	0.53	0.58	0.65	0.61	0.43	1.38	0.46	1.36

^a Minimum gelling concentrations were determined by adding gelator in the powder form into the oil, followed by a heating-cooling process. ^b Biphasic minimum gelling concentrations were determined by dissolving gelator in the mixed carrier solvent (ethyl acetate:ethanol = 3:2, v:v) at 110 - 200 mg/mL, which depends on the solubility of gelators.

poor solubility in essentially non-toxic carrier solvents including ethanol and ethyl acetate. As a result, dissolution of gelators requires heating or use of toxic solvent such as THF or a large amount of carrier solvent.²²

In general, solution-based PSOGs display a higher gelling ability than powder gelator.²⁰⁻²² For instance, to gel the same type of heavy crude oil (e.g., Arab Heavy) within mins, the biphasic minimum gelation concentration (BMGC) values of solution-based gelators **F-Leu-C4**²¹ and **Z-Ile-C4**²¹ are 2.5 and 3.7 % w/v (mg/100 μ L), which are 6.2 and 3.8 times smaller than 18% w/v needed for powder gelator **Ac-Ile-C8**.²⁰ Nevertheless, with these relatively low BMGCs, to gel one volume of Arab Heavy, the amounts of carrier solvents (ethyl acetate:ethanol = 3:2, v:v) needed to dissolve **Z-Ile-C4** and **F-Leu-C4**, having high solubilities of 150 and 200 mg/mL, are 25 and 13 vol%, respectively.

To minimize the use of flammable carrier solvents, enhancing gelling ability and/or increasing the solubility of PSOGs are the only ways forward. In this work, we report our discovery, via a combinatorial approach, of a PSOG (**6a-Phe-C4**, Fig. 1b), possessing not only an extremely high solubility of 380 mg/mL (0.85 M) in mixed carrier solvents (ethyl acetate:ethanol = 3:2, v:v) but also greatly enhanced gelling ability toward seven (un)weathered crude oils at room temperature. For instance, in the case of Arab Heavy, the amount of carrier solvents needed to dissolve **6a-Phe-C8** for oil gelation is reduced to 1.8 vol% of oil to be gelled, a value that is about 6 and 13 times less than those needed for **F-Leu-C4** and **Z-Ile-C4**, respectively.

During our effort to find low-cost PSOGs (e.g., **Z-Ile-C4**) to replace more costly Fmoc-containing PSOGs (e.g., **F-Leu-C4**), we found the more flexible linker in **Z-** generally results in lower gelling ability but slightly better solubility than the corresponding **Fmoc-**

that contains a more rigid linker.²¹ Prompted by these findings, we thought that it might be possible to identify PSOGs that are of high solubility while keeping the material cost low by introducing different flexible linkers in combination with low cost aromatic motifs **1-6** (Fig. 1b). In the very fortunate scenario, one or more of these highly soluble PSOGs may concurrently possess high gelling capacity toward crude oils of different types.

Centered around the above hypothesis, we synthesized a total of 116 organogelators by diversifying R₁-R₃ groups with n = 1 or 3 (Fig 1b and Table S1). A representative gelator **6a-Phe-C4** was shown in Fig. 1b to illustrate how the names for these 116 gelators were generated. As compiled in Table S1, solubility determined using mixed carrier solvents (ethyl acetate:ethanol = 3:2, v:v) gratifyingly shows that 12, 37 and 67 gelators have a solubility falling in the ranges of 110 - 200, 210 - 300 and 315 - 390 mg/mL, respectively. Interestingly, among 16 gelators having a solubility of 370-390 mg/mL, 10 of them are leucine derivatives, three from valine, two from phenylalanine and one from isoleucine. These solubility data are very encouraging particularly given the fact that the most soluble gelator **F-Leu-C4** previously reported by us only has a solubility of 200 mg/mL. Therefore, if one or more of these highly soluble gelators turn out to possess the same gelling capacity as **F-Leu-C4**, the big difference in solubility suggests that the amount of carrier solvents used can be reduced by about 50%.

The ability of these 116 gelators to gel oil was first tested using diesel, mineral oil and silicone oil (Table S1). Careful comparison of generated 696 MGC and BMGC values allows us to single out 14 gelators with very high gelling ability toward all three oils with MGCs or BMGCs mostly less than 1% w/v (Tables 1 and S1). Upon cross-examining the solubility and gelling ability of these 14 high capacity gelators, we surprisingly found that none of them is from 37

Table 2. MGC^a and BMGC^b values (% w/v, mg/100 μ L) determined for seven gelators in four crude oils using ethyl acetate/ethanol as the carrier solvent.

	Solubility (mg/mL)	Grissik ($\mu = 0.7$ mPa•s)		Arab Light ($\mu = 2.7$ mPa•s)		Arab Heavy ($\mu = 42.5$ mPa•s)		Ratawi ($\mu = 81.9$ mPa•s)	
		MGC	BMGC	MGC	BMGC	MGC	BMGC	MGC	BMGC
		3a-Ile-C8	150	5.20	1.35	4.80	2.74	3.63	1.45
5a-Ile-C4	360	7.36	1.68	2.28	2.47	3.96	2.08	3.70	2.04
5b-Ile-C6	360	4.86	3.60	9.37	4.46	5.85	4.37	4.86	4.80
3a-Val-C8	360	4.90	8.40	5.00	2.53	2.44	3.32	5.73	2.28
4a-Val-C4	340	2.00	1.83	4.00	3.50	2.56	2.00	2.37	2.16
1a-Phe-C8	110	3.20	1.63	2.90	1.55	3.26	3.71	2.28	1.93
6a-Phe-C4	380	2.13	1.30	4.19	2.00	1.60	0.70	2.85	1.18
F-Leu-C4^c	200	0.48	1.30	1.00	1.40	0.95	2.50	1.26	2.80
Z-Ile-C4^d	150	0.42	1.20	0.94	2.20	1.40	3.70	0.98	3.90

^a Minimum gelling concentrations were determined by adding gelator in the powder form into the oil, followed by a heating-cooling process. ^b Biphasic minimum gelling concentrations were determined by dissolving gelator in the mixed carrier solvent (ethyl acetate:ethanol = 3:2, v/v) at 110 - 200 mg/mL, which depends on the solubility of gelators. ^c from Ref 22. ^d from Ref 21.

gelators having an intermediate solubility of 210 - 300 mg/mL. Instead, seven such high capacity gelators emerge from those 12 gelators with relative poor solubility of 110 - 200 mg/mL with the remaining seven from those 67 gelators with high solubility of 310 - 390 mg/mL. Moreover, none of those highly soluble leucine derivatives could enter the top 14 finalist. These findings are mostly consistent with our own observations that gelators with a relatively poor solubility tend to exhibit high gelling ability, suggesting a great difficulty in developing gelators concurrently possessing high gelling ability and high solubility.

Nevertheless, such an ideal combination now appears to be achievable using a combinatorial approach. More specifically, out of 116 gelators, we identified seven high capacity gelators with high solubility of 330 - 380 mg/mL (Table 1). Expansion to include two more oils (e.g., pump oil and paraffin oil) undoubtedly demonstrates **6a-Phe-C4** as the best gelator among the 14 high capacity gelators (Table 1). Both MGCs and BMGCs for **6a-Phe-C4** against five non-crude oils consistently stay below 0.8 % w/v. Pleasantly, this gelator has a very high solubility of 380 mg/mL, which is even larger than the solubility of 359 mg/mL for NaCl in water.

While scanning electron microscopy (SEM) images clearly demonstrate the extensive formation of nanofibers for diesel gels formed from **5a-Ile-C4**, **3a-Val-C8**, **4a-Val-C4** and **6a-Phe-C4** at 1.5 % w/v, rheological studies suggest elasticity, remarkable stiffness and strength of the gels (Fig. S1).

From Table 1, we choose seven gelators to further investigate their gelling ability toward two types of light crude oil (Grissik, Arab Light) and two types of heavy crude oil (Arab Heavy and Ratawi). Comparing data presented in Tables 1 and 2 show that the MGCs for crude oils are much larger than those for non-crude oils. Nevertheless, further comparison between MGCs and corresponding BMGCs for these gelators surprisingly reveal a completely different

Table 3. BMGC values^a (% w/v, mg/100 μ L) determined in three weathered crude oils³² using ethyl acetate/ethanol as the carrier solvent.

	6a-Phe-C4	F-Leu-C4^b	Z-Ile-C4^c
Weathered Arab Light ($\mu = 11.1$ mPa•s)	1.68	2.30	3.30
Weathered Arab Heavy ($\mu = 334.2$ mPa•s)	2.15	3.20	7.20
Weathered Ratawi ($\mu = 343.0$ mPa•s)	2.00	3.50	N.D.

^b Biphasic minimum gelling concentrations were determined by dissolving gelator in the mixed carrier solvent (ethyl acetate:ethanol = 3:2, v/v) at 150 - 380 mg/mL, which depends on the solubility of gelators. ^b from Ref 22. ^c from Ref 21.

trend for gelling non-crude and crude oils. That is, while MGCs are generally smaller than BMGCs for non-crude oils, MGCs mostly become much larger than BMGCs in the case of crude oils. This comparative finding likely indicates that the larger hydrophobic molecules found only in crude oil could interact more favorably with these gelators containing a flexible linker, enabling these gelators to gel the crude oil more efficiently. This surmise can be additionally supported by the fact that **6a-Phe-C4** exhibits much smaller BMGCs (0.70 and 1.18 % w/v) than both **F-Leu-C4** (2.50 and 2.80 % w/v) and **Z-Ile-C4** (3.70 and 3.90 % w/v) with a more rigid linker for unweathered heavy crude oils Arab Heavy and Ratawi (Table 2). Similar trend persists for highly weathered heavy crude oils (Table 3).³¹

COMMUNICATION

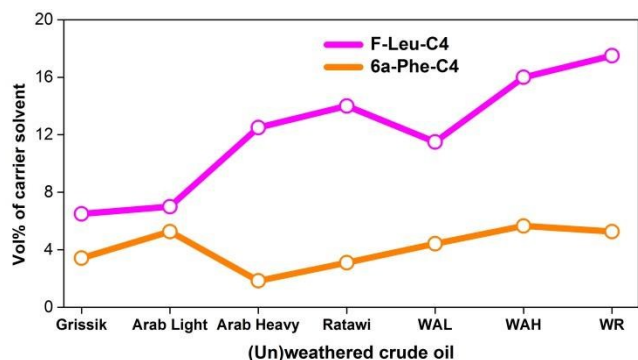


Fig. 2 Volumetric percentages of carrier solvents (ethyl acetate:ethanol = 3:2, v:v) vs volume of crude oil to be gelled determined for **6a-Phe-C4** and **F-Leu-C4**. WAL = weathered Arab Light, WAH = weathered Arab Heavy and WR = weathered Ratawi.

Table 4. PMGC values^a (% w/v, mg/100 μ L) and time needed for five powder gelators to gel Arab Light at room temperature in powder form.

Powder Gelators	PMGC (% w/v)	Time (h)
2a-Ile-C8	3.44	30
2a-Ile-C4	5.17	40
2a-Phe-C8	3.61	20
2a-Phe-C6	3.41	60
2a-Val-C6	4.47	25

^a PMGC refers to powder MGC at the specified gelation time.

On the basis of their solubility data and gelling ability, the amount of carrier solvent needed to dissolve gelators (**6a-Phe-C4** and **F-Leu-C4**) for gelling one volume of seven types of (un)weathered crude oils were calculated and presented in Fig. 2. Fig. 2 shows that the amount of carrier solvent required for **6a-Phe-C4** to gel one volume of crude oil all undergo considerable reductions when compared to those needed for **F-Leu-C4**. In more detail, volumetric percentages of carrier solvent over crude oil are 6.5, 7.0, 12.5, 14.0, 11.5, 16.0 and 17.5 vol% for **F-Leu-C4** against Grissik, Arab Light, Arab Heavy, Ratawi, weathered Arab Light (WAL), weathered Arab Heavy (WAH), and weathered Ratawi (WR), respectively. These percentages are reduced to 3.4, 5.3, 1.8, 3.1, 4.4, 5.7 and 5.3 vol% for **6a-Phe-C4** against the same seven types of crude oil. In general, heavy crude oil experiences better reduction in volume of carrier solvent. Particularly for Arab Heavy, such reduction amounts to as much as 5.8 folds.

Recently, we and the other two groups reported three types of gelators that can gel crude oil in powder form.^{19,20,23} In light of these preceding examples, we have also examined the ability of all 116 PSOGs to congeal Arab Light in powder form at room temperature under constant shaking of 300 rpm. Out of these 116 PSOGs, five of them were found to be capable of functioning as the powder gelator (Table 4). Simple inspection of the names of these five powder PSOGs interestingly uncover a common structure pattern, i.e., all structures of the five powder gelators carry an aromatic motif **2** linked to the peptidic backbone via a short one-carbon linker. From

Table 4, it can be seen that to completely solidify Arab Light at room temperature requires gelator loading of > 3 % w/v and very long gelation time of at least 25 h for all five powder gelators. This long gelation time is much expected since, in our view, fast dissolution of powder gelators into an oil to be gelled likely contradicts with gelators' own precipitation tendency in the same oil.²⁰ This long gelation time also compares very unfavorable with solution-based PSOGs such as **F-Leu-C4** and **6a-Phe-C4**, which could rapidly gel Arab Light within min at gelator loading of 1.3 and 2.0 % w/v, respectively.

Conclusions

In summary, we have demonstrated here a feasible and rapid approach toward combinatorial evolution of highly soluble high capacity phase-selective organogelators. The identified gelator **6a-Phe-C4** not only allows for rapid room-temperature gelation of seven types of (un)weathered crude oils but also effectively reduces the use of essentially non-toxic carrier solvent to 1.8 - 5.7 vol% of oil to be gelled. These values are 0.4 - 5.8 times smaller than 6.5 - 17.5 vol% needed for the best solution-based gelator **F-Leu-C4** recently discovered by us.²² These improvements greatly alleviate the burdens of transport of flammable carrier solvents especially in the event of large-scale marine oil spill, thereby making solution-based PSOGs one step closer to become a practically viable solution for large-scale marine oil spill treatment in the future.

Conflicts of interest

There are no conflicts of interest to declare.

Acknowledgements

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Notes and references

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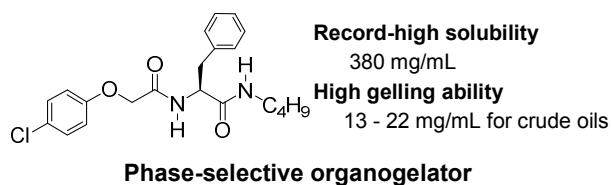
† Electronic Supplementary Information (ESI) available: Experimental procedures, gelling properties, SEM, and rheological data. See DOI 10.1039/b000000x/

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32. To mimic the weathering process of oil spilled into ocean, oil (200-600 mL) was placed on top of 200 mL of seawater in a one-liter bottle, exposed to sunlight at 25-32 °C in Singapore for one week, subject to a thermal treatment at 70 °C for two days in the fumehood, and then continuously left open for months in the fumehood at room temperature.

Table of Contents Entry



One Sentence highlighting the novelty of the work

Combinatorial screening identifies a high capacity phase-selective organogelator with record-high solubility for rapid room-temperature gelation of crude oils.