Synthesis of ZSM-5 zeolite from fly ash and its adsorption of phenol, quinoline and indole in aqueous solution

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Abstract
This work tries to synthesize ZSM-5 zeolite using fly ash (FA) by hydrothermal method and study the adsorption effect of the zeolite on phenol, quinoline and indole in aqueous solution. The zeolites were characterized with x-ray diffraction (XRD), scanning electron microscopy (SEM), x-ray fluorescence (XRF), fourier transformation infrared (FTIR) and N\textsubscript{2} adsorption-desorption isotherm. The characterization results showed that HZSM-5 zeolite was successfully synthesized. The higher mass ratio of sodium carbonate to Na\textsubscript{2}O in FA during melting is beneficial to improve the purity of ZSM-5 zeolite and its removal rate of organic matters. These results have been confirmed by XRD and principal component analysis (PCA). The adsorption process of phenol, quinoline and indole in aqueous can be well described with the double exponential kinetic model. The adsorption capacity of ZSM-5 zeolite for phenol, quinoline and indoles can be up to 24.41 mg g\textsuperscript{-1}, 35.99 mg l\textsuperscript{-1} and 34.05 mg g\textsuperscript{-1} respectively, and the removal rates can reach up to 82.80%, 84.86% and 83.20% respectively. The optimal pH value for adsorption ranges from 5 to 7.

1. Introduction

Fly ash (FA) is an industrial by-product derived from the boilers in coal-fired power plants, and typically composed of 90%–99% inorganic material, 1%–9% organic material and many other pollutants such as arsenic, cadmium, chromium, lead, mercury, and etc [1, 2]. The stock or landfill of FA not only occupies a lot of land, but also pollutes soil, water and air [3–5]. The chemical composition of inorganic material in FA mainly includes SiO\textsubscript{2} and Al\textsubscript{2}O\textsubscript{3}, and contains a small amount of Fe\textsubscript{2}O\textsubscript{3}, CaO, MgO, Na\textsubscript{2}O and the like [6, 7]. At present, the annual output of FA in China is more than 600 million tons, of which about 70% is utilized [8]. However, FA is mainly used as raw materials for cement, concrete and brick production, or for road construction and agricultural applications, while the high value-added utilization of FA is less [2].

In recent years, the high-value utilization of FA mainly includes the extraction of silica, alumina, rare metals and the synthesis of zeolite. Because the composition of FA is similar to zeolite, FA is widely used to synthesize zeolite. In our previous works, several zeolites were successfully synthesized from FA by hydrothermal method [6, 7]. Many researchers have reported the removal of pollutants such as arsenic [9, 10], radio cesium [11], lead and cadmium [12], fluoride [13], phosphate [14], ammonia ion [15], triphenylmethane dye, acid fuchsin [16] and crystal violet [17] from aqueous solution by zeolites synthesized from FA. In addition, the synthetic zeolite from FA was widely used to treat gaseous pollutants such as sulfur dioxide [18], nitric oxide, nitrogen dioxide, nitrous oxide, and carbon monoxide [19]. At present, there are many reports about the synthesis of ZSM-5 zeolite from FA [20–24]. ZSM-5 has high Si/Al ratio and is hydrophobic, and it has good affinity for organic compounds in wastewater. Therefore, ZSM-5 zeolite was used in the removal of organics in wastewater. The application of ZSM-5 in the adsorption of organic compounds, such as congo red [25], isopropanol [26], monobranched alkanes of naphtha [27] and methyl tert-butyl ether [28] have been reported.

Phenolic compounds and nitrogen containing heterocyclic compounds such as quinoline, indole and pyridine are toxic and carcinogenic substances which occur widely in the wastewater from coal gasification,
liquefaction and coking [29, 30]. This kind of wastewater belongs to refractory wastewater for individual biological treatment [30]. Advanced oxidation [31–35] and adsorption [36–38] were widely reported to treat this kind of refractory wastewater. Advanced oxidation has a better removal effect on refractory organics, but its cost is often higher, while adsorption process is not only simple and efficient, but also has a relatively low operating cost. In our recent work, the adsorption of phenol, quinoline and indole by ZSM-5 zeolite synthesized using the main chemical reagents of Na₂SiO₃, NaAlO₂ and tetraproply ammonium bromide (TPABr) has been briefly described [39]. It was found that ZSM-5 zeolite has good adsorption capacities of phenol, quinoline and indole in aqueous solution. Based on our previous work, in order to utilize FA and remove phenol, quinoline and indole from wastewater, this work intends to hydrothermally synthesize ZSM-5 zeolite using FA and investigate the adsorption effect of synthetic zeolite on three organic compounds in aqueous solution.

2. Experimental

2.1. Materials

FA was from a heating company in Tianjin, China. The crystal phases and chemical compositions of FA are shown in figure 3 and table 1 respectively. XRD showed FA contains quartz (SiO₂, PDF # 85–0798) and mullite (2SiO₂·3Al₂O₃, PDF #15–0776). Besides, there is a slight uplift in the range of 20°–30°, indicating that there is amorphous phase. The chemical compositions in table 1 show that FA should belong to Class C. The main chemicals utilized in this study are sodium hydroxide (NaOH, AR), sodium carbonate (Na₂CO₃, AR), hydrochloric acid (HCl, AR), tetrapropyl ammonium bromide (TPABr, 99%), phenol (C₆H₅OH, GC), quinoline (C₉H₇N, GC), indole (C₉H₇N, GC), sulphuric acid (H₂SO₄, AR), sodium dihydrogen phosphate (NaH₂PO₄·2H₂O), dibasic sodium phosphate (Na₂HPO₄, AR), methanol (CH₃OH, AR) and distilled water.

2.2. Preparation of zeolites

Firstly, FA was ground by a mechanical grinder and screened by a 100-mesh sieve. Then, the FA was evenly mixed with corresponding mass of Na₂CO₃ (the mass ratio of Na₂CO₃ to FA was 0.5, 0.8 and 1.2) and the corresponding samples were labeled as FA0.5, FA0.8 and FA1.2. After fully mixing, mixed material was put into muffle furnace and calcined at 800 °C for 2 h. After cooling, the material was grounded, and about 7M HCl aqueous solution was added into the material according to the solid mass/liquid volume ratio of 1(g):5(ml), and then the mixed material was heated at boiling state for 2 h. After cooling, the material was filtered, washed and dried for subsequent zeolite synthesis.

Zeolite was synthesized using tetrapropyl ammonium bromide (TPABr) as a template and the treated FA as Si and Al sources. Material compositions were listed in mass ratio as follows: 15 H₂O: 0.44 TPABr: 1 treated FA. pH was adjusted to about 11 with 0.1M NaOH and H₂SO₄ solution. The mixture was aged at room temperature for 24 h, then transferred to a hydrothermal reactor and reacted at 180 °C for 48 h. At the end of reaction, the solid was filtered and washed to about pH 9.0, and then dried at 105 °C. Finally, the product was calcined in a muffle furnace at 550 °C for 2 h. The synthetic pathway is shown in figure 1. The synthetic samples were recorded as ZSM-5(FA0.5), ZSM-5(FA0.8) and ZSM-5(FA1.2), in which 0.5, 0.8 and 1.2 refer to the mass ratio of Na₂CO₃ to FA during melting treatment.

2.3. Characterization

X-ray diffraction (XRD) of products was carried out with a D/Max-2500 x-ray diffractometer. S4Pioneer x-ray fluorescence (XRF) spectrometer was used to determine the chemical compositions of sample. Specific surface area and pore size were determined through N₂ adsorption/desorption isothermal tests (AutosorbIQ2-MP, USA). Morphologies were observed by S4800 scanning electron microscope (SEM) from Hitachi Company, Japan. Fourier transformation infrared (FTIR) was run on Nicolet 6700 infrared spectrometer (Nikoli instrument company, USA), the wavelength range is 400 cm⁻¹–4000 cm⁻¹, and resolution is 4 cm⁻¹.

| Samples | SiO₂ | Al₂O₃ | MgO | CaO | Fe₂O₃ | SO₃ | P₂O₅ | Si/Al |
|---------|------|-------|-----|-----|-------|-----|------|-------|
| Raw FA  | 34.40| 14.37 | 1.44| 22.94| 13.89 | 7.25| 1.27 | 4.07  |
| FA0.5   | 79.36| 6.86  | 0.22| 2.09 | 3.39  | 0.76| 1.19 | 19.68 |
| FA0.8   | 86.61| 2.60  | 0.13| 1.50 | 1.82  | 0.65| 1.19 | 56.62 |
| FA1.2   | 85.38| 2.80  | 0.16| 1.71 | 2.72  | 0.66| 1.09 | 51.85 |
2.4. Adsorption experiments of organics

A certain volume and concentration of the mixed solution of phenol, quinoline and indole was added to a conical flask and the conical flask was placed in a water bath constant temperature oscillator. After reaching the set temperature, zeolite samples were added into the conical flask, and the filtrate was obtained by 0.45 μm filter membrane after a certain time. The filtrate sample was diluted, and then HPLC (High Performance Liquid Chromatography) was used to determine the residual organic matter concentration. The corresponding analytical methods have been reported in our early work [39]. The removal rate of phenol, quinoline and indole can be calculated by equation (1).

\[ y = \left( \frac{c_0 - c_t}{c_0} \right) \times 100\% \]  

(1)

Where \( y(\%) \), \( c_0(\text{mg l}^{-1}) \) and \( c_t(\text{mg l}^{-1}) \) represent removal rate, initial organic concentration and organic concentration at \( t \) moment, respectively. Adsorption capacity was calculated by equation (2).

\[ q_t = \left( \frac{c_0 - c_t}{w} \right) V \]  

(2)

Where \( q_t(\text{mg g}^{-1}) \), \( V(\text{L}) \) and \( w(\text{g}) \) respectively expressed as adsorption capacity, solution volume and adsorbent weight.

2.5. Principal component analysis

In order to investigate the influence of the mass ratio of sodium carbonate to FA during melting treatment on the adsorption property of synthetic zeolite for phenol, quinoline and indole in simulated wastewater, principal component analysis (PCA) was done using the data of removal rate. The principal component analysis was carried out by MATLAB software, and the corresponding formula of PCA was reported by Ni et al [40].

3. Results and discussion

3.1. Influence of pretreatment on FA

After alkali fusion and acid leaching, the XRD for FA samples are shown in figure 2. It is found that the increase of the mass ration of sodium carbonate to FA can reduce quartz content. When the mass ratio of sodium carbonate to FA is 0.5 or 0.8, the x-ray diffraction peaks of quartz (2\( \theta \) = 20.86° and 26.64°) in treated sample are clear. When the mass ratio of sodium carbonate to FA is 1.2, the x-ray diffraction intensity of quartz is greatly weakened. These results prove that the melting treatment of sodium carbonate is conducive to the transformation of quartz in FA.

The XRF results of samples are shown in table 1. It shows that the impurity such as Fe, Mg, Ca, S and P decreased after alkali fusion and acid leaching. At the same time, the Si to Al molar ratio is enhanced from about 4.07 to over 19.68. This phenomenon is beneficial to the synthesis of ZSM-5 zeolite.

3.2. Characterization of zeolites

Using the treated FA as raw materials, the XRD results of hydrothermally synthesized products are shown in figure 3. It can be seen that zeolites synthesized with the three kinds of treated FA all have typical MFI characteristic peaks near 2\( \theta \) = 7.7°, 8.8°, 23.10° and 23.65° [41]. The analysis of jade 6.0 indicates that the XRD
peaks of the product are well matched with HZSM-5 zeolite (PDF#49-0657). The SiO₂ characteristic peaks of the products synthesized with the FA treated under 0.5 or 0.8 mass ratio of Na₂CO₃ to FA are clear. However, the zeolite synthesized with high alkali treated FA has no clear diffraction peaks for quartz. The diffraction peak of quartz in zeolite products should be due to the fact that quartz in the raw material may not participate in the synthesis of zeolite. The results show that the alkali fusion treatment of FA has a significant effect on the purity of synthetic zeolite.

The SEM of zeolites synthesized with different FA samples are shown in figure 4. The zeolite particles have clear three-dimensional block shape. Its size is about 10 μm and are different from those reported by Feng et al [42], but is similar to that of traditional ZSM-5 zeolite reported by Li et al [43].

### 3.3. PCA analysis of organic adsorption

In order to find out the best zeolite for further study on the adsorption of phenol, quinoline and indole in aqueous solution, adsorption experiments of single organic matter (namely phenol, quinoline and indole) were designed. The experimental conditions are as follows: the concentration of organic matter is 250 mg l⁻¹, the temperature is 25 °C, the adsorption time is 4 h, the dosage of zeolite is from 2 to 15 g l⁻¹, and the adsorbents are
ZSM-5 (FA0.5), ZSM-5 (FA0.8) and ZSM-5 (FA1.2), respectively. The removal rate of different zeolites for three organic compounds is shown in figure 5. It can be seen that the synthetic zeolite can effectively remove phenol, quinoline and indole from aqueous solution.

Because of the different zeolite dosage and various organics, it is difficult to intuitively judge the zeolites with the highest comprehensive adsorption capacity for the three organic compounds. Therefore, in this work,
principal component analysis was used to calculate the comprehensive adsorption performance of different zeolites for the three organics and the results are shown in Table 2. Clearly, the score of ZSM-5(FA1.2) is the highest, thus ZSM-5(FA1.2) has the best comprehensive adsorption capacity for phenol, quinoline and indole in aqueous solution. When the concentration of zeolite is 2 g l\(^{-1}\), the adsorption capacities of ZSM-5(FA1.2) for phenol, quinoline and indoles are 24.41 mg g\(^{-1}\), 35.99 mg l\(^{-1}\) and 34.05 mg g\(^{-1}\), respectively. Thus, the following work mainly focuses on ZSM-5(FA1.2) zeolite. Based on our previous research report [39], under the same adsorption experimental conditions, the adsorption capacities of ZSM-5 synthesized by pure chemical reagents for phenol, quinoline and indole are 43.54 mg g\(^{-1}\), 83.10 mg g\(^{-1}\) and 69.51 mg g\(^{-1}\), respectively, and is clearly higher than those of ZSM-5 synthesized by fly ash.

### 3.4. BET, XRF and FTIR for ZSM-5(FA1.2)

Figure 6 is N\(_2\) adsorption/desorption curves for ZSM-5(FA1.2). The N\(_2\) adsorption/desorption isotherm belongs to typical type I curves (Langmuir isotherms) and has H4 loop lag, indicating that the sample has microporous characteristics. The pore volume and equivalent BET specific surface area for ZSM-5(FA1.2) are showed in Table 3. In addition, the chemical compositions of the zeolite tested by XRF is also shown in Table 3. The ratio of silicon to aluminum of the product is greater than 27, which proves that the product belongs to high silicon zeolite.

The FTIR results of ZSM-5(FA1.2) is shown in figure 7. The characteristic skeleton vibration peaks of ZSM-5 zeolite are around 1225 cm\(^{-1}\), 1093 cm\(^{-1}\), 790 cm\(^{-1}\), 550 cm\(^{-1}\) and 450 cm\(^{-1}\) [44]. The FTIR spectrogram for ZSM-5(FA1.2) is consistent with those reported by Pan et al [44]. Besides, there is a stretching vibration peak of water molecules near 1625 cm\(^{-1}\) and an O–H vibration peak at 3446 cm\(^{-1}\), which are caused by water in the zeolite.

#### Table 2. Comprehensive score of organic adsorption by the three zeolites.

| Zeolites     | ZSM-5(FA0.5) | ZSM-5(FA0.8) | ZSM-5(FA1.2) |
|--------------|--------------|--------------|--------------|
| Score        | 81.08        | 126.32       | 133.47       |

#### Table 3. Chemical compositions and surface properties for ZSM-5(FA1.2).

|          | SiO\(_2\) (wt\%) | Al\(_2\)O\(_3\) (wt\%) | CaO (wt\%) | Fe\(_2\)O\(_3\) (wt\%) | P\(_2\)O\(_5\) (wt\%) | SO\(_3\) (wt\%) | Si/Al | Pore volume (cm\(^3\) g\(^{-1}\)) | \(S_{\text{BET}}\) (m\(^2\) g\(^{-1}\)) |
|----------|------------------|-------------------------|------------|-------------------------|----------------------|----------------|-------|----------------------------------|-------------------------------------|
| ZSM-5(FA1.2) | 83.86            | 5.20                    | 1.92       | 2.01                    | 1.22                 | 0.32           | 27.38 | 0.156                              | 310.11                               |
3.5. Organic adsorption by ZSM-5 (FA1.2)

The change trends of solution concentrations during the adsorption of phenol, quinoline and indole by ZSM-5 (FA1.2) zeolite are shown in figure 8. The experimental conditions are: 500 mg l\(^{-1}\) total initial concentration (namely \(c_{\text{phenol}} = 300\) mg l\(^{-1}\), \(c_{\text{quinoline}} = 150\) mg l\(^{-1}\) and \(c_{\text{indole}} = 50\) mg l\(^{-1}\)), 10 g l\(^{-1}\) of zeolite concentration and 25 \(^\circ\)C. The concentration for each component decreased rapidly in 5 min and then remained almost stable, indicating that the adsorption of phenol, quinoline and indole by ZSM-5 is a fast and physical process. After 100 min, the removal rates of phenol, quinoline and indole are about 24.08\%, 61.90\% and 44.70\%, respectively and the total removal efficiency of three organic compounds in simulated wastewater is about 37.50\%.

According to the data in figure 8 and the dosage of adsorbent (10 g l\(^{-1}\)), the relationship between adsorption capacity and time can be obtained and shown in figure 9. The adsorption capacity data of phenol, quinoline and indole are respectively fitted by the pseudo-first-order, pseudo-second-order and double exponential kinetic models [45].

\[
pseudo - first - order: \quad q_t = q_e [1 - \exp(-k_t t)] \\
\]
\[
pseudo - first - order: \quad q_t = \frac{k_t q_e^2 t}{1 + k_t q_e t} \\
\]
Where \( q_e \) (mg g\(^{-1}\)) and \( k_1 \) (min\(^{-1}\)) are the adsorption capacity at the equilibrium time, the pseudo-first-order model constant and the pseudo-second-order model constant, respectively. \( D_1 \) (mg l\(^{-1}\)) and \( D_2 \) (mg l\(^{-1}\)) = constants of rapid and slow steps; \( k_{d1} \) (min\(^{-1}\)) and \( k_{d2} \) (min\(^{-1}\)) = constants controlling the mechanism; \( m \) (g l\(^{-1}\)) = absorbent concentration; \( t \) (min) = time.

The values of the parameters for these kinetic models are all greater than 0. Using MATLAB software and nonlinear optimization technology, the fitting effect is shown in figure 9 and the model parameters are shown in table 4. For the three kinetic models, the adsorption process of phenol, quinoline and indole by the synthetic zeolite is more in line with the double exponential kinetic model and the corresponding regression index \( R^2 \) for phenol, quinoline and indole are all greater than 0.99. The results show that the adsorption of phenol, quinoline and indole by ZSM-5 (FA1.2) can be divided into two stages: fast stage and slow stage. These phenomena are consistent with the experimental data (seen in figure 8). The regression effect of the pseudo-first-order kinetic model is the worst in the three kinetic models. The equilibrium adsorption capacities obtained by the three kinetic models are close to those measured by experiments. Except the relative error between the equilibrium adsorption capacity of the first-order kinetic model of indole and the experimental value is about 8.2%, the other relative errors are all less than 5%.

Figures 10(a) and (b) show that the dosage of ZSM-5 and the total initial concentration of organic matter have an impact on removal rate. When temperature is 25 °C, the total organic concentration \( C_{phenol}:C_{quinoline}:C_{indole} = 6:3:1 \) is 150 mg l\(^{-1}\) and adsorption time is 4 h, the removal rate of organics increases with the rise of
When the zeolite dosage is 15 g l\(^{-1}\), the removal rates of phenol, quinoline and indole can reach to 82.80%, 84.86% and 83.20% respectively. The removal rate of organic matter decreased with the increase of the initial concentration (figure 10(b)). If the total initial concentration is 250 mg l\(^{-1}\), the removal rates are only about 33.68% for phenol, 56.31% for quinoline and 38.20% for indole, respectively.

Figure 11 shows the effect of pH on the removal rate of organics. The total concentration is 200 mg l\(^{-1}\) (\(c_{\text{phenol}} : c_{\text{quinoline}} : c_{\text{indole}} = 6 : 3 : 1\)) and the adsorbent dosage is 5 g l\(^{-1}\). The adsorption experiments were run at 25 °C for 4 h. The results show that pH has important influence on organics adsorption. For quinoline, if pH is less than 7, removal rate increases with the increase of pH, and when pH is greater than 7, removal rate decreases sharply with the increase of pH. If pH = 7, removal rate is about 60.98%. For phenol and indole, if pH is less than 5, removal rates increase with the increase of pH, and when pH is greater than 5, removal rates decrease with the increase of pH. If pH = 5, removal rates are about 41.98% for phenol and about 34.73% for indole, respectively. It can be seen that the pH range of 5–7 is favorable for the adsorption of mixed organics of phenol, quinoline and indole in aqueous solution. Strong acidic or alkaline environment is not conducive to the adsorption of phenol, quinoline and indole by ZSM-5 zeolite.

4. Conclusions

The main conclusions can be got as follows.
(1) Alkali fusion and pickling of FA are beneficial to the synthesis of ZSM-5 zeolite. Moreover, during melting the higher mass ratio of sodium carbonate to FA is beneficial to the synthesis of high purity ZSM-5 zeolite.

(2) The principal component analysis shows that ZSM-5 zeolite synthesized from the FA treated under higher mass ratio of sodium carbonate to FA has better comprehensive adsorption capacity for phenol, quinoline and indole.

(3) The adsorption process of phenol, quinoline and indole in aqueous solution by ZSM-5(FA1.2) zeolite are in good agreement with the double exponential kinetic model, and ZSM-5(FA1.2) zeolite has better adsorption capacity and removal rate for phenol, quinoline and indole.

(4) The pH range of 5–7 is favorable for the adsorption of mixed organics of phenol, quinoline and indole by ZSM-5(FA1.2) zeolite.

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