

Effective Adsorption of Nutrients from Simulated Domestic Sewage by Modified Maifanite

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Research Article

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Abstract

Modified maifanite (MMF) was prepared by synthesized method with sulfuric acid treatment and high temperature calcination, and evaluated as an effective adsorption material to remove the nutrient salt in waste watery. Compared with the raw maifanite (RMF), the MMF exhibited the higher adsorption capacity and higher removal efficiency. The results showed that the adsorption rates of total phosphorus (TP), total nitrogen (TN), ammonia nitrogen (NH₃-N), nitrate nitrogen (NO_x-N) and Chemical Oxygen Demand (COD) by MMF (RMF) were 86.7% (76.7%), 44.9% (34.5%), 29.1% (20.8%) and 79.8% (13.0%) respectively at 20 °C for 24 h. MMF kept the basic structure and composition of maifanite with stronger surface roughness and more adsorption active sites. This study suggests that MMF can be further applied to treat domestic sewage and eutrophic water.

Highlights

- MMF (acidification-calcination modification) was prepared and characterized.
- MMF exhibited a higher adsorption capacity than RMF.
- The mechanism of MMF on pollutants removal was mainly chemical adsorption.
- MMF could be further applied to treat domestic sewage and eutrophic water.

1. Introduction

Since the early 1960s, more and more attention has been paid to the water environmental problems caused by eutrophication (Lin et al., 2021; Heisler et al., 2008; Kemp et al., 2005). In recent years, excessive discharge of N and P into natural freshwater bodies led to serious eutrophication, which may further lead to harmful algal blooms and deterioration of water quality (Reddy et al., 2018a; Withers et al., 2017). To reduce the content of N and P, various techniques and materials have been developed, including biological removal process (Pang et al., 2019c; Arun et al., 2019a; Liu et al., 2007), oxygen enriched aeration (Schierholz et al., 2006; Sahoo and Luketina, 2006; Sahoo and Luketina, 2005; DeMoyer et al., 2003b), chemical precipitation (Donnert and Salecker, 1999; Penetra et al., 1999; Schindler, 1974), flocculation (Hu et al., 2003c; Desjardins et al., 2002) and adsorption (Huang et al., 2014; Hano et al., 1997), etc. Among these techniques, adsorption is usually regarded as an effective and attractive approach. Selecting an adsorbent with high adsorption capacity and Eco-friendly an important step for the adsorption approach.

Maifanite is a kind of layer silicate mineral, abundant in China, low price, the non-toxic (Han et al., 2020; Chen et al., 2019b) and has a network structure with large area of honeycomb pores. Maifanite can release some beneficial elements such as K, Na, and Fe needed by organisms. This makes maifanite become a great potential as an eco-friendly material for the restoration of freshwater lakes (Liu et al., 2020). However, the specific surface area and porosity of the natural mineral maifanite are limited because of the high impurity and the pore blockage. In addition, the adsorption sites of surface oxides are separated from pollutants, which makes the adsorption efficiency not high. In view of the above shortcomings, the modification of maifanite can greatly improve its adsorption capacity and application. Maifanite modified by chitosan and mercaptan group can adsorb mercury and methylmercury more effectively than natural maifanite (Wang et al., 2020). In order to promote the removal of Cr (VI) by microorganisms, zinc series layered double hydroxides were loaded on maifanite (Gao et al., 2020). The adsorption capacity of zinc layered double hydroxides modified maifanite made by Chen et al. was 3.5 times that of natural maifanite for Cd (II) (2019b). The fluoride in water can be effectively removed by the composite modified maifanite adsorption membrane of activated alumina and polyvinylidene fluoride (Meng et al., 2020). The arsenic removal of titanium dioxide modified maifanite can reach the drinking water standard at pH = 5–7 (Zhang et al., 2016c). However, most of studies focused on the removal of heavy metals by maifanite and few of the reports investigated on the removal of N and P by maifanite, especially MMF. In this paper, MMF samples were prepared using compound modification method of acidification process (2.5 mol/L H₂SO₄) and calcination (400°C) (Liu et al., 2018b). Batch experiments were

conducted to identify the effect of adsorbent dose, dynamic adsorption time, and static adsorption time on the removal of N and P by maifanite in simulated domestic sewage.

2. Materials And Methods

2.1. Modified mineral materials

The raw maifanite (RMF) granules, were obtained from Kangyuan Maifanite Products Factory (Shandong, China), which have a bluish gray color, and the density is 2.7 g/cm^3 . The RMF granules were soaked in deionized water for 10 h, then washed to neutral state. After that, the samples were dried at 105°C for 24 h. Finally, the samples were cooled to room temperature for further studies. The MMF was synthesized by the acidification method combined with calcination method. The RMF granules were mixed with $2.5 \text{ mol/L H}_2\text{SO}_4$ solution in 1:3 liquid/solid ratio at 90°C in the thermostatic water bath for 2 h, and calcined at 400°C for 2 h to enhance its sorption capacity according to the method proposed by our previous study (2018b), named as MMF.

2.2. Experimental and analytical equipment

TP, TN, $\text{NH}_3\text{-N}$, $\text{NO}_x\text{-N}$ and COD were measured by ultraviolet spectrophotometer (DR4000/U, HACH company, USA). XRD patterns of maifanite samples were obtained by Cu K_α radiation (30 kV, 15 mA) in the range of $(2\theta) 5 \sim 65^\circ$. In the wavenumber range of $400 \sim 4000 \text{ cm}^{-1}$, Fourier transform infrared (FTIR) spectra of maifanite samples were measured by Fourier transform infrared spectrometer (Nicolet 6700, USA). X-ray photoelectron spectroscopy (XPS) (ESCALAB 250XI, USA) was used to measure the valence and content of surface elements in maifanite. Scanning electron microscope (SEM) (JSM-5610LV, Japan) was used to obtain images at 2000 times. The chemical composition of maifanite was determined by X-ray fluorescence (XRF) (RU-200B/D/MAX-RB RU-200B, Japan). The cation exchange capacity (CEC) of maifanite was determined by $\text{NH}_4\text{Cl-NH}_4\text{OAC}$ method. Specific surface area and porosity analyzer (ASAP 2020M USA) were used to determine the specific surface area and total porosity of maifanite granules. The point of zero charge (pH_{pzc}) of maifanite was determined by the batch equilibrium method.

2.3. Adsorption experiments

In this experiment, artificial solution was used to simulate domestic wastewater. A series of dynamic and static adsorption experiments were conducted to test the adsorption effect of RMF and MMF on N and P in simulated domestic sewage. 150 mL domestic sewage was added into 250 mL conical flasks, a certain amount of RMF and MMF were added, respectively, and then 3 drops of trichloromethane were added to prevent microbial activity.

2.3.1. Dosage test

1 g, 2 g, 4 g and 8 g of adsorbents were added into the conical flask containing wastewater respectively, and the adsorbents were continuously shaken in a shaking table at $20 \pm 2^\circ\text{C}$ for 24 h under 200 rpm. A certain amount of supernatant was extracted and centrifuged at 4000 rpm for 10 minutes, then the concentration changes of nitrogen, phosphorus and COD in the supernatant were determined.

2.3.2. Dynamic adsorption test

The sample was put into a constant temperature shaker at $20 \pm 2^\circ\text{C}$ for 24 h at 200 rpm. A certain amount of supernatant was extracted and centrifuged at 4000 rpm for 10 min, and then the concentration changes of N, P and COD in the supernatant were determined.

2.3.3. Static adsorption test

The sample was placed at $20 \pm 2^\circ\text{C}$ for 0, 1, 2, 4, 8, 16 and 32 h to extract a certain amount of supernatant, and centrifuged according to the conditions in the dynamic adsorption test to determine the concentration of N, P and COD.

3. Results And Discussion

3.1. Dosage test

Figure 1 showed the adsorption of 5 indicators in wastewater by MMF and RMF. When the solid-liquid ratio of medical stone to wastewater was 4:75, the adsorption effect was the best. In order to meet economic benefits, this solid-liquid ratio was used in both static and dynamic tests.

3.2. Dynamic and static adsorption test

The static and dynamic adsorption relationship of MMF and RMF on simulated artificial domestic sewage was shown in Fig. 2. It could be seen that the adsorption effect of MMF was better than RMF, and the adsorption rates of MMF and RMF were faster in the first 5 hours, and then the speed gradually slowed down, and the adsorption capacity gradually decreased until the equilibrium was reached. Figure 3 showed that the adsorption equilibrium concentrations (q_e) from high to low were dynamic MMF > static MMF > Dynamic RMF > static RMF, which indicated that the adsorption capacity of MMF for pollutants was the highest in the stirred environment. The adsorption of maifanite to pollutants conformed to the pseudo second-order kinetic model ($R^2 > 0.99$), indicating that the adsorption process of maifanite to pollutants was mainly chemical adsorption. The adsorbents corresponding to the respective maximum reaction equilibrium constants (K_2) of TP, TN, $\text{NH}_3\text{-N}$, $\text{NO}_x\text{-N}$ and COD were dynamic MMF, static RMF, static RMF, dynamic MMF and static RMF. Therefore, under dynamic conditions, the adsorption equilibrium of MMF for TP and $\text{NH}_3\text{-N}$ can be reached first, while under static conditions, the adsorption equilibrium time of RMF for TN, $\text{NO}_x\text{-N}$ and COD was shorter.

3.3. Characterization of MMF and RMF

3.3.1. X-ray diffraction

Figure 4 showed that the main components of maifanite used for adsorption are quartz and feldspar. The characteristic peaks displayed by MMF and RMF are basically the same, indicating that the modification of RMF did not cause damage to the skeleton structure, and that the calcination temperature of 400 °C was a reasonable modification condition. The characteristic peak heights of RMF were higher than MMF, and some peaks even disappeared, indicating that sulfuric acid acidification and high-temperature roasting can remove internal impurities in maifanite, and the water in the components was lost after decomposition and volatilization. These changes explained the increase in adsorption capacity of maifanite. Using the Bragg equation $n\lambda = 2d \sin \theta$ (Table 1), it was found that the modification conditions had a great effect on the interlayer spacing.

3.3.2. FTIR

The FTIR spectra (Fig. 5) showed that the absorption peaks of RMF at 3430 cm^{-1} (O-H group (Acosta et al., 2003a)), 1035 cm^{-1} (PO_4^{3-} (Gogoi et al., 2015)), 776 cm^{-1} , (Mg-Fe-OH group (Chinoune et al., 2016a)) and 588 cm^{-1} , (Fe-O group (Tsai et al., 2007 ; 2003a)) were respectively shifted to 3423 cm^{-1} , 1032 cm^{-1} , 777 cm^{-1} , 589 cm^{-1} after modification. The absorption peak of O-H group in MMF was smaller than that in RMF, and the relative position difference was obvious, which indicated that during the modification process, water molecules on the surface of maifanite vibrate and chemical reaction took place, Acid treatment resulted in protons entering into maifanite and eroding O-H group, which was consistent with previous studies (Komadel, 2016b, Rodriguez et al., 1995). According to the comparison between RMF and MMF, PO_4^{3-} extended, but the absorption peaks of Mg-Fe-OH group and Fe-O group did not change obviously.

3.3.3. XPS

Compared with RMF, the peak of $\text{Al}^{3+}2p$ is added in the XPS (Fig. 6) of MMF, which indicated that the structure type of Al increased and the binding morphology changed after acid treatment and high temperature treatment, both of them were

mainly in the form of Al_2O_3 (Tago et al., 2017; Li et al., 2013). In RMF, Fe^{2+} was the main component of Fe, and Fe^{3+} only accounted for 11.3%. In MMF, Fe existed in the form of Fe_2O_3 (Wang et al., 2017; Smolders et al., 2017), which promoted the oxidation of maifanite. Fe^{2+} was oxidized to Fe^{3+} , and $\text{Fe}^{3+}2\text{p}_{1/2}$ was the most. Compared with RMF, MMF added CaTiO_3 , accounting for 42.6%. In the modification process, part of the carbonate in CaCO_3 was lost in the form of CO_2 , and Ca^{2+} is combined with a small amount of Ti in MMF, which hindered the loss of Ti. It could also be found from Table 1 that the proportion of Ti increased.

3.3.3. SEM

The SEM images of RMF and MMF were depicted in Fig. 7. As shown in Fig. 7, the maifanite was a multi-layered structure. The surface of maifanite had finely broken packets and small bulges. It indicated that the basic structure of the maifanite has not been severely damaged after modification.

However, compared to RMF, which had a flatter surface and smoother edges, MMF was mainly manifested by greater fragmentation and depression on the surface, more cracks, debris pockets and holes. Sulfuric acid modification enhanced the particle gap, which was consistent with the previous study (Ferrazzo et al., 2020). Simultaneously, uneven edges with irregular fractures appeared due to acid and roasting reasons. All of these changes increased the surface area of maifanite, and provided more adsorption sites for pollutants adsorption.

3.3.4. Chemical compositions

The specific gravity of Al, Ca, Fe, Na, Mg and P in MMF was lower than that in RMF (Table 1), The review of acid modified aluminosilicate minerals by Komadel also concluded that although the modification method eroded by sulfuric acid will lead to a large number of metal cations loss, it was conducive to enhance the physical and chemical properties of clay minerals adsorption (2016b), and high temperature can activate cations (Jones et al., 2004). However, the specific gravity of SiO_2 increased, which indicated that acid modification had little effect on the dissolution of SiO_2 and did not change the basic composition and structure of maifanite. Although acid modification made the metal oxides that contribute to the removal of anionic pollutants dissolved the maifanite in the form of ions, it also greatly enhanced the adsorption capacity of cationic pollutants such as $\text{NH}_3\text{-N}$. Part of the impurities dissolved out, which was beneficial for the maifanite to expose the active sites for adsorbing anions and form the Lewis acid center, which improved the removal of domestic wastewater pollutants by the maifanite to a certain extent. High-temperature roasting increased the micro cracks and voids on the surface of the maifanite without changing the main components of the maifanite, and expand the contact area between the pollutants and the maifanite.

3.3.5. Structure properties

The pH_{pzc} of MMF was higher than RMF (Table 1) which indicated that MMF was easier to adsorb phosphate and $\text{NO}_x\text{-N}$ in neutral environment. The cation exchange capacity of MMF was larger, which was helpful for MMF to remove cation pollutants from wastewater by cation exchange. The specific surface area of MMF was about 12 times that of RMF, and the interlayer spacing of MMF increased by 0.64 nm. These changes provided sites for adsorption of more pollutants, and larger volume of organic pollutants could enter maifanite more easily through larger pores and interlayer gaps. Therefore, the structure properties of MMF were better than RMF in the adsorption of domestic wastewater pollutants.

Table 1 The main chemical compositions and structure properties of RMF and MMF, respectively.

Chemical composition								Structure property			
Sample	SiO ₂	Al ₂ O ₃	Na ₂ O	CaO	Fe ₂ O ₃	MgO	TiO ₂	pH _{PZC}	CEC (meq/100g)	d ₀₀₁ (nm)	S _{BET} (m ² /g)
RMF	61.38	15.84	5.16	5.08	3.18	1.82	0.39	5.26	9.89	3.07	3.57
MMF	62.44	15.47	5.31	4.41	2.88	1.54	0.43	8.24	37.26	3.73	42.61

4. Conclusions

The main conclusion drawn from this study are: (1) The nutrients adsorption capacity of MMF was higher than RMF. (2) The adsorption capacity of maifanite was enhanced with the increasing of maifanite dosage. (3) Without destroying the basic structure of maifanite, the modifier can make maifanite have larger specific surface area and more active sites for pollutants adsorption. (4) The removal of pollutants from simulated wastewater by MMF was mainly carried out by chemical adsorption, and the adsorption equation can be obtained by pseudo second order kinetics fitting. (5) The cation capacity and electronegativity of MMF were higher than those of RMF, and the proportion of ion exchange and electrostatic adsorption of MMF increased.

Declarations

Ethics approval and consent to participate

Not applicable.

Consent to publish

Not applicable.

Authors' contributions

Zisen Liu and Yi Zhang had the ideas for the article. Yilingyun Zou, Yunli Liu, Feng Luo, Rou Wang and Yi Zhang conducted literature search and data analysis, and Zisen Liu, Yilingyun Zou, Yi Zhang and Zhenbin Wu drafted and/or critically revised the works. All authors read and approved the final manuscript.

Availability of data and materials

All data generated or analysed during this study are included in this published article.

Competing interests

The authors declare no competing interests.

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Figures

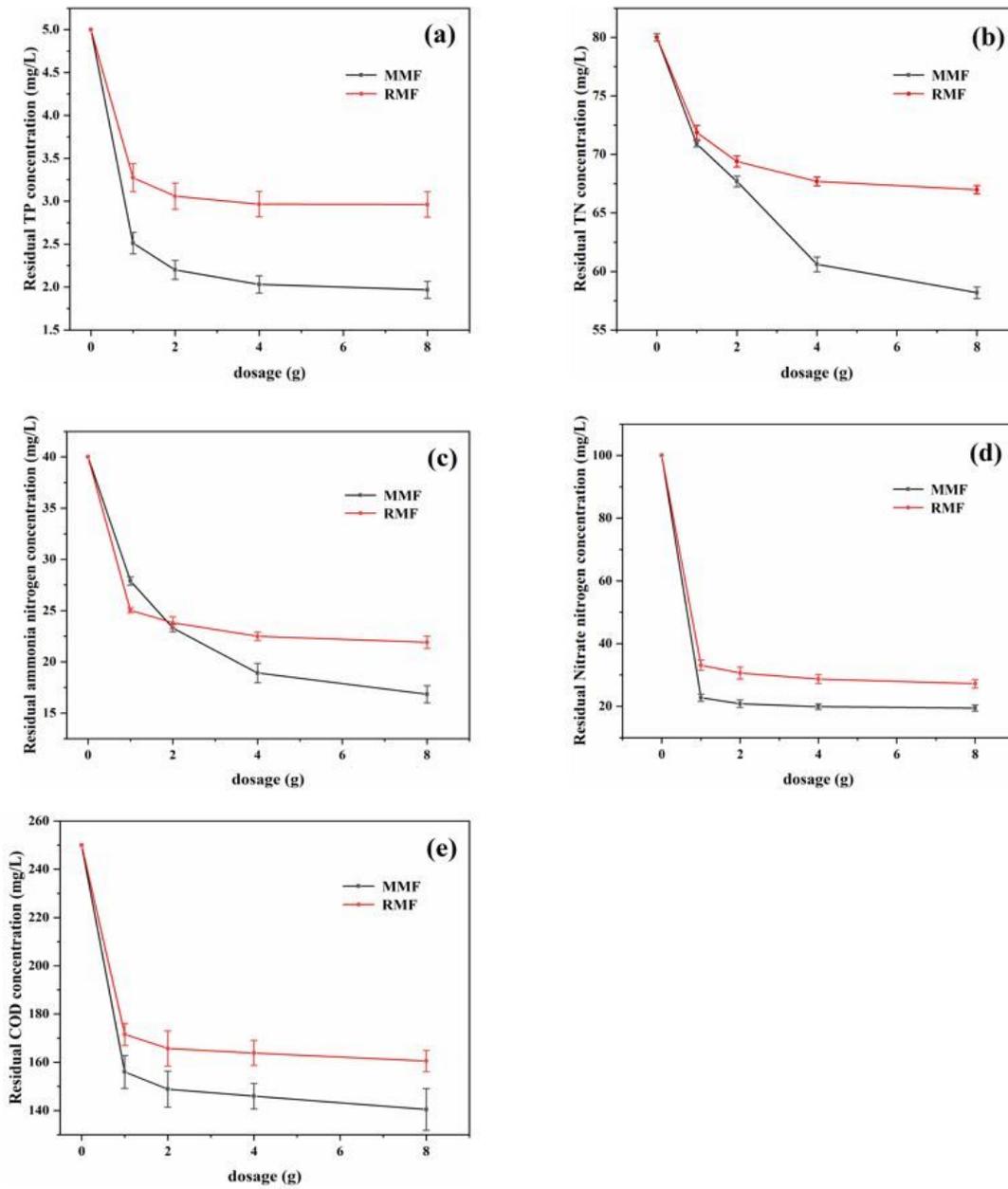


Figure 1

The relationship between the dosage of MMF and RMF and the adsorption capacity of pollutants ((a) TP, (b) TN, (c) NH₃-N, (d) NO_x-N, and (e) COD).

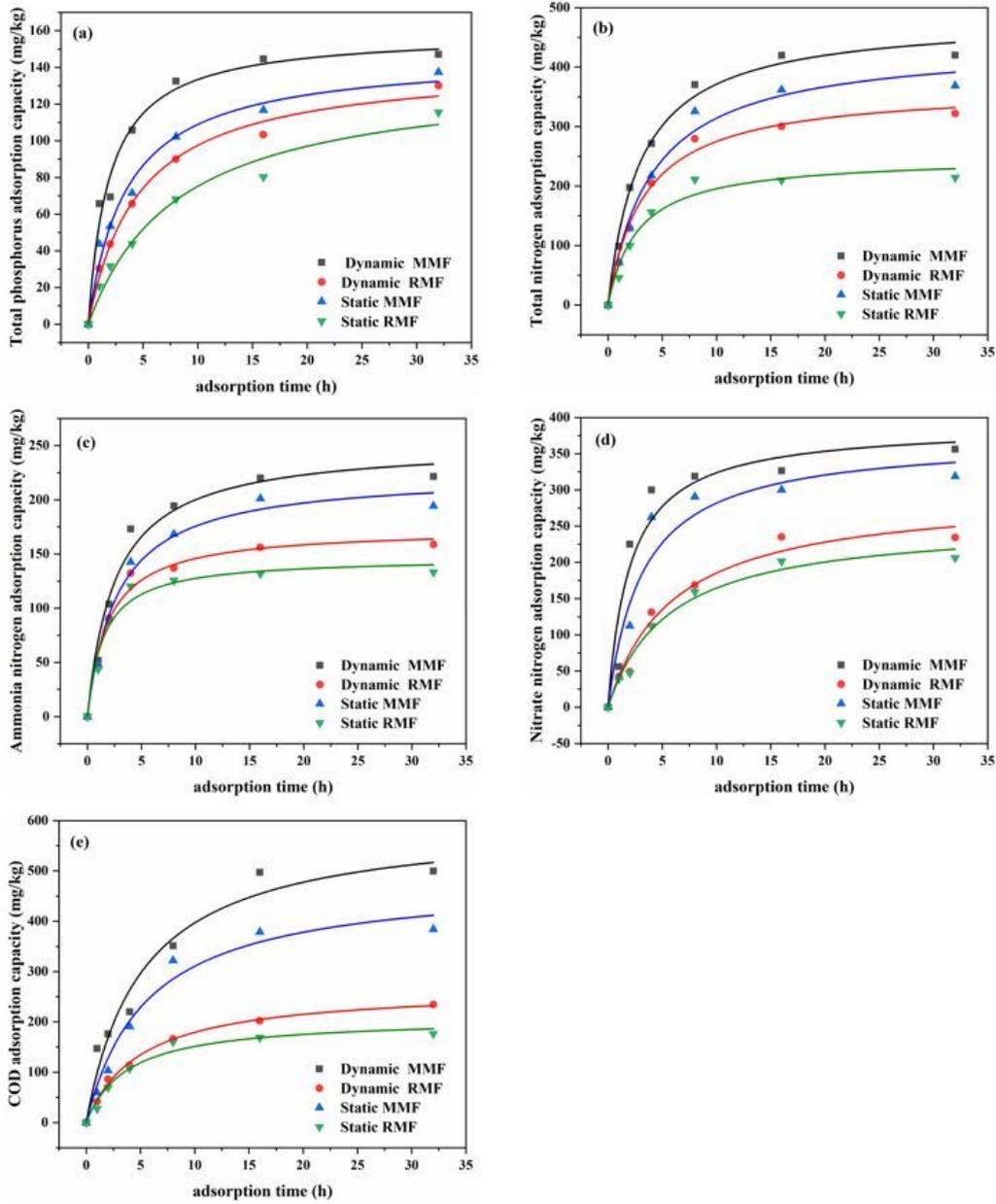


Figure 2

The relationship between the dosage of MMF and RMF and the concentration of pollutants ((a) TP, (b) TN, (c) NH₃-N, (d) NO_x-N, and (e) COD).

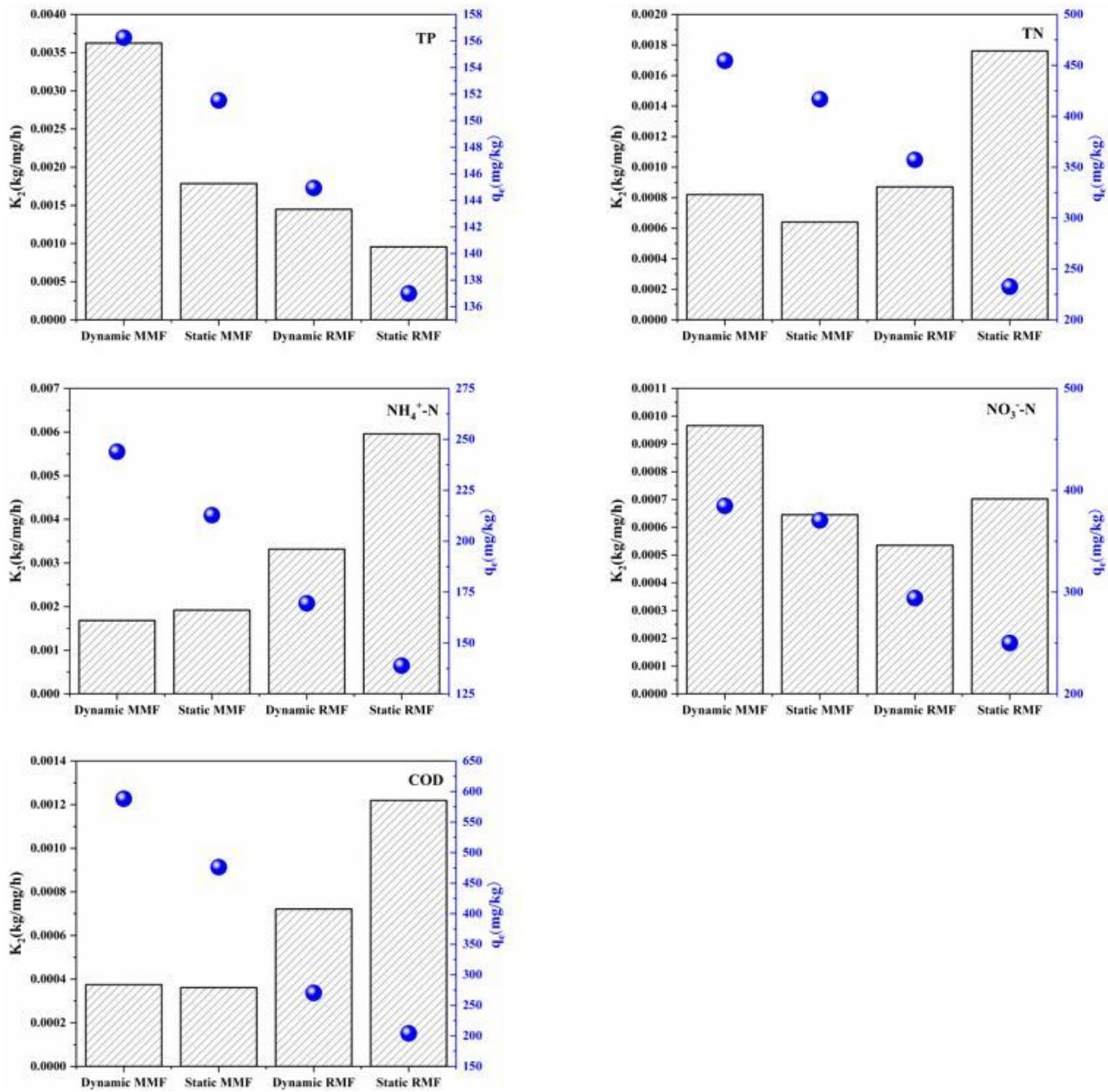


Figure 3

Static and dynamic adsorption relationship of MMF and RMF on simulated artificial domestic sewage.

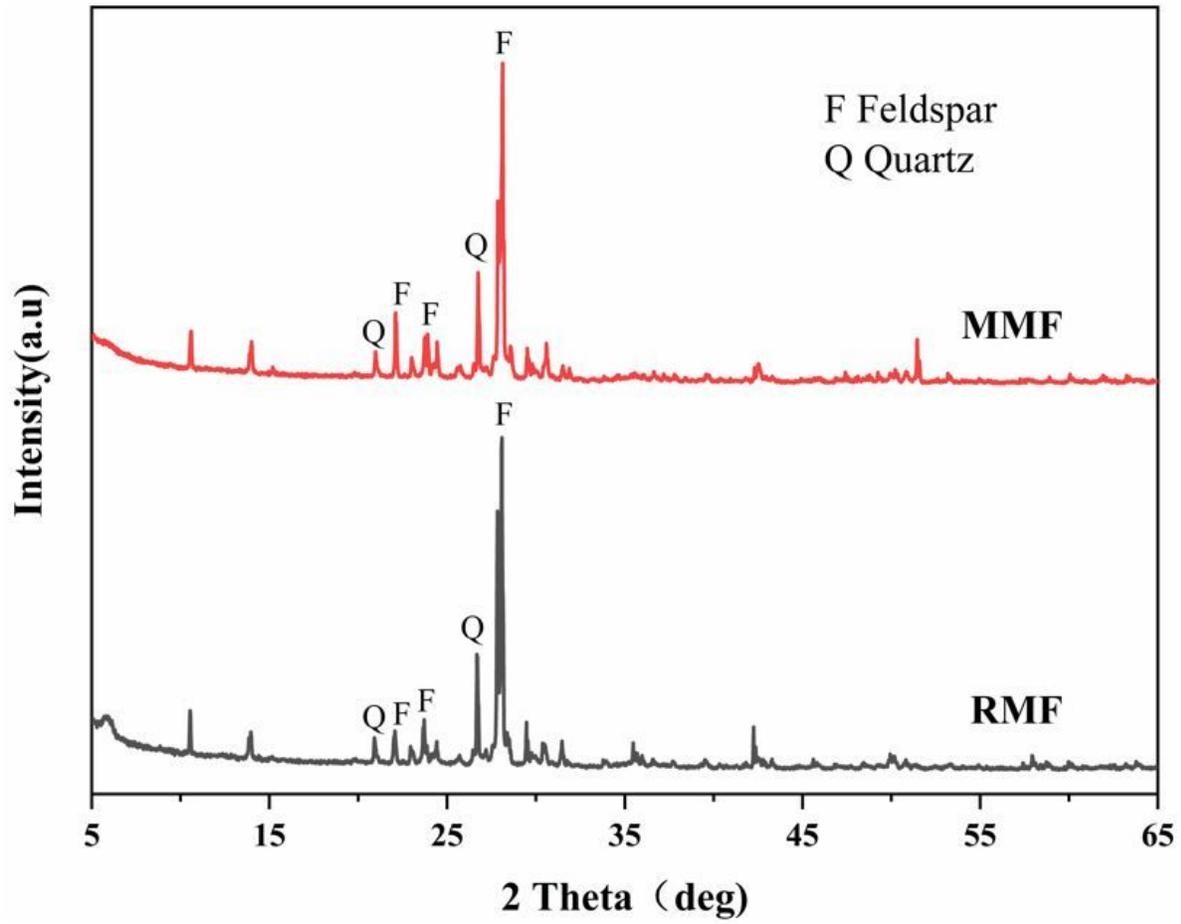


Figure 4

X-ray diffraction patterns of MMF and RMF, respectively.

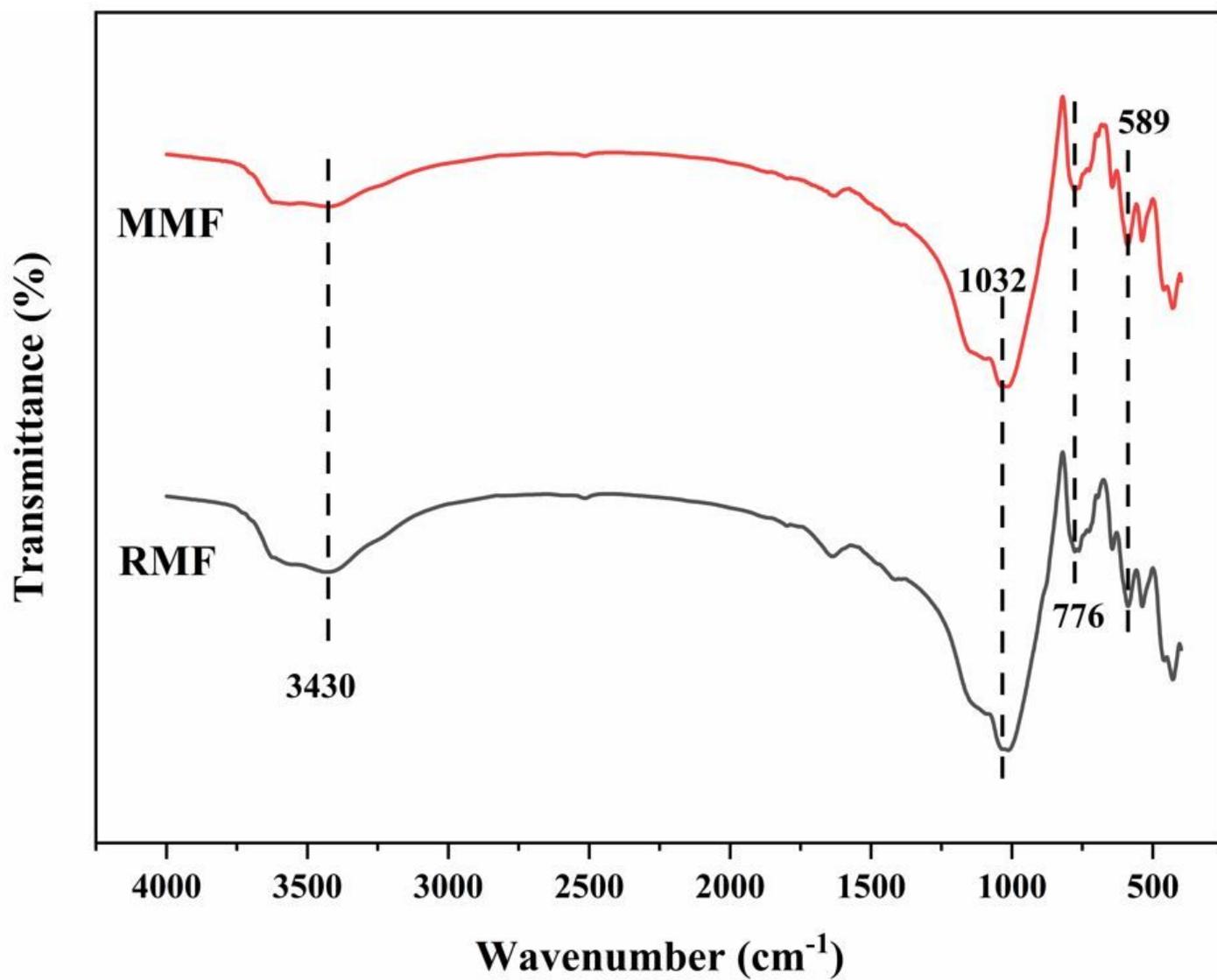


Figure 5

FTIR spectra of MMF and RMF, respectively.

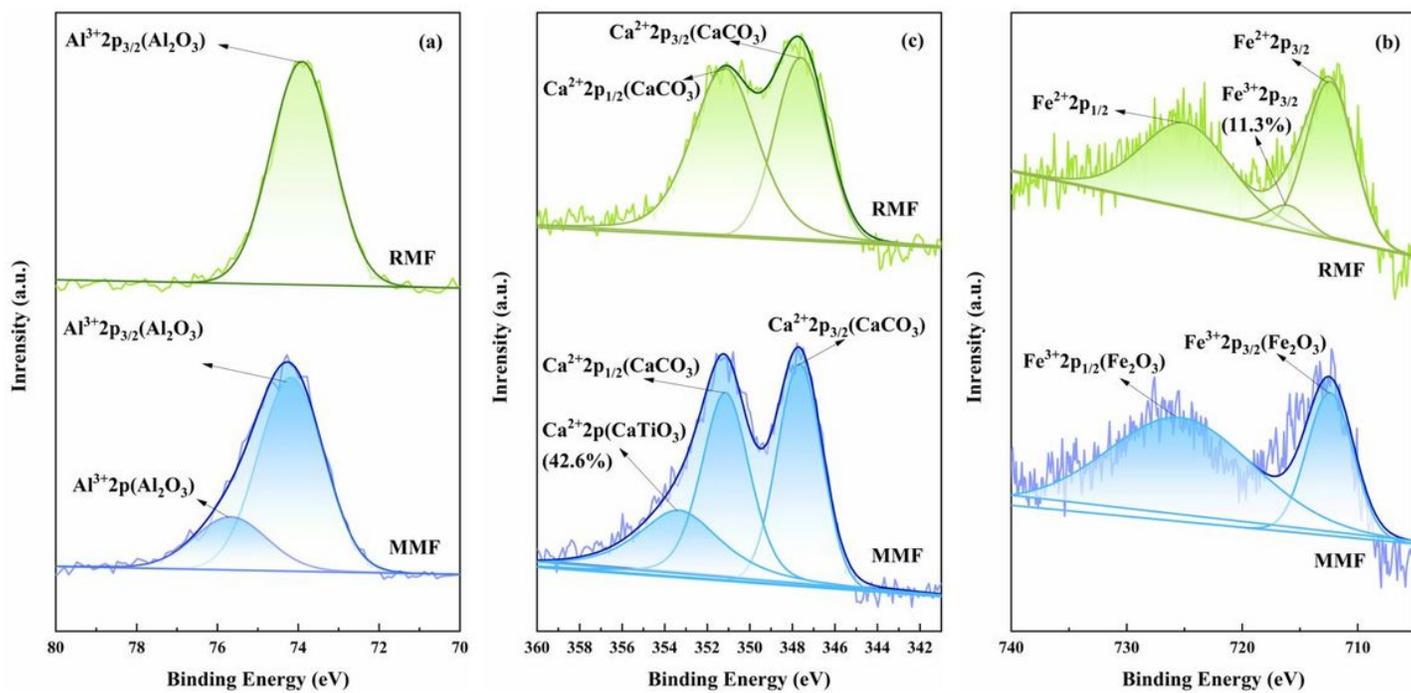


Figure 6

XPS spectra of MMF and RMF, respectively ((a) Al, (b) Fe, and (c) is Ca).

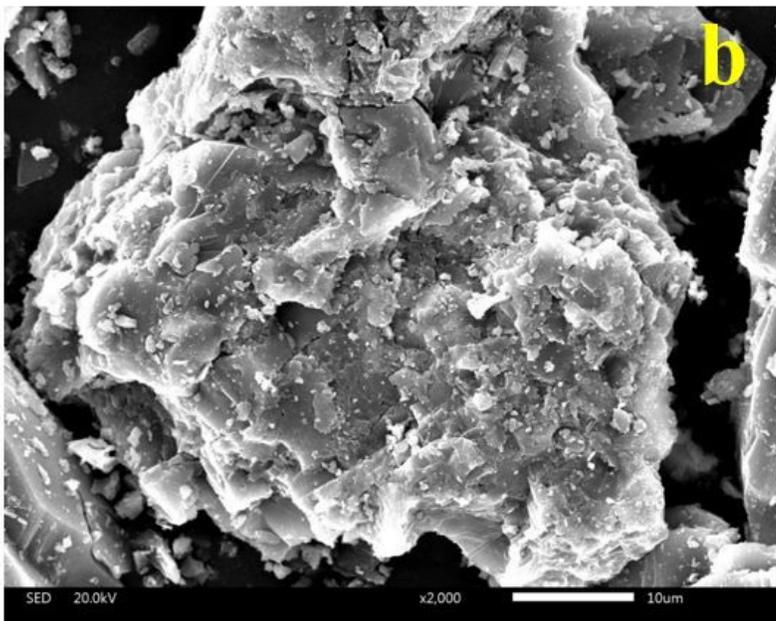
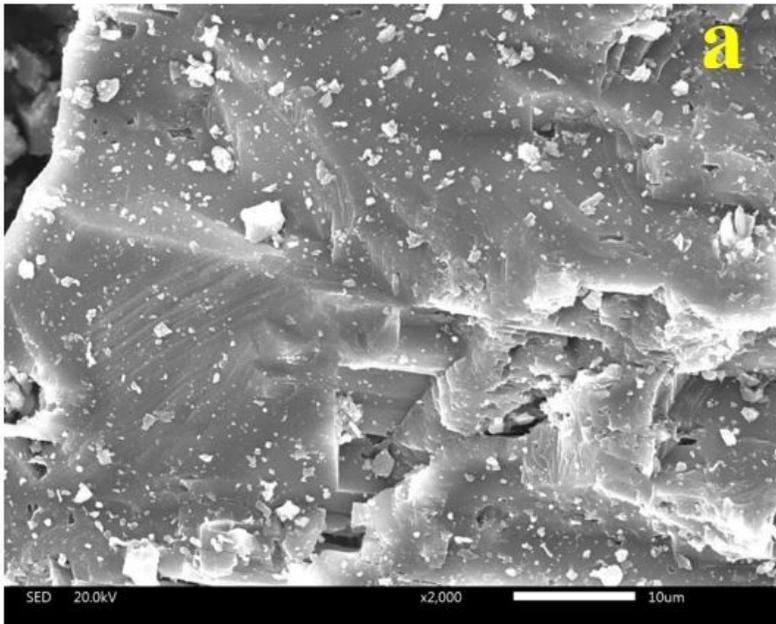


Figure 7

SEM of RMF (a) and MMF (b), respectively.

Supplementary Files

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