



## Phosphate removal of acid wastewater from high-phosphate hematite pickling process by in-situ self-formed dynamic membrane technology

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Received 12 November 2010; Accepted 26 June 2011

### ABSTRACT

Phosphate removal from pickling milling wastewater of high-phosphate hematite mineral by in-situ self-formed dynamic membrane reactor (DMR) developed with raw and activated red mud (RM-raw and RM-a) was studied in this paper. The results indicated that RM dosage, velocity gradient of the mixer ( $G$  value), reaction time ( $t$ ), and pH were main effects on phosphate removal. The pH of the actual mineral processing wastewater is 2.50–2.53 while the phosphate concentration is 98.85 mg/l. After the treatment, the removal rate of phosphorus with RM-raw and RM-a can reach 94.33% and 99.72%, respectively, with the dosage of 23.00 g/l and 20.00 g/l, respectively. Correspondingly, effluent pH was respectively 8.12 and 3.06, and both turbidity could reach the effluent requirement. Brush cleaning was a very effective cleaning method, and the membrane flux of DMR could be restored. Also, it demonstrated that acid reuse from the phosphate-contained acidic wastewater from wastewater produced in high-phosphate hematite pickling process could be achieved by in-situ self-formed dynamic membrane technology.

*Keywords:* Acidic regeneration; Activation; Dynamic membrane reactor; Mineral processing wastewater; Phosphate removal; Red mud

### 1. Introduction

Iron ore is important strategic mineral resource, with the acceleration of world economic integration in the 21st century, the demand for iron ore resources is growing, thus the environmental problems caused by mine wastewater will be more serious and the pollution will become a global issue which increasingly draws attention of the humankind, especially in China [1]. The acid pickling milling wastewater from hematite mineral processing studied in current work was produced in iron ore mineral processing technology with high phosphate concentration. In recent decades, a large quantity of acidic wastewater

containing phosphate discharged directly into environment has caused serious water pollution [2,3].

In order to respond to the demand for lowering the emission of the wastewater into the environment, many technologies for phosphate removing have been developed. The current technologies include physical chemical methods, photochemistry methods and biological methods [4–9]. Adsorption as the most widely used physical chemical methods is suited to treat inorganic phosphate acid wastewater [10,11]. In recent years, considerable attention have been paid to improve the performance of the adsorbents widely used are nature material, waste residue and active alumina as well as its modified material, porous material and synthetic adsorbent. Red mud (RM) is a waste by-product during the

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alkaline leaching of bauxite in the Bayer process, which causes serious environmental problems due to its high alkalinity and large amount [12]. RM has been used to remove fluoride, hexavalent chromium, dyes, chlorophenols, phosphate,  $\text{Cu}^{2+}$ ,  $\text{Zn}^{2+}$ ,  $\text{Ni}^{2+}$  and  $\text{Cd}^{2+}$  from aqueous solution owing to its high content of aluminum, iron, and calcium [13–18]. While the direct use of raw RM as adsorbent without activation for wastewater treatment is not appropriate because of the high dosage and alkalinity.

Dynamic membrane reactor is a kind of high and innovative sewage treatment technology. The dynamic membrane technology for wastewater treatment has gained great attention in recent years owing to its cost-effective membrane module, reduced energy consumption and high effluent quality [19,20]. The efficient entrapment function is achieved by making use of the contamination layer on the surface of the membrane. The dynamic membrane formed on the relatively large-pore mesh increases the intrinsic membrane retention capacity, leading to a high solid-liquid separation efficiency and a high filtration flux [21–28].

In this study, the in-situ self-formed dynamic membrane reactor used to treat the acid pickling milling wastewater of high-phosphate hematite mineral was developed with the activated RM, aiming to achieve the comprehensive reuse of the wastewater on-line with a low pH value and ultra-high phosphate removal.

## 2. Materials and methods

### 2.1. Materials

The acid wastewater tested in the experiment was taken from Enshi Chaoyang Iron Ore, Hubei province, China. The total phosphorus concentration (TP) (existence form  $\text{PO}_4^{3-}$ —P) was 98.85 mg/l, the pH value was 2.53, and the turbidity was 600~800 NTU. The membrane component of the dynamic membrane reactor was ceramic membrane (DR-50, Pingxiang, Jiangxi province, China).

The raw red mud (RM-raw) was obtained from Shandong Aluminium Industry Corporation, Shandong province, China. The particle size was averaged 20 mesh. The preparation of the activated RM (RM-a) was mainly described as follows: Firstly, the raw RM was washed and air dried, then pretreated at 800°C for 2 h and transferred into the glass reactor with HCl (the concentration was 0.5~1 mol/l) at 40 g~60 g (raw RM)/l(HCl) stirring for 1 h with water bathing. Then, the suspended liquids were fully precipitated, and the prepared products were washed, dried, and sieved through a 60 mesh sieve, sealed and preserved. Much preliminary work has been done to illustrate that the RM sieved through 60 mesh

sieve was appropriate for the experiment. The phosphate was not fully adsorbed when the particle size is bigger, while the precipitation after the treatment was not sufficient when the size is a little smaller.

### 2.2. Testing

#### 2.2.1. Batch testing

Acid pickling milling wastewater from high-phosphate hematite mineral processing (600 ml) was added into two stirrers respectively, then dephosphorization agents (RM-raw and RM-a) were put into the stirrers, respectively, and stirred with the velocity gradient 63.6. The phosphate concentration and pH value of supernate was measured after settling 4 h.

#### 2.2.2. Dynamic membrane reactor

The test equipment of the dynamic membrane reactor was in-situ membrane dynamic separation system formed of DR-50 ceramic membrane, miraco bubble aerate system and mixed liquor. Intermittent inflow was employed when the dynamic membrane reactor was running, a given amount of dephosphorization agent was added into the coagulation reactor containing actual mineral wastewater, effluent flowed through the dynamic membrane reactor under the condition of aeration after a certain time of coagulation. The flow chart of the technology in the experiment was shown Fig. 1.

### 2.3. Analysis methods

The phosphate was measured using the ammonium molybdate spectrophotometric method with a ultraviolet spectrophotometer (DR 4000/U, HACH company, USA). A pH meter (PHS-3C, Shanghai LeiCi instrument plant, China) was used to measure the pH of the solutions. The specific surface area of RM was determined by the BET nitrogen gas sorption method using a full automaticity specific surface area analyser (Gemini 2360, Micromeritics, USA). The compositions and the phases of raw and activated RM were measured with the full spectrum of direct reading inductively coupled plasma emission spectrometer (Optima 4300 DV, Perkin Elmer Ltd., USA) and X-ray diffractometer (D/MAX-RB, RIGAKU Corporation, Japan). The SEM micrograph of RM was determined using a analytical transmission electron (TEM) (H-600 STEM/EDX PV9100, HITACHI, Japan).

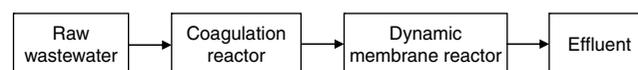


Fig. 1. Flow chart of technology in the experiment.

All the chemicals and reagents used in this study were of analytical grade. All glassware and sample bottles were soaked in diluted HCl solution for 12 h, washed and then rinsed three times with deionized water. Deionized water was used for the preparation of solutions. All experiments were conducted in duplicate and the average values were used for data analysis. Each water quality was tested according to Water and Wastewater Monitoring and Analysis Methods (4th edition, Chinese).

### 3. Results and discussion

#### 3.1. The characteristics of the dephosphorization agents

The components of RM-raw and RM-a are listed in Table 1. RM-raw consists of a heterogeneous mixture of several minerals, and some metal oxide or metal hydroxide plays an important role in the process of phosphate removing, while some inorganic minerals and metal ions (such as  $\text{Na}^+$ ,  $\text{K}^+$ , etc.) and other impurities in the components have a negative effect on phosphate removal [29]. In addition, the electrostatic repulsion with phosphate radical ion in solution would be brought about because of the existence of superficial hydroxyl which often possess electronegativity, consequently results in lower RM adsorption capacity.

The composition and property of raw and activated RM were given in Table 1. The composition of the activated RM (RM-a) was different from the raw sample. The main compositions were turned to ferric hydroxide and aluminum hydroxide, and some soluble salts were removed (Fig. 2). Besides, the alkalinity of RM reduced greatly after activation treatment. The pH of raw RM was 10.19 (solid/liquid ratio 1:20, dissolved in distilled water), and the pH of RM-a was decremented to 4.38; The BET surface area of the RM-a indicated that the activation treatment increased the surface area of the RM, the moisture in the RM-raw was reduced in the process of roasting, the membrane clung to the surface of the RM-raw, which impeded phosphate removing, was washed away in acid activated process, and interior pores were dredged at the same time. Scanning electron microscope (SEM) pictures were listed in Fig. 3. The RM-raw was relatively smooth and flat, and the RM-a showed many new cavities and coarsened exterior, probably due to the removing of some acid-soluble salts and water inside the RM.

Table 1  
Composition and property of RM-raw and RM-a (wt.%)

	$\text{Na}_2\text{O}$	$\text{Al}_2\text{O}_3$	$\text{SiO}_2$	$\text{CaO}$	$\text{TiO}_2$	$\text{Fe}_2\text{O}_3$	$\text{MgO}$	$\text{ZnO}$	pH	SBET ( $\text{m}^2/\text{g}$ )
RM-raw	15.06	25.96	23.23	2.39	1.73	28.69	0.16	0.63	10.19	14.76
RM-a	12.15	27.63	23.69	2.01	2.53	30.18	0.15	0.42	4.38	23.39

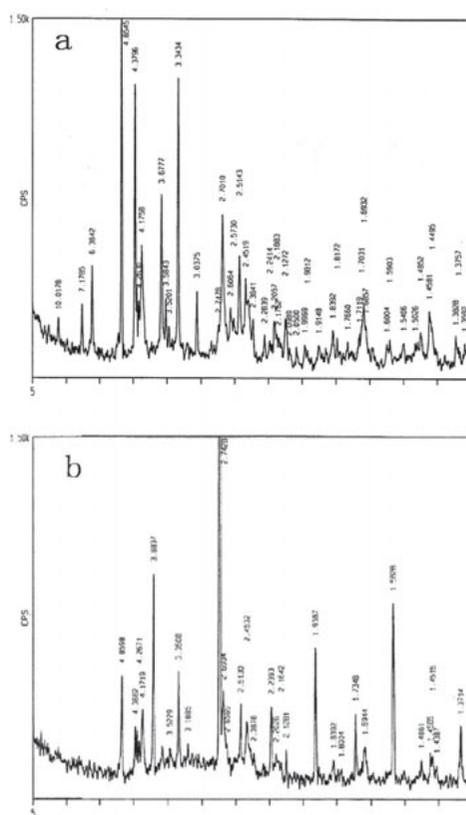


Fig. 2. XRD patterns of RM-raw and RM-a (a: RM-raw, b: RM-a).

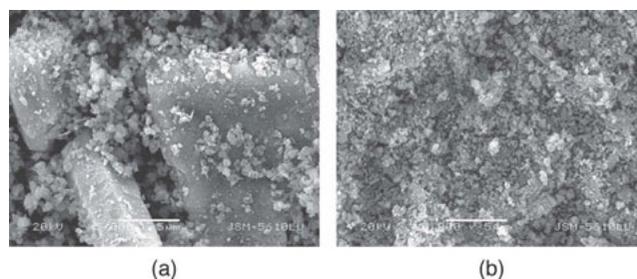


Fig. 3. SEM photographs of RM-raw and RM-a (a: RM-raw, b: RM-a).

#### 3.2. The effect of operating parameters

Much preliminary work has been done to optimize the experimental conditions. The result of the batch research indicated that the main effects on the treatment of the acid wastewater were the dosage of dephosphorization agent, velocity gradient ( $G$  value), reaction

time, pH and so on. During the preliminary treatment of the actual mineral processing wastewater with pH 2.50~2.53, the turbidity 600~800NTU, and phosphate concentration 98.85 mg/l, the optimum reaction time of RM-raw and RM-a was 60 min and 45 min, respectively, and the optimum velocity gradient ( $G$  value) of both the RM-raw and RM-a was 63.6.

### 3.2.1. Effect of the dosage of RM on the phosphate removing

The effect of the dosage of RM on the removing of phosphate was shown in Fig. 4. From Fig. 4, the optimum dosage of RM-raw was 23 g/l, the corresponding phosphate removal was 92.47%. When the dosage of RM-raw was less than 23 g/l, the phosphate uptaking increased with the increasing of the dosage of the RM-raw. While abnormal phenomenon appeared when dosage was less than 3.5 g/l, that was because phosphate was adopted by RM to form flocculent precipitate which could not be adequately deposited, and the liquid-solid was not fully separated, resulting in a lower phosphate removal. The phosphate removal gradually declined with the increase of the dosage when it was higher than 23 g/l, which may be due to the impact of the increased alkalinity. For the RM-a, colloidal phenomena did not appear and the liquid-solid was separated quickly. The phosphate removal reached 99.65 when the dosage of RM-a was 20 g/l.

### 3.2.2. Effect of reaction time on the phosphate removal

Fig. 5 showed the phosphate removal with reaction time 15 min, 30 min, 45 min, 60 min, 75 min and 90 min. As seen from Fig. 5, the RM-raw and RM-a had the similar phosphate removing trend, while the phosphate removal of RM-a was higher than that of the RM-raw. The phosphate removal achieved the peak value at 60 min for RM-raw, and at 45 min for the RM-a. RM-a could reach higher phosphate removal in a shorter reaction time.

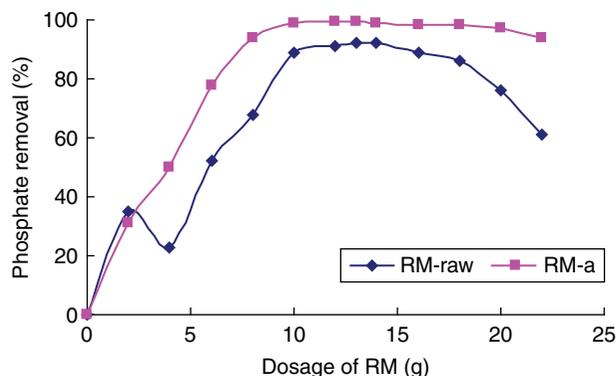


Fig. 4. Effect of dosage of the RMs on the phosphate removal.

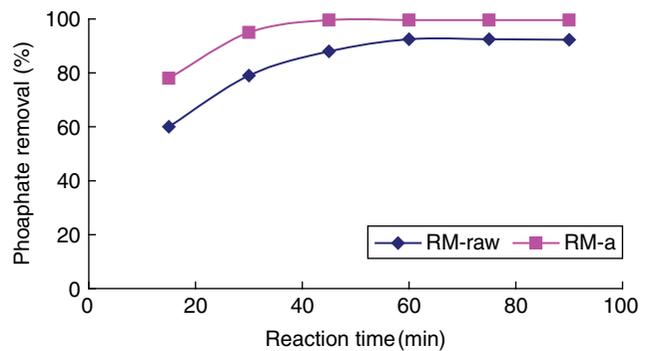


Fig. 5. Effect of the reaction time on the phosphate removal.

### 3.2.3. Effect of the dosage of RM on the effluent pH

The effect of the dosage of RM on the effluent pH was shown in Fig. 6. Both the effluent pH were increased by varying degrees with the increase of the dosage of RM, while the influence of RM-a was much less. As seen from Fig. 6, when the dosage of RM (RM-raw and RM-a) were 23 g/l, the effluent pH was 8.02 and 3.09 respectively. It is thus clear that the effluent pH was much lower after being treated by RM-a, which was more conducive to deal with the acidic wastewater, and it is useful for the reuse of the acidic wastewater to maintain the low pH value.

## 3.3. Performance of dynamic ceramic membrane

### 3.3.1. Analysis of dynamic membrane reactor

The mineral processing wastewater was treated by RM-a—in-situ dynamic membrane reactor (DMR) combined technology, and the dynamic membrane reactor was in-situ self-formed DMR developed with RM-a. The mixed liquor in the stirrer after stirring flowed through the ceramic membrane components under the continuous aeration with a velocity of 0.5 m<sup>3</sup>/h, and the settled

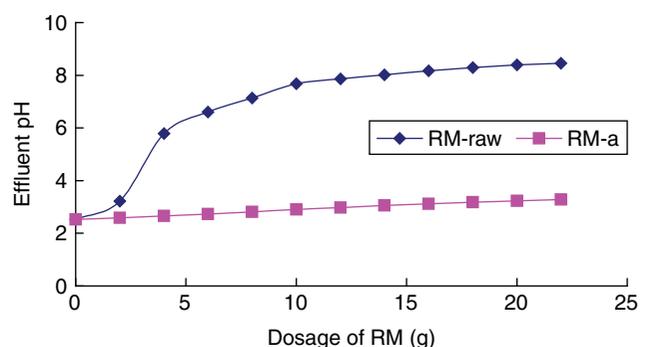


Fig. 6. Effect of the dosage of the RMs on the effluent pH.

hydraulic head used as the pressure of filtration membrane in the DMR was 40 cm. The test was under the condition of the constant pressure of filtration membrane and intermittent operation.

A certain dosage of RM-raw and RM-a were added into the coagulation reactor according to the optimized conditions of the batch test respectively, and the effluent flowed through the DMR under the condition of aeration after a certain time of stir. The effluent turbidity of DMR was shown in Table 2, the variation of effluent pH and the effect of phosphate removal by DMR was shown in Fig. 7 and Fig. 8, respectively. The turbidity removal efficiency of the DMR was satisfactory, both the effluent turbidity was reduced below 5 NTU after 30 min when the influent turbidity was 380 NTU, afterwards, the effluent turbidity tended to be stable. Both the effluent pH was increased by small margins with the running time increased, while the ascensional range of the RM-a—dynamic membrane reactor was less.

As seen from Fig. 8, the phosphate removal of the RM-raw and RM-a—DMR was only 79.8% and 86.1% respectively for the first 15 min, it could be explained that the dynamic membrane have not been completely formed, the liquid-solid was not fully separated, the phosphate removal could reach 94.33% and 99.72% respectively in 120 min, and the removal percentage was constant after that time. The removing efficiency of the phosphate was better through the treatment of DMR, that was because the polluted layer (filter cake

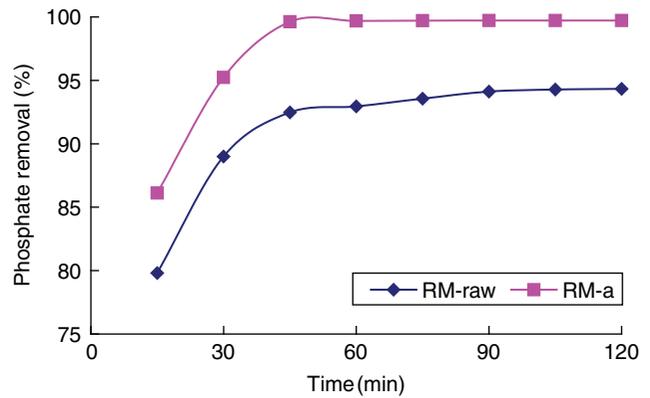


Fig. 8. Effect of phosphate removal by DMR.

and gel layer) on the surface of the carrier could react with the phosphate in the wastewater, and the efficient removing of phosphate could be further achieved by the action of adsorption and interception. It is thus clear that the RM-a—DMR had efficient phosphate removal rate, strong solid-liquid separating performance and lower effluent acidity. The on-line circulation and entirely reuse of the acidic wastewater could be achieved by the combination technique of RM-a and the DMR.

3.3.2. Analysis of membrane fouling

The membrane flux was counted according to the reaction time of DMR for analysing the membrane fouling condition, and the membrane fouling was investigated by studying the change of the membrane flux. The variation curve of the DMR was shown in Fig. 9. The membrane flux was attenuated more rapidly from 625 l/m<sup>2</sup>h to 510 l/m<sup>2</sup>h in an early stage before a steady value was reached, and since then the membrane flux was maintained at about 500 l/m<sup>2</sup>h. At this moment, the gel layer had essentially formed, and the self-formed dynamic membrane emerged primarily.

Table 2  
Variation of effluent turbidity of DMR

	T/min	0	15	30	60	90	120	180
RM-raw	Turbidity (NTU)	380	28.6	4.56	1.76	1.76	1.59	1.30
RM-a	Turbidity (NTU)	380	16.9	4.33	1.72	1.69	1.58	1.29

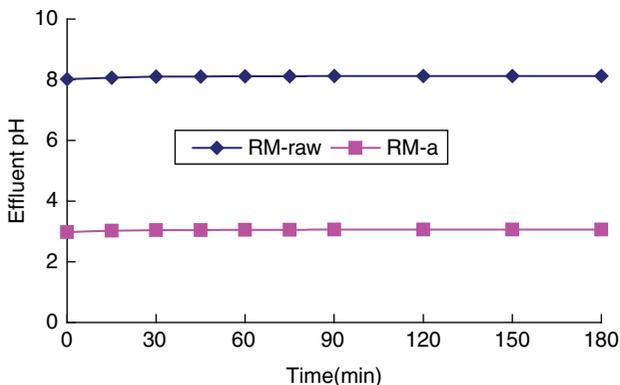


Fig. 7. Variation of effluent pH of DMR.

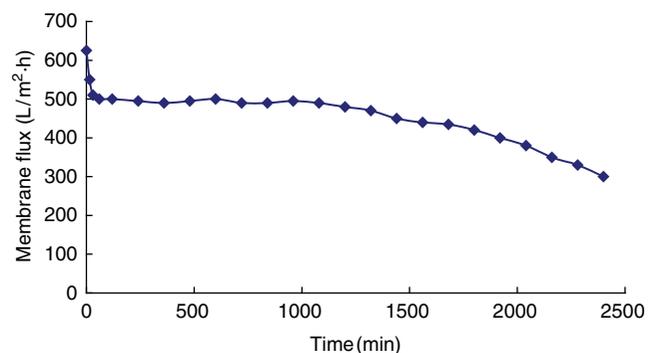


Fig. 9. Variation curve of the membrane flux.

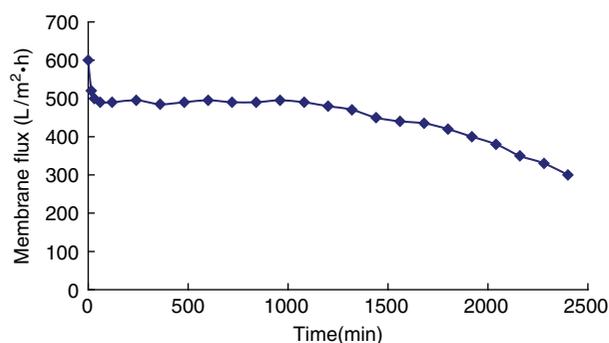


Fig. 10. Variation curve of the membrane flux after brush cleaning.

The membrane flux depression scope was steady for a long time, and the flux was not reduced until 30 h later. This could be the result of the aeration of membrane matrix, great shearing force and flow velocity through membrane was formed, and the settled sludge on the surface of membrane was deposited to the bottom of the reactor, so the filter cake was maintained at a stable level of relatively thin and effectively slowed down the rate of membrane fouling.

The membrane fouling of dynamic membrane was inevitable in the process of treatment, a certain degree of membrane fouling was beneficial for wastewater treatment, but when the membrane was heavily polluted, and the flux was decreased to influence the predefined processing requirements, the membrane was needed to clean timely to restore the water flux.

Brushing was employed as the membrane cleaning method in the experiment, considering that the inorganic ceramic membrane was featured with easy cleaning, structural stability, good stability, high mechanical robustness and long operating life. Fig. 10 showed the recovered membrane flux of the dynamic membrane by brush cleaning. The membrane flux was recovered to 600 l/m<sup>2</sup>h, and the dynamic membrane was formed again in shorter time. The result showed that the membrane flux of the dynamic membrane could be basically restored by brush cleaning, which was a very effective cleaning method in the experiment.

#### 4. Conclusions

Some conclusions of the study were listed as follows:

- (1) The characteristics of the red mud were changed after the activation method: the surface area increased, the adsorption capacity improved, and the alkalinity was reduced obviously. The optimum dosage of RM-raw and RM-a was 23 g/l and 20 g/l,

respectively, the corresponding phosphate removal was 92.47% and 99.66%, respectively, and the effluent pH value was 8.02 and 2.98, respectively.

- (2) The in-situ self-formed dynamic membrane reactor was featured by efficient interception function, better solid-liquid separating effect, short treatment cycle and easy cleaning, and the efficient removing of phosphate could be further achieved by the action of adsorption and interception. The phosphate removal of the RM-raw and RM-a—DMR could reach 94.33% and 99.72% respectively, the corresponding effluent pH was 8.12 and 3.06 respectively.
- (3) The membrane flux of the dynamic membrane could be basically restored by brush cleaning, a very effective cleaning method in the experiment. The on-line circulation and entirely reuse of the acidic wastewater could be achieved by the combination technique of RM-a and the DMR.

#### Acknowledgements

This work was supported by National Nature Science Foundation of China (No. 50808172, 30870221, 50909091), Distinguished Young Scientist Fund of Hubei Province, China (No. 2010CDA093) and Major Science and Technology Program for Water Pollution Control and Treatment of China 11th Five-Year Plan (No. 2009ZX07106-002-004).

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