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Microwave-assisted synthesis of UIO-66 and the adsorption performance to dyes

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In this paper, a microwave-assisted method in the presence of additive has been employed to fast and high-yieldly prepare the UIO-66. The microwave/additive method (MWHAc-UIO-66) gives rise to the full octahedral morphology of ~250nm and the highest surface area 1533.53m^2/g, which is confirmed by surface area of MWBA-UIO-66 (1661.01 m^2/g) with benzoic acid as the additive. The adsorption results of low-concentration acid chrome blue k show that MW-UIO-66 and MWHAc-UIO-66 have excellent adsorption ability (~98%) and HHAc-UIO-66 is not bad (~85%), which are far more than activated carbon. Moreover, the saturated adsorption amount of MWHAc-UIO-66 to acid chrome blue k is up to ~15%. The adsorptions of different dyes show that MWHAc-UIO-66 is favorable to the charged dyes. The reusable experiments indicate that sulfuric acid can facilitate the dye desorption on the MWHAc-UIO-66 instead of base or salt.

KEYWORDS: MOF material; microwave-assisted synthesis; additive; dye; adsorption; reuse

1. INTRODUCTION

MOF materials with tunable channel, high surface area and high crystalline, have been extensively explored by the view of host-guest chemistry including gas-storage, gas-separation, sensing and drug delivery etc.[1-4]. The pore size of MOF materials may permit themselves to incorporate species from ion/small gas molecules to big inorganic/organic compounds. Intensive investigations have been afforded on the behavior of small gas molecule, displaying the excellent ability of MOF material, but corresponding reports on the big species are relatively less. The host-guest chemistry of the big species is really essential because it is relevant to a variety of processes such as pesticides, drugs, light harvesting and water treatments [5-10].

Every year, a lot of waste water containing dyes of different size and charge has been directly drained into water system without any treatment. Even though the dye waste water is primarily disposed, the low-concentration dyes are still probably harmful to human health. This results in serious ecologic and environmental disaster all over the world. As the dye is chemically stable to the light and oxidant, physical adsorption is alternative method to treat the dye waste water. The hydrophobic activated carbon is almost unavailable to trap the low-concentration charged dyes, so new adsorption materials are absolutely necessary to be explored.

At present, UIO-66[11] has increasingly attracted the interests because of the potential applications, especial host-guest chemistry fields such as catalysts [12,13], gas separation/adsorption[14~18] and even cell material [19]. This is owing to the unique stability of UIO-66 originated in some sense from $Zr_6(OH)_4O_4$ SBU [11,20,21], which is confirmed by PCN-222 [22] and MOF-525/MOF-535 [23]. Since Cavka et al.[11] firstly synthesized UIO-66 in a simply system of $ZrCl_4$/BDC/DMF and determined the structure by powder diffraction of very small and aggregated crystals, attempts have been made to prepare the high quality and functional UIO-66. Currently, several methods have employed to prepare the UIO-66, including direct synthesis, precursor synthesis and additive synthesis with the mono-carboxylate acid (formic acid, acetic acid and benzoic acid and so on) as the additive. In the case of direct synthesis [11], UIO-66 can not always be obtained, and the crystallinity and yield can not be guaranteed. For the precursor synthesis, Guillerm et al. firstly utilized $Zr_6(OH)_4O_4(OMc)_{12}$ (OMc=Methacrylate) [24] as the source of $Zr_6(OH)_4O_4$ cluster, and then exchange OMc with terephthalic or *trans*,*trans* muconic acids, to synthesize the UIO-66 and analogy of UIO-66[25]. Employing the precursors with targeted SBU (secondary building unit) seem like a prepared route to achieve the MOF materials[26], but not all precursors with the targeted SBU can be directly purchased or easily prepared. For additive synthesis, Schaate et al. [21] describe in detail the synthesis of UIO-66 from nanometer to single crystal, in which $Zr_6(OH)_4O_4$ is formed in situ by adding the benzoic acid and acetic acid as the additive. Most recently, Biswas and Van Der Voort report the synthesis of a series of functionalized UIO-66 in the presence of formic acid [27]. Additionally, some of Zr-based MOF materials are prepared by the presence of additive [22,23,28,29]. So, the additive synthesis is an excellent route to get the high quality of UIO-66 including shape, size, crystallinity and surface area and so on.

The microwave-assisted synthesis as alternative method has been widely applied for chemical synthesis of organic and inorganic materials. The microwave irradiation can accelerate the crystallization of porous materials requiring several days within several hours. The preparations of MOFs, such as MOF-5[30], MIL-53[31], MIL-101[32] and even PSM of UIO-66[33], have attained by microwave-assisted synthesis. In this work, three methods, including heating/additive, microwave and microwave/additive are employed to prepare the UIO-66, respectively. The results show that UIO-66 can be prepared fast and high-yieldly by the microwave-assisted method in the presence of additive. For clarity, the products of UIO-66 prepared by microwave irradiation, microwave irradiation/acetic acid, microwave irradiation/benzoic acid and heating/acetic acid are denoted as MW-UIO-66, MWHAc-UIO-66, MWBA-UIO-66 and HHAc-UIO-66.

2. EXPERIMENTAL SECTIONS

2.1. Synthesis and Characterization.

2.1.1. Synthesis.

MWHAc-UIO-66. Typically, $ZrCl_4$(150mg, 0.644mmol) was dissolved into solution of acetic acid(5ml, 87.36mmol)/DMF(40ml, 516mmol) to clear solution and continuously stirred for half an hour. Then, BDC (120mg, 0.723mmol) was dissolved into the solution. Finally, the mixed solution with molar ratio of DMF : HAc : BDC : $ZrCl_4$ = 800 : 135 : 1.12 : 1 was put into the microwave oven and irradiated at 100°C for 2 hours. The product was centrifuged at 6000rpm for 5min, washed three times by ethanol and dried at 60°C overnight. The yield (based on the $ZrCl_4$) is close to 90%.

MWBA-UIO-66. The MWBA-UIO-66 was prepared accordingly to above process, in which $ZrCl_4$ was dissolved into the mixture of benzoic acid/DMF with 30 times of the benzoic acid. The aftertreatment was the same as above procedure. The yield (based on the $ZrCl_4$) is close to 85%.

MW-UIO-66. The preparation of MW-UIO-66 was carried out except for the additive according to the above procedure. The yield (based on the $ZrCl_4$) is close to 80%.

HAC-UIO-66. The mixture of DMF/acetic acid/BDC/$ZrCl_4$ with molar ratio of DMF : HAc : BDC : $ZrCl_4$ = 800 : 135 : 1.12 : 1 was put into oven at 120°C for 24hrs. The aftertreatment was the same as microwave irradiation. The yield (based on the $ZrCl_4$) is close to 70%.

2.1.2 Characterization Techniques.

Powder X-ray diffraction (PXRD). Powder X-ray diffraction (PXRD) patterns were recorded on a Rigaku D/MAX PC2200 diffractometer for Cu Kα radiation (λ =1.5406 Å), with a scan speed of 5°/min.

The thermal gravimetric analyses (TG). The thermal gravimetric analyses (TG) were performed on Pyris Diamond TG/DTA instrument used in nitrogen environment with a heating rate of 10°C/min.

TEM. The morphologies of the samples were inspected on a transmission electron microscope (JEOL-2000ex).

Distribution of Particle size. The particle size was determined by ZetaPals Potential Measurement Analyzer with 5% PVP of aqueous solution.

N_2 isothermal adsorption. N_2 isothermal adsorption experiments were performed

at 77 K with a V-Sorb 2800P apparatus using nitrogen as the probing gas. The samples were vacuumed for 6hrs at 120°C before the data were collected.

2.2 adsorption experiments of dyes

The adsorption performances of dyes on UIO-66. The adsorption performances of dyes on UIO-66 were carried out in the aqueous solution under room temperature. The aqueous stock solutions of dyes (500 ppm) were prepared by dissolving dyes in deionized water. The aqueous dye solutions were diluted to different concentrations from 50 to 250 ppm. The dye concentrations were determined using ultraviolet spectrophotometer. The calibration curve was obtained from the spectra of standard solutions (1–300 ppm).

The saturated adsorption amount. The saturated adsorption amount was carried out. 50mg MWHAc-UIO-66 was soaked into 50ppm/50ml, 150ppm/50ml and 250ppm/50ml acid chrome blue k, respectively.

The adsorptions of charged dyes on MWHAc-UIO-66. The adsorptions of charged dyes on MWHAc-UIO-66 were carried out with 50mg MWHAc-UIO-66 soaked into 50ppm/50ml dye aqueous solutions.

The reusable experiment. For the reusable experiment, the used MWHAc-UIO-66 was soaked three times in 0.05M H_2SO_4 (50ml) and dried at 120°C.

3. RESULTS AND DISCUSSIONS

3.1. Characterizations of the Materials.

3.1.1. Powder X-ray diffraction (PXRD).

In comparison with the simulated PXRD pattern of UIO-66, the results of PXRD show that all prepared products are verified to be UIO-66. The difference between diffraction peaks is very slight and attributed to the crystal orientations (Supporting Information, Figure S1).

3.1.2. The thermal gravimetric analyses (TG).

All TGs are performed directly from the prepared products without furthermore post-treatment. Like PXRD, the TG curves do not render the significant distinct. The thermal stability of the four products can reach about 500°C and the weight loss of the four products are about 60% or so, which is in consistence with the calculated value of 60.2% (based on the formula $Zr_6O_4(OH)_4(C_8H_4O_4)_6$) (Supporting Information, Figure S2).

3.1.3. TEM and N_2 adsorptions.

The morphologies and sizes of samples are observed by TEM diagram (Fig.1). But MW-UIO-66 is inter-grown aggregate of very small irregular crystals, the other three products are perfect and uniform octahedral morphologies, which means that the additive (mono-carboxylate acid) is favor to form the single crystal of UIO-66. The particle sizes of MWBA-UIO-66, MWHAc-UIO-66 and HHAc-UIO-66 are <100nm, ~250nm and ~400nm, respectively. In case of heating method, the reaction time is longer up to 24h, so the particle size is the biggest one. Under the same condition, the particle size of MWHAc-UIO-66 is bigger that that of MWBA-UIO-66. The distribution of particle size is determined by the ZetaPals Potential Measurement Analyzer with 5% PVP of aqueous solution (Table 1, Fig.2). For the sake of the aggregation of the nano-particles, the particle sizes of MWBA-UIO-66 are larger than the crystallite sizes obtained from TEM, whereas the particle sizes of HHAc-UIO-66 are smaller than that from TEM due to the sedimentation of weighted particles.

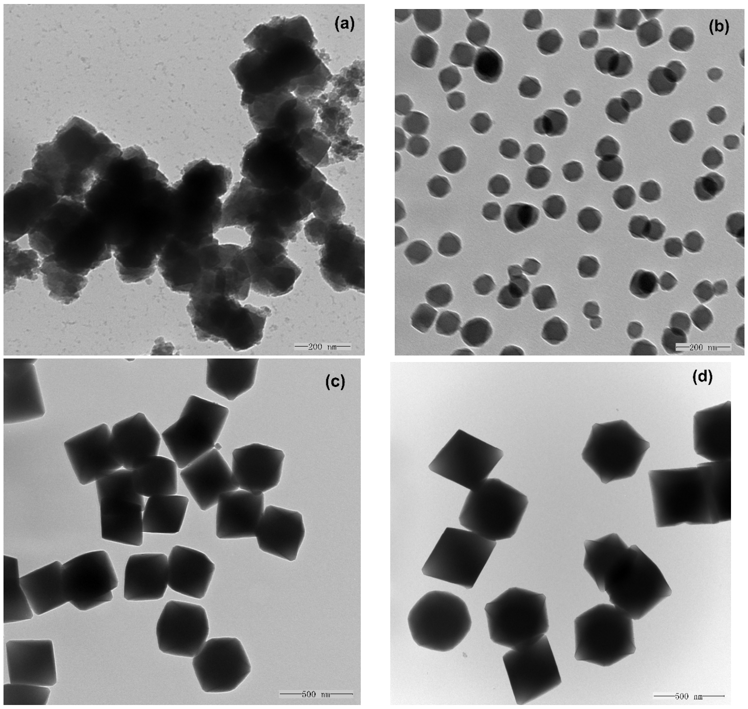

Fig.1 The TEM diagrams of MW-UIO-66 (a), MWBA-UIO-66 (b), MWHAc-UIO-66 (c), and HHAc-UIO-66 (d).

Table 1. Particle size determination

Sample	d_{water}/nm*	d_{TEM}/nm
MWBA-UIO-66	112~169(136)	<100
MWHAc-UIO-66	232~240(233)	~250
HHAc-UIO-66	204~312(242)	~400

* Data for particle sizes from ZetaPals Potential Measurement Analyzer with 5% PVP of aqueous solution (the maximum of the distribution is given in parentheses).

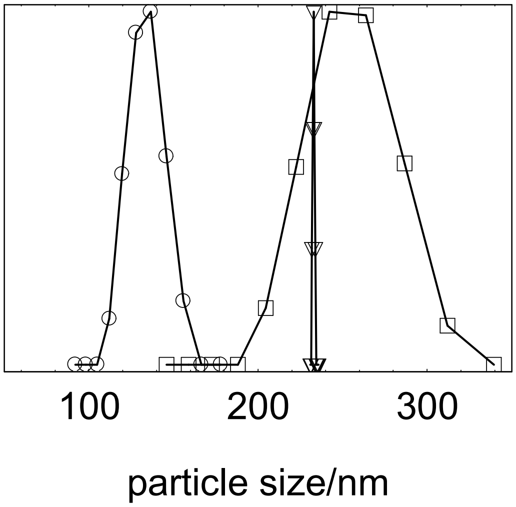

Fig.2 The distribution of particle size: MWBA-UIO-66 (○); MWHAc-UIO-66 (▽); HHAc-UIO-66 (□).

Because the nitrogen is very small, stable and non-polar (Am = 0.162nm^2, 77K), it is usually served as the probing molecule to determine the surface area of material. A comparison of Langmuir surface area determined from N_2 sorption isotherms of four products, are given in Table 2 (Supporting Information, Figure S4). Without any additive, the surface area of MW-UIO-66 is the smallest one about 888.05 m^2/g in spite of utilization of microwave irradiation. With the presence of additive, surface area of HHAc-UIO-66 (1194.44 m^2/g) is more than MW-UIO-66 despite tradition heating. In the presence of additive and microwave irradiation, the surface area of MWHAc-UIO-66 is the biggest one up to 1533.53 m^2/g. The effects of additive and microwave irradiation on the preparation of UIO-66 can be confirmed by MWBA-UIO-66 (1661.01 m^2/g). The surface area to the same MOF material is tightly correlated with the prepared method, for example that the surface area of MIL-101 ranges from 2500 m^2/g to 6000m^2/g with different prepared methods. This means that one will do enormous after-treatments in order to achieve the high surface area MIL-101. For UIO-66, the aftertreatment is no less than that of MIL-101[21]. However, by the method of additive/microwave in this work, UIO-66 with the high surface area could be prepared just after activation that is of course necessary to MOF material rather than doing enormous after-treatments.

Table 2. Comparison of Langmuir surface area determined from N_2 sorption isotherms of UIO-66 synthesized under different conditions

Sample	Synthesis condition		Langmuir surface area/
	MW irradiation	Additive	(m^2/g)
MW-UIO-66	Yes	No	888.05
HHAc-UIO-66	No	Yes	1194.44
MWHAc-UIO-66	Yes	Yes	1533.53
MWBA-UIO-66	Yes	Yes	1661.01

3.2. Adsorption performance of dyes.

3.2.1. The adsorption performance of the different UIO-66s to the dye.

The adsorption performances to molecules with formula weight ranging from 100Da to 1000Da are very important because they represent actually industrial procedure. The dye at this level is one of main water contaminations. With regard to the results of 77K N_2 isothermal adsorption, the acid chrome blue k (50ppm aqueous solution) as the probe molecule is employed to investigate the adsorption performance of UIO-66 synthesized by the three methods with the activated carbon as the reference. As shown in the Fig.3, the activated carbon is almost unavailable to the acid chrome blue k owing to its hydrophobicity. The concentration of acid chrome blue k in the HHAc-UIO-66 declines to 8ppm over 80% adsorption. The MW-UIO-66 and MWHAc-UIO-66 can make the concentration of acid chrome blue k decline to less than 1ppm nearly to 98% adsorption.

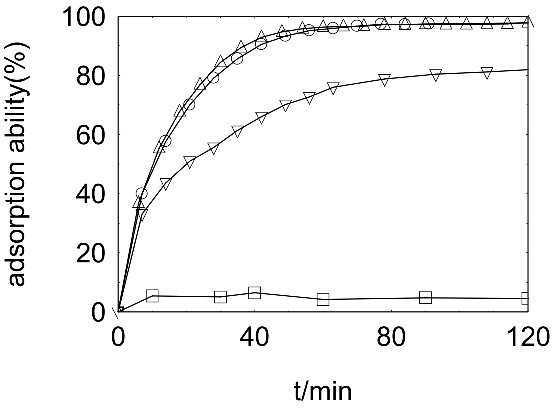

Fig.3 The adsorption ability to acid chrome blue K. MW-UIO-66 (a, △); MWHAc-UIO-66 (b, ○); HHAc (c, ▽); activated carbon (d, □).

It is interesting that the adsorption ability of MW-UIO-66 to acid chrome blue k is better than that of HHAc-UIO-66 even though the surface area of HHAc-UIO-66 (N_2, 77K) is more than that of MW-UIO-66. For the bulky molecules like dye of which size is bigger than that of adsorbent, the adsorption possibly occurs on the external surface according to ~0.6nm nanogate and ~1.1nm cavity of UIO-66. This means that the adsorption ability is closely dependent on the surface environment such as the surface charge distribution, the surface tension and the surface roughness etc., rather than the surface area at N_2 and 77K. Microwave irradiation can accelerate the reaction and shorten the reaction time, which presumably results in more active surface of UIO-66. Nevertheless, the situation becomes acceptable that MW-UIO-66 and MWHAc-UIO-66 possess the near adsorption ability to acid chrome blue k regardless of the surface area at N_2 and 77K and is significantly better than HHAc-UIO-66.

3.2.2. The saturated adsorption amount of MWHAc-UIO-66.

The saturated adsorption amount is determined by three concentrations (50mg/ml, 150mg/ml, 250mg/ml). The results of saturated adsorption amount are shown in Fig.4. The results indicate that the saturated adsorption amount of MWHAc-UIO-66 is about 150mg/g.

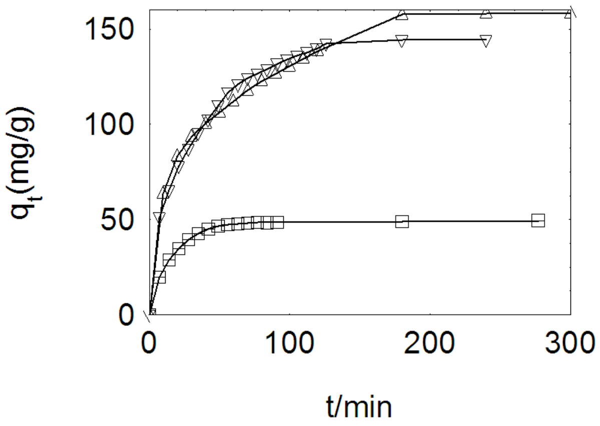

Fig.4 The adsorption amount of acid chrome blue k soaked in MWHAc-UIO-66. The concentration of acid chrome blue k: 50ppm (a, □); 150ppm (b, ▽); 250ppm (c, △).

3.2.3. The adsorption performance of MWHAc-UIO-66 to the charged dyes.

The adsorptions of several charged dyes on MWHAc-UIO-66 are carried out with 50mg MWHAc-UIO-66 soaked into 50ppm/50ml dye aqueous solutions. The molecular weights and charges of the used dyes are listed in Table 3, noting that uncharged dyes are not considered because they do not dissolve in the water. The results show that UIO-66 can absorb the charged dyes (Fig.5). The adsorption of charged dyes on the UIO-66 is influenced by many factors such as shape, size, charge, temperature, concentration, pH and so on, and hence this comparison hardly gives rise to the regular results.

Table 3 Molecular weight of dye molecules with different charges.

	Acid Chrome Blue K	FBA351	Crocein scarlet MOO	Acid Red 1	MO
Z	3	2	2	2	1
Formula	$C_{16}H_9N_2Na_3O_{12}S_3$	$C_{28}H_{20}Na_2O_6S_2$	$C_{22}H_{14}N_4Na_2O_7S_2$	$C_{18}H_{13}N_3Na_2O_8S_2$	$C_{14}H_{15}N_3NaO_3S$
Mw	586.40	562.56	556.48	509.42	327.33

FBA351: 4,4'-bis(2-sulfonatostyryl)biphenyl disodium; MO: methyl orange.

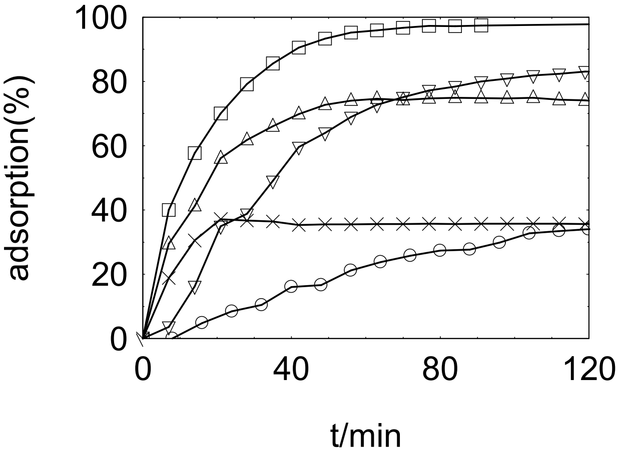

Fig.5 The adsorption of dyes on the MWHAc-UIO-66. Acid chrome blue K(a, □); Acid Red 1(b, ▽); FBA351(c, ○); acid red 9(d, △); MO(e, X).

3.2.4. The reuse.

The reuse is necessary for the industrial process. Desorption of dyes on the UIO-66 can not be attained just by soaking with deionized water or ultrasonic vibration. The salt and base including $NaNO_3$ and NaOH are unavailable, especially NaOH which results in the structural collapse. The acids such as HAc, HCl and H_2SO_4 are investigated, respectively. Comparably, H_2SO_4 is more effective. The results of reusable experiments of two cycles are shown in Fig. 6. After the 2nd reuse, the adsorption ability decreases a little bit. This is confirmed by the results of PXRD on which the crystalline decreases even though the crystal structure does not collapse (Supporting Information, Figure S6).

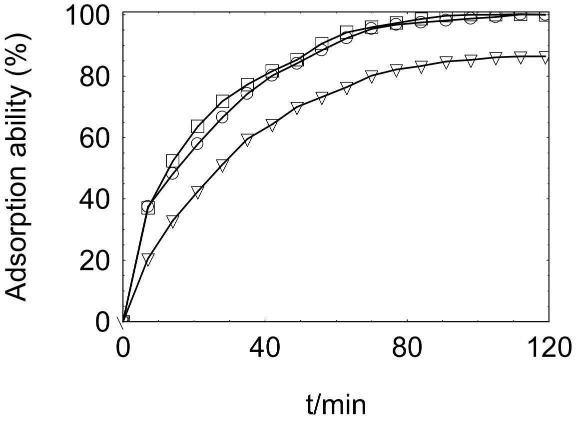

Fig.6 The reuse of MWHAc-UIO-66 on the adsorption of acid chrome blue K (150ppm): adsorption (a, ○); 1st cycle (b, □); 2nd cycle (c, ▽).

4. CONCLUSION

In conclusion, the UIO-66 is fast and high-yieldly prepared in the presence of additive (benzoic acid and acetic acid) by the microwave irradiation. The results show that the additive can favorite the growth of single crystal and microwave can shorten the reaction time so as to improve the surface activity. The UIO-66 can adsorb the charged dyes but the regular result is unavailable. The reusable experiment shows that the sulfate acid can fabricate desorption of dyes on the UIO-66.

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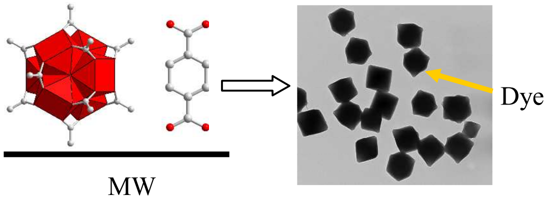

Highlight

UIO-66 nano-particles with octahedral morphology and high surface area have been prepared fast and high-yieldly by the method of additive/microwave.

MOF Material

Microwave-assisted synthesis of UIO-66 and the adsorption performance to dyes

Yafeng Li*, Yan Liu, Wenyuan Gao, Limei Zhang, Wei Liu, Jingjing Lu, Zheng Wang