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An Ionic Liquid on a Porous Organic Framework Support: A Recyclable Catalyst for the Knoevenagel Condensation in an Aqueous System

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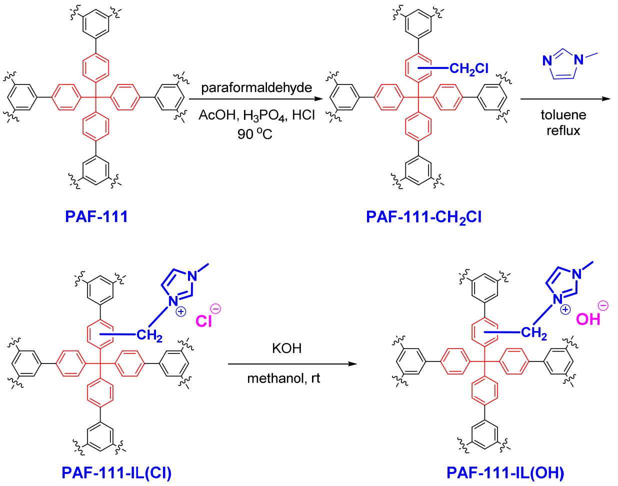
Abstract: Porous aromatic frameworks (PAFs) featured by the high density of phenyl rings in their robust frameworks are attractive platforms for catalysis because of their extremely high stability, high surface area and adjustable pore size. In this paper, two novel PAF-supported ionic liquids were constructed by introducing ionic liquid units onto the framework of a PAF material named **PAF-111** using a series of stepwise post-synthetic modifications. The basic PAF-supported ionic liquid with hydroxyl anion exhibited high catalytic activity, high stability and could undergo at least 10 cycles without any activity loss when catalyzing Knoevenagel condensation reaction under aqueous conditions. It is expected that our study will further promote the development of designing and applying functional PAF materials for catalysis in aqueous system.

In recent years, a tremendous development has been seen in the field of porous organic materials which were built entirely from organic building blocks via strong covalent bonds.[1] Covalent organic frameworks (COFs) and porous organic polymers (POPs) are two representative types of porous organic materials. Generally, COFs are all prepared by reversible reactions, therefore COFs usually have orderd structures but the resultantly formed covalent bonds such as boron-oxygen bonds or imine bonds could reduce the stability of COFs under certain conditions and can interfere with some aplications. By contrast, POPs constructed by very stable covalent bonds have high stability but usually with flexible structure. In the last decades, our group has developed a series of porous aromatic frameworks (PAFs) which is a new type of porous organic material.[1e, 2] Because PAFs are constructed by high density of rigid aromatic building blocks, they usually have robust frameworks and perfect stability, which could combine the exceptional features of COFs and POPs.[2e, 3] Since the seminal work of PAF-1[4] reported by our group, PAFs have been used for diverse applications such as adsorption,[3, 5] separation,[6] sensing[7] and heterogeneous catalysis[8]. Due to that the catalytic active sites are more exposed in the pores constructed by the robust frameworks of PAFs, PAF materials have been proved by our group to have distinctive advantages as platforms for organocatalysis[9] and organometallic catalysis[10]. However, most of the reported PAF-based catalysts were used in organic solvents. Considering that PAF materials have highly stable absolute organic frameworks, the systematic investigation of the PAF-based catalysts in aqueous system is very attractive for the reserchers in this field.

In this paper, Ionic liquid catalytic sites will be introduced into the pores of PAF materials and the performence of the resultant catalysts in aqueous system will be studied. Ionic liquids have been found to have many advantages in the area of green chemistry.[11] Supported ionic liquid, which is highly advantageous because of the ease of separation, is necessary in chemical industry. Considering the need of easy accessibility to the catalytic sites, PAFs with hierarchically porous structures are one of the best candidates as the platform for immobilizing ionic liquid.

Taking into account the above discussion, the hierarchically porous material **PAF-111**, which was synthesized in our previous literature,[12] was selected as the supported material. In order to introduce the ionic liquid site, a series of stepwise post-synthetic modifications was applied in our strategy (Scheme 1). First, a chloromethyl group was attached to the framework of **PAF-111** as an electrophilic site, through the chloromethylation reaction using paraformaldehyde in a mixed acid solution, affording **PAF-111-CH$_2$Cl**. Then through the newly introduced electrophilic chloromethyl site, the ionic liquid functional group could be easily introduced onto the framework by the S$_N$2 substitution reaction using *N*-Methylimidazole as nucleophilic reagent, and the obtained material was named **PAF-111-IL(Cl)**.



Scheme 1. Synthetic route for **PAF-111-IL(Cl)** and **PAF-111-IL(OH)**.

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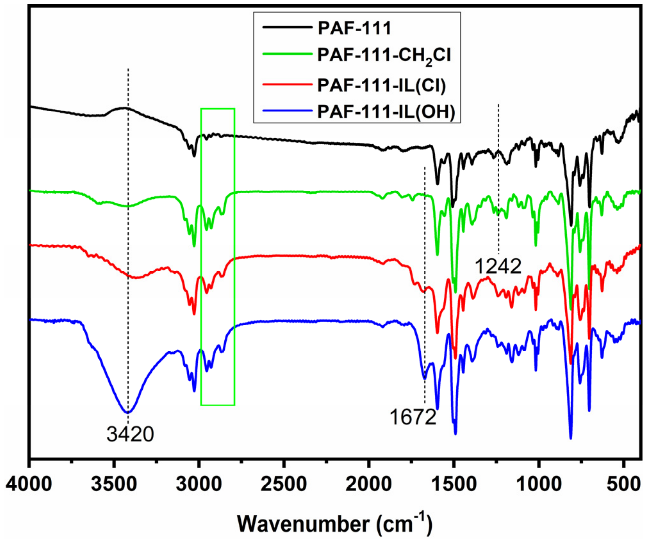


Figure 1. FT-IR spectra of **PAF-111**, **PAF-111-CH$_2$Cl**, **PAF-111-IL(Cl)** and **PAF-111-IL(OH)**.

Then through a simple anion exchange reaction, the chloride anion could be easily transformed to hydroxyl anion (OH^-), and the designed immobilized basic ionic liquid **PAF-111-IL(OH)** could be obtained.

After obtaining the PAF materials in Scheme 1, Fourier transform infrared (FT-IR) spectra were first conducted to verify if the strategy successfully led to the desired catalyst. As shown in Figure 1, compared with **PAF-111,** in the FT-IR spectrum of **PAF-111-CH$_2$Cl**, the newly appeared peak at 1242 cm^{-1} and the newly appeared peaks in the region of 2830-2980 cm^{-1} could be respectively ascribed to bending vibration and stretching vibration of saturated C−H bonds linked to the chlorine atom. These new peaks indicated the successful introduction of chloromethyl group into the PAF material. Compared with **PAF-111-CH$_2$Cl**, in the FT-IR spectra of **PAF-111-IL(Cl)** and **PAF-111-IL(OH)**, the similar peaks in the region of 2830-2980 cm^{-1} indicated the existence of saturated C−H bonds and the new band at 1672 cm^{-1} could be assigned to C=N bonds of the imidazole ring. In addtion, the strongly enhanced broad band at 3420 cm^{-1} in the FT-IR spectrum of **PAF-111-IL(OH)** could be assigned to stretching vibration of hydroxyl group. These results agree well with the designed structures of **PAF-111-IL(Cl)** and **PAF-111-IL(OH)**, clearly indicating that the ionic liquid was successfully immobilized into the PAF material.

The porosities of the obtained materials were demonstrated by nitrogen adsorption–desorption isotherms, as shown in Figure 2. The isotherms of **PAF-111**, **PAF-111-CH$_2$Cl**, **PAF-111-IL(Cl)** and **PAF-111-IL(OH)** in Figure 2 all showed a rapid uptake at low relative pressure, indicating the existence of micropores in the above PAF materials. The Brunauer–Emmett–Teller (BET) surface areas for **PAF-111, PAF-111-CH$_2$Cl**, **PAF-111-IL(Cl)** and **PAF-111-IL(OH)** were 857 m^2 g^{-1}, 749 m^2 g^{-1}, 702 m^2 g^{-1} and 666 m^2 g^{-1} respectively. The changes of the BET surface areas for the above PAF materials matched well with the sizes of the corresponding functional groups in them.

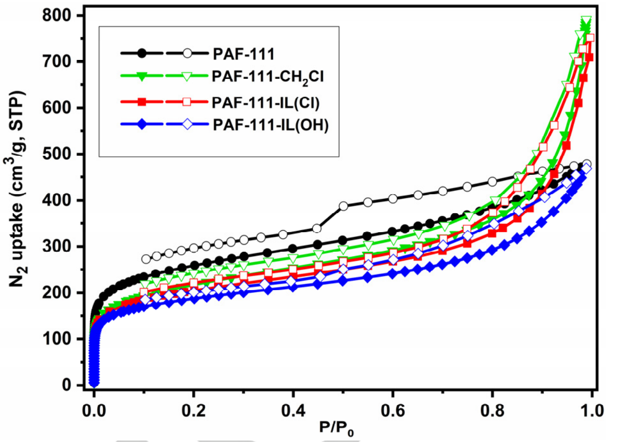


Figure 2. Nitrogen adsorption (solid symbols)–desorption (open symbols) isotherms measured at 77 K for **PAF-111**, **PAF-111-CH$_2$Cl**, **PAF-111-IL(Cl)** and **PAF-111-IL(OH)**.

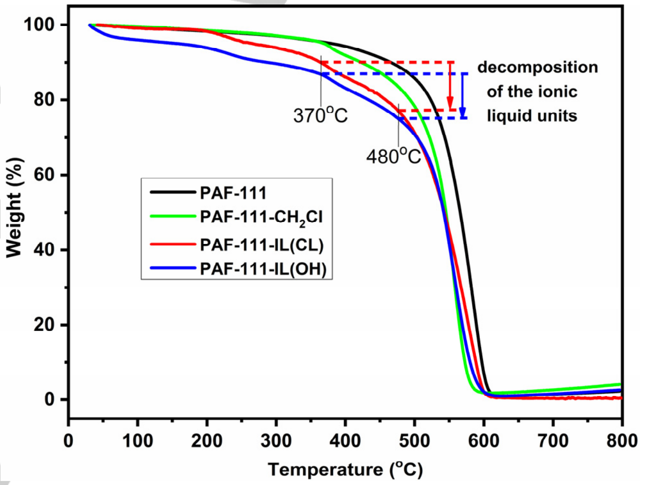
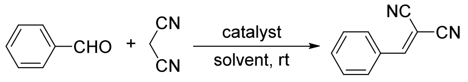


Figure 3. TGA plots of **PAF-111**, **PAF-111-CH$_2$Cl**, **PAF-111-IL(Cl)** and **PAF-111-IL(OH)**.

Then the thermogravimetric analysis (TGA) was used to test the thermal stability of the obtained PAF materials. As shown in Figure 3, the TGA curves of **PAF-111** showed no obvious weight loss below 450 °C, suggesting its high thermal stability. Compared with **PAF-111**, **PAF-111-CH$_2$Cl** showed an obvious weight loss starting at 370 °C, which might be ascribed to the decomposition of chloromethyl group on the framework. In the case of **PAF-111-IL(Cl)** and **PAF-111-IL(OH)**, there was a slow loss of mass below 370 °C, which might be due to the gradual loss of some guest molecules in the pores of the PAF material. The loss of mass below 370 °C for **PAF-111-IL(OH)** was more obvious than that for **PAF-111-IL(Cl)**, which indicated that in **PAF-111-IL(OH)** there were stronger interaction forces (such as intermolecular hydrogen bonding) between the ionic liquid units and the guest molecules. In addition, **PAF-111-IL(Cl)** and **PAF-111-IL(OH)** both showed a slow weight loss between 370-480 °C, which might be attributed to the decomposition of the ionic liquid units hung onto the framework. For all the above PAF

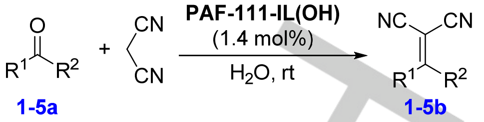
Table 1. The control experiments for **PAF-111-IL(OH)** catalysed Knoevenagel condensation reaction.[a]

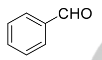
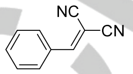
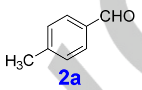
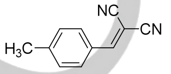
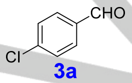
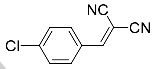
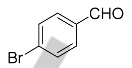
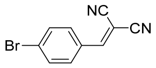
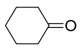
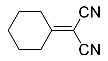


entry	catalyst (catalyst loading)	Solvent	Time	Yield[b]
1	**PAF-111-IL(OH)** (1.4 mol%)	CH_2Cl_2	1h	53%
2	**PAF-111-IL(OH)** (1.4 mol%)	MeOH	1h	62%
3	**PAF-111-IL(OH)** (1.4 mol%)	H_2O	40min	97%
4	**PAF-111-IL(OH)** (2.8 mol%)	H_2O	25min	97%
5	**PAF-111-IL(Cl)** (1.4 mol%)	H_2O	40min	49%
6	No catalyst	H_2O	40min	8%
7	**PAF-111-CH₂Cl** (10 mg)	H_2O	40min	9%
8[c]	The supernatant liquid of the H_2O suspension of **PAF-111-IL(OH)**		40min	8%

[a] Reaction conditions (unless other noted): A mixture of 0.80 mmol benzaldehyde, 0.80 mmol malononitrile and the catalyst in 4 mL of H_2O was stirred at room temperature for the indicated time. The catalyst loading was determined based on the elemental analysis: 2.80 wt% of nitrogen for **PAF-111-IL(Cl)** suggested 1.0 mmol g^{-1} of ionic liquid unit in the material and 3.08 wt% of nitrogen for **PAF-111-IL(OH)** suggested 1.1 mmol g^{-1} of ionic liquid unit in the material. [b] Isolated yield. [c] 10 mg **PAF-111-IL(OH)** was immersed in 4 mL of H_2O for 40min at room temperature. After filtration, to the liquid was added 0.80 mmol benzaldehyde and 0.80 mmol malononitrile, then the resulted mixture was stirred at room temperature for 40 min.

Table 2. PAF-111-IL(OH) catalysed Knoevenagel condensation reaction.[a]



entry	carbonyl compound	Product	Time	yield[b]
1	 **1a**	 **1b**	40min	97%
2	 **2a**	 **2b**	2.5h	96%
3	 **3a**	 **3b**	2h	96%
4	 **4a**	 **4b**	2.5h	93%
5	 **5a**	 **5b**	30min	95%

[a] Reaction condition: A mixture of 0.80 mmol carbonyl compound **1a-5a**, 0.80 mmol malononitrile and 10 mg **PAF-111-IL(OH)** (1.1 mmol g^{-1} of ionic liquid unit in the material which was calculated from the data of elemental analysis) in 4 mL of H_2O was stirred at room temperature for the indicated time. [b] Isolated yield.

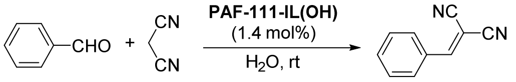
materials, the obvious decomposition of the frameworks could be observed at about 480-630 °C. The above analysis demonstrated the high thermal stability of the obtained PAF materials including the desired immobilized catalyst. Furthermore, **PAF-111-IL(Cl)** and **PAF-111-IL(OH)** could not be decomposed or dissolved in almost all common organic solvents and water, even in boiling water. It is very important that their excellent stability in water could lay a good foundation for the latter catalysis study in aqueous catalysis.

After the elaborate characterization of the desired **PAF-111-IL(Cl)** and **PAF-111-IL(OH)**, their catalytic properties were then studied. Knoevenagel condensation reaction, which is a highly effective organic C=C bond forming reaction and ofen used to test the catalytic activities of basic catalysts, was selected as the model reaction to investigate their catalytic performance. First, using benzaldehyde and malononitrile as model substrates, some control experiments were performed and the results were listed in Table 1. As shown in entries 1-3 of Table 1, among the screened solvents (CH_2Cl_2, MeOH and H_2O), H_2O gave the best reaction rate and yield of the current catalytic Knoevenagel condensation reaction. In other words, **PAF-111-IL(OH)** as catalyst could be used in aqueous system and gave a high catalytic activity under these conditions. The entries 3 and 4 of Table 1 demonstrated that increasing the catalyst loading from 1.4 mol% to 2.8 mol% could improve the reaction rate (Table 1).

It was found that **PAF-111-IL(Cl)** also could catalyse the current reaction, however, with a lower yield in the same conditions (Table 1, entry 5). **PAF-111-IL(Cl)** showed a lower catalytic activity than **PAF-111-IL(OH)** for the current reaction, which might be due to the weaker basicity of **PAF-111-IL(Cl)** in comparison with **PAF-111-IL(OH)**. In addition, the reaction could occur slowly in the absence of catalyst (Table 1, entry 6). Using **PAF-111-CH₂Cl** in the reaction system gave the same result with that in the absence of catalyst, indicating that **PAF-111-CH₂Cl** has not catalytic activity for this reaction. Results of the above investigations suggested that **PAF-111-IL(OH)** is indeed efficient catalyst for the Knoevenagel condensation reaction under aqueous conditions. The entry 8 of Table 1 clearly demonstrated that the supernatant liquid of the H_2O suspension of **PAF-111-IL(OH)** have no catalytic activity for the Knoevenagel condensation reaction. It indicated no leakage of catalytically active species from the **PAF-111-IL(OH)** catalyst during the catalysis process and thus the current catalytic reaction proceed via a heterogeneous process.

Under the above optimized conditions, using a series of carbonyl compounds and malononitrile as the reaction substrates, the catalytic performance of **PAF-111-IL(OH)** was further tested at a 1.4 mol% catalyst loading at room

Table 3. The recycle test of **PAF-111-IL(OH)** catalysed Knoevenagel condensation reaction.[a]



cycle	Time	yield[b]	cycle	Time	Yield[b]
1	40min	97%	6	40min	96%
2	40min	97%	8	40min	95%
4	40min	95%	10	40min	97%

[a] The reaction conditions and the recycle procedure could be found in the supporting information. [b] Isolated yield.

temperature. As shown in Table 2, various aromatic aldehydes with different substituent groups on the phenyl ring (Table 2, entries 1-4) could all react with malononitrile and give high isolated yields in considerably short reaction times. Besides the above aromatic aldehydes, the cyclohexanone could also give an excellent yield with a short reaction time (Table 2, entry 5) under the same conditions.

The recyclability, which is an important factor for a heterogeneous catalyst, of **PAF-111-IL(OH)** was then evaluated by subjecting **PAF-111-IL(OH)** to 10 cycles of the Knoevenagel condensation reaction of benzaldehyde and malononitrile. The experimental results were listed in Table 3. In each cycle, the reaction was driven to react for the same time. It was found that the catalytic activity of **PAF-111-IL(OH)** was very stable and after 10 cycles **PAF-111-IL(OH)** showed no loss of catalytic activity. After 10 cycles the FT-IR spectrum (Figure S6) of the recycled **PAF-111-IL(OH)** was measured and showed that there was no obvious change compared with the fresh **PAF-111-IL(OH).** The porosity of the recycled catalyst was aso evaluated by nitrogen adsorption–desorption isotherms (Figure S7). The BET surface area for the recycled **PAF-111-IL(OH)** was 520 m^2 g^{-1}. The slight decrease of the surface area might be due to that some reaction substrates or products were adsorbed in the pores of the catalyst. However, the high surface area of the recycled catalyst could demonstrate that the recycled catalyst still had high porisity.

In conclusion, through post-functionalization method, ionic liquids were introduced into the pores of **PAF-111** as catalytic sites, obtaing two PAF-supported ionic liquids named **PAF-111-IL(Cl)** and **PAF-111-IL(OH)**. **PAF-111-IL(OH)** exhibited high catalytic activity for Knoevenagel condensation reaction in aqueous system**.** Benefited by the outstanding stability of the frameworks of the PAF-based catalyst, **PAF-111-IL(OH)** showed excellent recyclability, that is, there was no activity loss in the current aqueous catalysis system after at least 10 cycles. This result demonstrates that PAF-based catalysts could perform excellently in aqueous system. This study presents an enticing prospect of using PAFs as an ultrastable platform to immobilize ionic liquids and will promote the application of PAF materials for aqueous catalysis.

Experimental Section

Details of the experimental procedures are provided in the Supporting Information.

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Conflict of Interest

The authors declare no conflict of interest.

Keywords: aqueous systems • ionic liquids • heterogeneous catalysis • Knoevenagel condensation • porous aromatic frameworks

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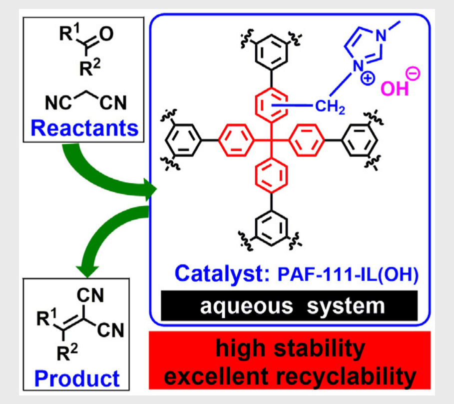
COMMUNICATION

Two new porous aromatic framework supported ionic liquids have been synthesized via a serious of post-functionalization reactions. Thereinto, **PAF-111-IL(OH)** is capable of catalyzing Knoevenagel condensation reaction in aqueous system with high catalytic activity and excellent recyclability.



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An Ionic Liquid on a Porous Organic Framework Support: A Recyclable Catalyst for the Knoevenagel Condensation in an Aqueous System