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Simultaneous wastewater decoloration and fly ash dechlorination during the dye wastewater treatment by municipal solid waste incineration fly ash

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ABSTRACT

In this paper, the simultaneous decolorization of dye wastewater and dechlorination of municipal solid waste incineration (MSWI) fly ash were evaluated by using MSWI fly ash as adsorbent for synthetic dye wastewater (methylene blue, MB) and real dye wastewater treatment. After mixing with dye wastewater, the chlorine content in MSWI fly ash can be reduced effectively from 10.95% ± 0.53% to 0.44% ± 0.06% (below the standard (1%) of cement substitution material in China), and the residual heavy metals in fly ash obtained from toxicity characteristic leaching procedure (TCLP) can meet the US EPA leaching standards. Simultaneously, the color and total organic carbon (TOC) in wastewater can be significantly removed due to the adsorption of dye on MSWI fly ash. MB adsorption on MSWI fly ash followed Langmuir model and the monolayer adsorption capacity of MB was estimated to be 10.20 mg/g. The results indicate that using MSWI fly ash to treat dye wastewater is a promising method which can put into effect the dechlorination of MSWI fly ash and the decolorization of dye wastewater at the same time.

Keywords: MSWI fly ash; Dye wastewater; Methylene blue; Color removal; Wet dechlorination

1. Introduction

Dyes are widely used in various industrial processes to impart a desired color to their products (food, paper, rubber, textile and plastics). The effluents of these industrial processes are highly colored and difficult to dispose because of the wide varieties and complex structures of these dyes [1,2]. The commonly used methods for dyes removal, such as chemical coagulation, chemical oxidation, membrane filtration and biological degradation, suffer from one or another limitation, and none of them is successful in complete removal of the color from wastewater [3]. Adsorption is a widely used method for the removal of both organic and inorganic pollutants from aqueous effluents. However, commercially available adsorbents, such as activated carbon [3–6], zeolite [6,7] are quite expensive. Thus, there is a growing interest in searching for cheaper materials for dyes adsorption and some efforts have been made on exploring low-cost adsorbents based on peat [8], lignite [9], red mud, activated carbon prepared by naturally occurring materials [10–14] and coal fly ash [15–18].

Similar to coal fly ash, MSWI fly ash has high porosity and large surface area, which can serve as an ideal adsorbent [19]. As incineration is becoming a popular

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method for municipal solid waste disposal in large cities (e.g. Shenzhen, Shanghai, Beijing, Tianjin, Guangzhou, and Hangzhou) of China, large amount of MSWI fly ash generated from incineration process is required to be safely treated. Currently, solidification followed by landfill is a prevailing technology for MSWI fly ash disposal. However, the toxic components can leach from landfill solids and may pollute groundwater [20]. In addition, a shortage of landfill sites is a serious problem encountered in populated regions (as is the case in large cities of China). Therefore, recycling MSWI fly ash as construction resources could be a potential way of alleviating the problem. Typically, chlorine contents between 10 and 20 wt.% are found in MSWI fly ash. Whereas, ordinary construction material requires low chlorine content, for instance, the chlorine content of Portland cement needs to be less than 0.02 wt.% to avoid corrosion and heavy metal leaching [21]. So, various pretreatment methods were established to reduce the chlorine content of MSWI fly ash. Among them, washing by fresh water is a cheap and effective method to remove soluble chlorides and heavy metals contained in MSWI fly ash, but large amount of fresh water is consumed in this process [21–24]. From the view of resource conservation and cost saving, using industrial wastewater to wash MSWI fly ash may be an alternative. Considering the adsorption ability of fly ash, maybe the aqueous contaminations in industrial wastewater could also be removal in this process.

In order to verify the feasibility of the above-mentioned hypotheses, MSWI fly ash was introduced into synthetic methylene blue (MB) aqueous solution and real dye wastewater to investigate the synergic effects of dye wastewater decoloration and MSWI fly ash dechlorination. Heavy metals leaching behavior of MSWI fly ash after washing by MB solution was also examined.

2. Materials and methods

2.1. Materials

MSWI fly ash was collected from Yuqiao municipal solid waste incineration plant in Shanghai, China. The collected fly ash was dried at 105°C for 24 h and then screened with a 120 mesh sieve for experimental use. The specific surface area of fly ash is 9.8 m^2/g. The fly ash particles follow a near normal distribution with an average particle size (d_{50}) of 25.33 μm and d_{90} of 51.48 μm. The chemical compositions of fly ash were identified by XRF (X-ray fluorescence) and the results are (given in percent of oxides) shown in Table 1.

MB aqueous solution and real dye wastewater were used to test the adsorption capacity of MSWI fly ash in this experiment. MB solution was prepared by dissolving analytical grade of MB (Shanghai reagent factory, Shanghai, China) in distilled water without pH adjustment. The real dye wastewater was obtained from Shennan textile factory in Shanghai, China. This wastewater was in deep blue color, with the chroma of 170, pH of 8.58 and total organic carbon (TOC) concentration of 92.2 mg/l.

Table 1
Chemical compositions of the fly ash (wt %)

CaO	32.4
SiO_2	15.1
Cl	16.5
SO_3	11.5
K_2O	6.50
Al_2O_3	4.79
Fe_2O_3	4.50

2.2. Adsorption studies

Adsorption studies were performed in 250 ml Erlenmeyer flasks. To avoid the photo-degradation of dyes, the flasks were covered with aluminum foil. Each conical flask was filled with 100 ml of MB solution with varying concentrations or real dye wastewater. A certain amount of adsorbent was added to each conical flask, and then the flasks were agitated in a temperature controlled shaker (25°C) at 100 rpm for a desired time period. After agitation, the mixture was kept at room temperature for 10 min and subsequently was filtered. The filtered solution was taken for MB concentration (or chroma) and TOC concentration determination, and the collected adsorbent was dried at 105°C to a constant weight for chloride concentration measurement and other characterizations.

2.3. Charaterization

MB concentration was determined on a UV-visible spectrophotometer (ET99731, Tintometer GmbH, Germany) by measuring absorbance at the wavelength of 665 nm. TOC concentration was determined by TOC-VCPH analyzer (Mutil C/N 2100, Shimadzu, Japan). Chroma was determined by Chroma instrument (CN60M/SD9011, Westmid, China) and pH was measured by a pH meter (pHS-3C, Shanghai Ridao Scientific Instrument Co., Ltd, China).

Chloride iron concentration in MSWI fly ash was determined by a volumetric method using 0.1 N silver nitrate solution and K_2CrO_4 as indicator (JC/T420-1991, China). In order to evaluate the leaching behavior of heavy metals in MSWI fly ash, the leaching tests were carried out according to US EPA SW-846 TCLP test method 1311 [25]. Specifically, fifty grams of fly ash was added into a polypropylene plastic bottle containing 1000 ml of dilute acetic acid (pH = 2.85) solution. The suspension was shaken at 30 rpm for 18 h on an end-over-end shaker and then filtered [26]. The concentrations of heavy metals in filtered solution were detected by inductively coupled plasma atomic emission spectrometer (ICP-AES, Shimadzu, ICPS-7510).

A sequential extraction scheme developed by Klemm and Bhatty [27] was used in this experiment to analyze the distribution and specification of heavy metals in the fly ash samples before and after adsorption/washing. This method can divide heavy metals into four speciation fractions, namely bound to soluble and carbonates, iron and manganese, organic matter/sulfide and residual fraction. Then ICP-AES (Shimadzu, ICPS-7510) was used to quantify the levels of heavy metals in each fraction.

The X-ray diffraction (XRD) was used to identify the crystalline mineral phases present in MSWI fly ash. The XRD patterns of samples were collected using Cu Kα radiation at 40 kV and 50 mA on a RB XRD diffractometer at a scanning speed of 3°/min in the range of 2θ =10–60°.

A scanning electron microscope (SEM) equipped with energy dispersive X-ray spectroscopy (EPS) (JSM-6700F) was used to characterize the basic constituents and morphological information of the raw and washed MSWI fly ash.

Infrared spectra of the fly ash samples before and after MB adsorption were collected on a Perkin-Elmer 2000 FTIR after 400 scans within 4000-400 cm^{-1} at a resolution of 4 cm^{-1} by measuring the IR absorbance of KBr disc that contained 1–2 wt% of dry MSWI samples.

3. Results and discussion

3.1. MB adsorption by MSWI fly ash

MB adsorption was performed at a fixed fly ash loading of 10 g/L with different initial dye concentrations in 10–200 mg/L. The effect of contact time on MB removal is shown in Fig. 1. Results indicate that a contact time of 150 min assured attainment of the adsorption equilibrium in all cases. It can be observed that MB adsorption presented a two stage kinetic behavior, with a rapid initial adsorption during the first 10 min, followed by a much slower process. A similar behavior was reported in the literatures for dye adsorption over bottom ash [28], raw date pits [29] and coffee husks [30]. Oliveira et al. [30] attributed this behavior to a possible change in adsorption mechanism (in relation to that occurring at lower MB concentrations) because of the persistent presence of MB concentration gradient between in solution and on the surface. However, an exact explanation was not given. Senthilkumaar et al. [31] thought that two stages during the dye molecule adsorption corresponds to adsorption on the adsorbent surface and subsequently into the porous structure of the adsorbent through diffusion, with the later one taking more time. The faster adsorption of MB during the first 10 min at all initial MB concentrations indicate that MB adsorption occurs mainly on the surface of the MSWI fly ash. The equilibrium removal efficiency of MB in the concentration range of 10–200 mg/L was 82.1–88.2%, similar to that of MB adsorption by coal fly ash [32] and activated carbon [33], suggesting MSWI fly ash is an ideal absorbent for dye removal.

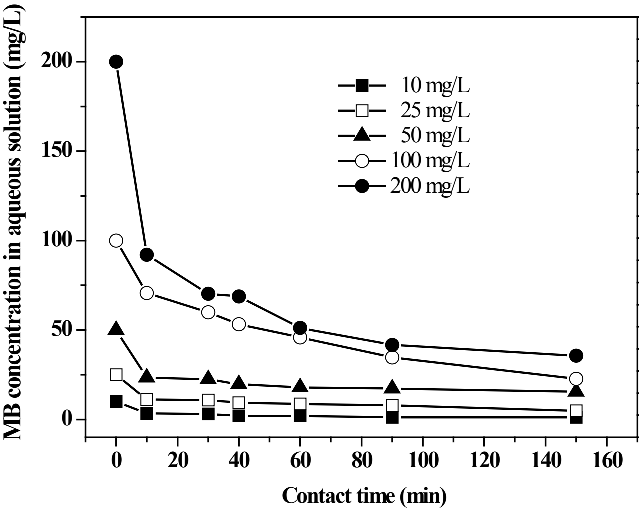


Fig. 1. Effect of contact time on MB adsorption by fly ash (25°C). Initial MB concentration: (■) 10 mg/l, (□) 25 mg/l, (▲) 50 mg/l, (○) 100 mg/l and (●) 200 mg/l.

The effect of adsorbent dosage on MB removal was studied at a fixed MB concentration (100 mg/l) with varied fly ash dosages ranging from 6 to 20 g/l. The effect of adsorbent dosage on MB removal is shown in Fig. 2. It is clear that MB removal efficiency increased with fly ash dosage. After the first 30 min of adsorption, the MB removal percentage exceeded 40% for smaller adsorbent amount (6–10 g/l) and more than 55% for high adsorbent concentrations (12–20 g/l). It also can be seen that contact time had different effects on the MB removal

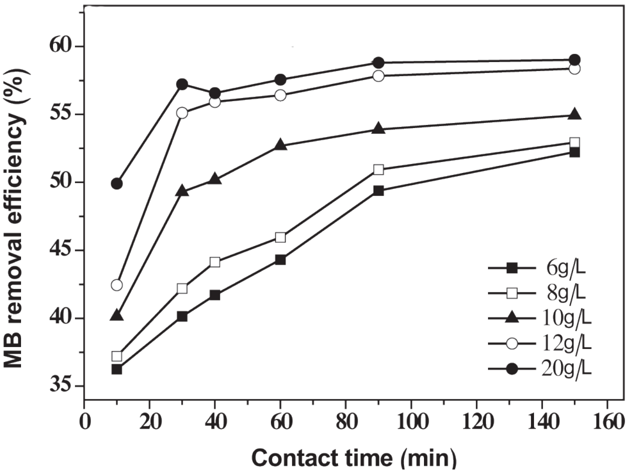


Fig. 2. Effect of adsorbent concentration on MB removal (25°C) fly ash concentration: (■) 6 g/l, (□) 8 g/l, (▲) 10 g/l, (○) 12 g/l and (●) 20 g/l.

when different adsorbent amounts were used. At low adsorbent dosages (6–8 g/l), the removal of MB increased almost linearly with contact time. However, a rapid MB removal during the first 30 min and then followed by a slight increase of removal efficiencies can be observed at elevated adsorbent dosages (12–20 g/l). The phenomena can be easily explained with the increase in surface area, i.e. more active adsorption sites when more adsorbent is used. After 150 min of adsorption, the MB removal efficiencies at 1.2–2.0 g/l of R–F were very close, ranging from 56.9 to 59.0 %.

In order to quantify the MB adsorption capacity onto fly ash, the Langmuir isotherm model was applied to fit the experimental data. As can be seen in Fig. 3, the data validated the linear form of Langmuir model [Eq. (1)] expressed below:

$$C_e/q_e = 1/KQ_0 + \left(1/Q_0\right)C_e \tag{1}$$

where C_e is the equilibrium concentration (mg/l) and q_e is the amount adsorbed per specified amount of adsorbent (mg/g), K is the Langmuir equilibrium constant and Q_0 is the amount of adsorbate required to form a monolayer.

According to the slop and intercept of the line shown in Fig. 3, the Langmuir monolayer adsorption capacity of MB was 10.20 mg/g. Table 2 lists some reported maximum adsorption capacities of MB on various adsorbents derived from agricultural and industrial solid wastes. It can be noted that the adsorption capacity of MB on MSWI fly ash is lower than some agricultural solid wastes derived adsorbents such as coffee husks [30], meranti sawdust [34], cedar sawdust [35] and date pits [36], and lies within the range of industrial solid wastes derived adsorbents [37–39]. Considering that MSWI fly ash is a kind of solid waste/byproduct of municipal solid waste incineration plants and available free of cost, the low adsorption capacity is acceptable.

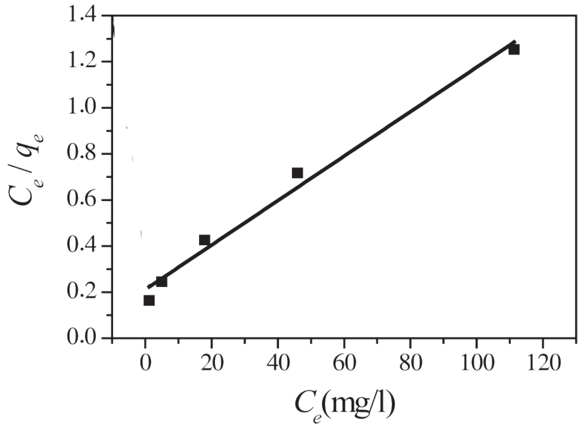


Fig. 3. Langmuir isotherm plot of MB adsorption onto fly ash at 25°C.

Table 2
Comparison of adsorption capacities of various adsorbents for MB

Adsorbent	Maximum monolayer adsorption capacity (mg/g)	Reference
Coffee husks	90.09	[30]
Meranti sawdust	120.48	[34]
Cedar sawdust	142.36	[35]
Date pits	80.31	[36]
Coal fly ash	12.70	[37]
Fly ash (baggasse)	6.46	[38]
Fly ash	2.85	[39]
MSWI fly ash	10.20	This experiment

3.2. Characteristics of MSWI fly ash

3.2.1. MSWI fly ash dechlorination

In order to investigate dechlorination effect of MSWI fly ash, 20 g/l of fly ash was added into MB solution with the dye concentration of 100 mg/l. After 20 min of agitation and 10 min of standing, the adsorbent was separated and dried at 105°C for chlorine content determination. Fig. 4 shows the chlorine content in fly ash and MB solution washed MSWI fly ash (MW-FA). For comparison, the chlorine content in MSWI fly ash washed with tap water (TW-FA) obtained in previous study was also provided. Note that the chlorine content in MSWI fly ash decreased significantly from 10.95%±0.53% to 0.44%±0.06% after washing with MB solution. Compared with tap water, the MB solution showed similar effect on MSWI fly ash dechlorination. It should be pointed out that the chlorine content in MW-FA is lower than the control objective (1%) for cement substitution material (GB 175-2007, China).

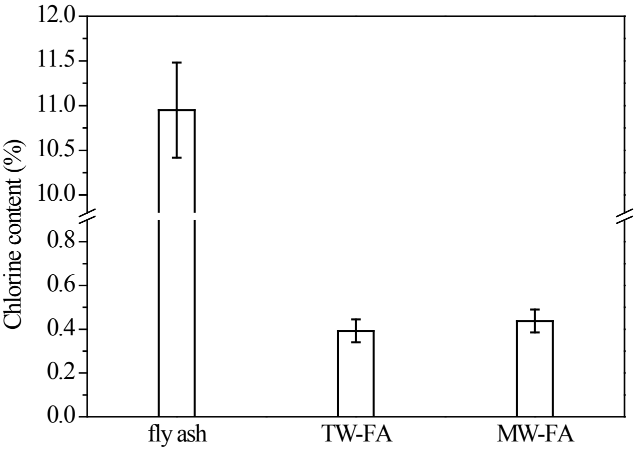


Fig. 4. Chlorine content in fly ash, TW-FA and MW-FA.

3.2.2. TCLP and speciation fractions characteristics of heavy metals

TCLP analysis was carried out to identify the heavy metals in fly ash, MW-FA and TW-FA. As listed in Table 3, after washing with tap water and MB solution, the leaching concentrations of heavy metals in MSWI fly ash dropped remarkably and can meet US EPA standard (US EPA 40 CFR part 261.24 2005) and China's hazardous waste identification standard (GB5085.3-2007, China).

Different speciation fractions of heavy metals in fly ash and MW-FA are shown in Fig. 5. The easily soluble fractions of heavy metals in fly ash were present in high proportions, e.g. Zn, Pb, Cd and Cr bound to soluble and carbonate speciation were 65%, 63%, 86% and 25%, respectively. These soluble fractions can easily leach out and thus limit the recycle and re-application of MSWI fly ash. After washing with MB solution, Pb, Zn and Cd in MSWI fly ash bound to soluble and carbonates fraction reduced close to zero, and the fraction of Cr bound to soluble and carbonated decreased to 19.0%. These indicate that the heavy metals in MW-FA are more stable than fly ash.

3.2.3. Phase and chemical composition

The XRD patterns of fly ash and MW-FA are illustrated in Fig. 6. The two samples exhibited different crystalline mineral phases. For fly ash, SiO_2 and CaO were the major mineral phase, but some minor phases such as $CaSO_4$, $CaCO_3$, $CaCl_2$, NaCl, as well as KCl also existed. For MW-FA, however, some soluble minor phases, such as $CaCl_2$, NaCl and KCl were gone and not detected, but $CaCO_3$ as the new phase was detected.

Some chemical species are also revealed with the characteristic IR vibrations (Fig. 7). From FTIR spectrum of fly ash, carbonate is featured with one peak at 1414 cm^{-1} (ν_3 mode) and another one at 875 cm^{-1} (ν_2 mode). After washing by MB solution, the carbonate in MW-FA is characterized with one degenerated ν_3 mode (1459 and 1418 cm^{-1}) together with one ν_2 mode at 875 cm^{-1}. Similarly, sulfate is featured with 1155 (degenerated ν_3 mode) and 673 cm^{-1} (ν_4 mode) in fly ash and characterized with 1155 (degenerated ν_3 mode) and 677 cm^{-1} (ν_4 mode) in MW-FA. Apparently, the characteristic peaks of carbonate and sulfate were shifted or split after the fly ash being washed by MB solution. In addition, the band at 3400–3500 cm^{-1} is due to the O–H vibration and the absorption bands around 1630 cm^{-1} represented C=C stretching. These peaks were intensified after the fly ash being washed with MB indicated that those functional

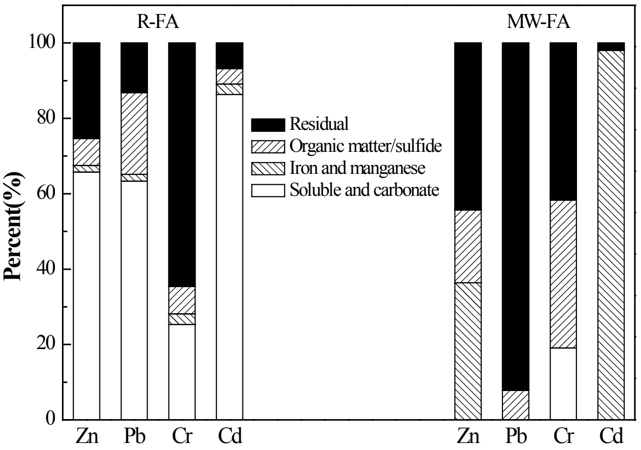


Fig. 5. Speciation fractions of heavy metals in fly ash and MW-FA.

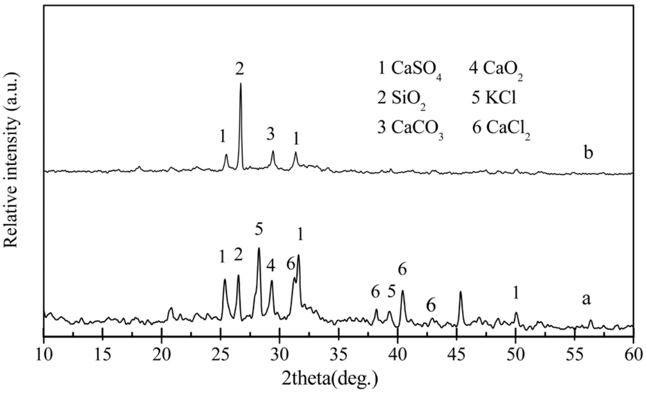


Fig. 6. XRD patterns of (a) fly ash and (b) MW-FA.

Table 3
TCLP characteristics of fly ash, TW-FA and MW-FA

Heavy metal	Leaching concentration (mg/l)			Standard (mg/l)
	Fly ash	TW-FA	MW-FA	
Pb	12.1–16.9	0.2–0.5	1.21–1.31	5.0[a]
Zn	40.1–45.8	0.31–0.49	0.11–0.29	50[b]
Cd	2.1–3.9	0.01–0.03	0.06–0.08	1.0[a]
Cr	0.21–0.79	0.11–0.21	0.31–0.39	5.0[a]

[a] US EPA standard: US EPA. 40CFR part 261.24 2005
[b] China's hazardous waste identification standards (GB5085.3-2007)

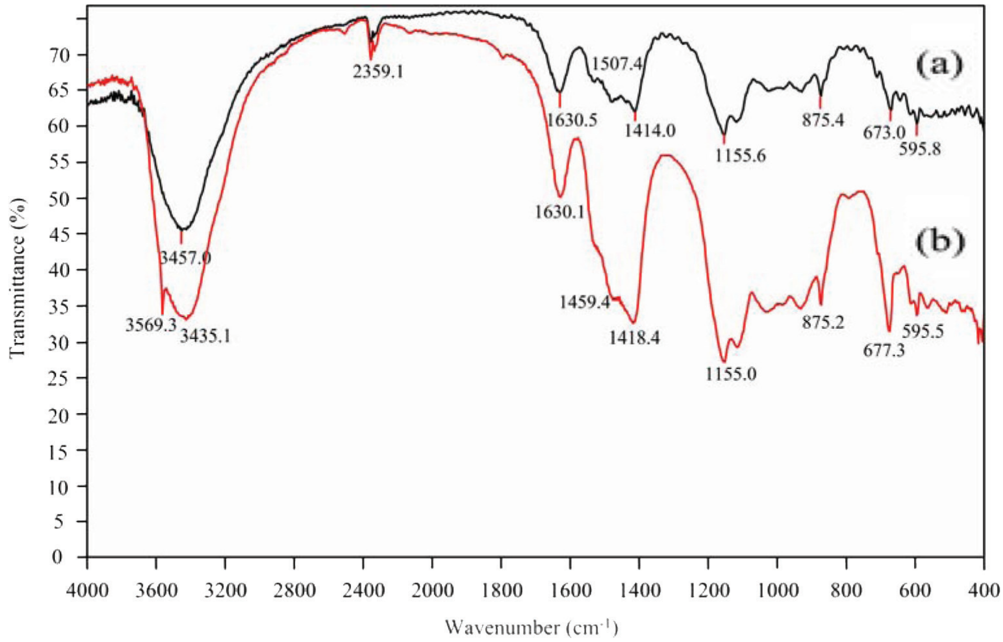


Fig. 7. FTIR spectra of (a) fly ash and (b) MW-FA.

groups related to MB were transferred onto the surface of MSWI fly ash.

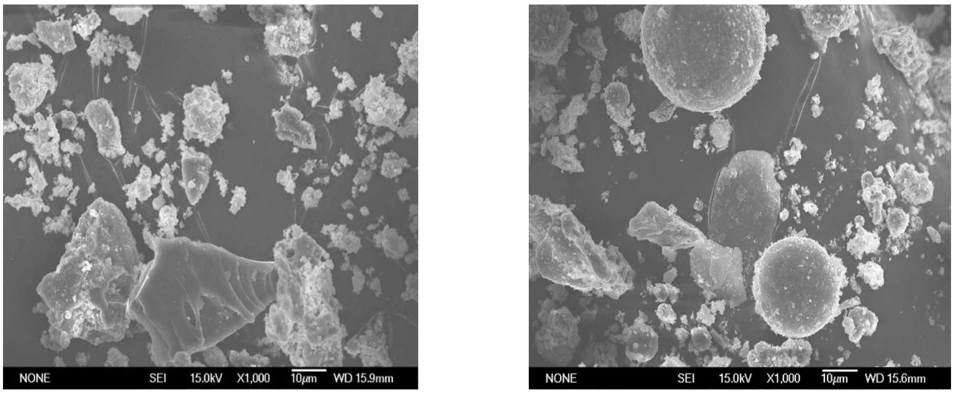
SEM images (Fig. 8) indicate that fly ash particles were essentially present in irregular shape. After washing with MB solution, the surface of fly ash particles became smoother, indicating that some crystalline materials originally attached onto the surface of fly ash particles were effectively removed.

3.3. Treatment of real dye wastewater by MSWI fly ash

The decoloration of real dye wastewater with MSWI fly ash was tested at different fly ash dosages (2.5–20.0 g/l) with fixed contact time (20 min).

As shown in Fig. 9, along with the increase of adsorbent dosage, the chroma of wastewater first decreased rapidly and then increased slightly, however the pH of wastewater increased monotonously. It should be noted that 5.0 g/l of fly ash can make the chroma of wastewater dropped to 50, which is the discharge limit of dye wastewater in China (GB 4287–1992, China). When adsorbent amount was doubled to 10.0 g/l, the chroma of wastewater further decreased to 40. However, when fly ash dosage was added up to 20 g/l, the chroma of wastewater rose inversely to 60, which may be related to the pH change [34].

The effect of fly ash dosage on TOC removal was similar to that on chroma removal. For the lowest adsorbent amount (2.5 g/l) used in this experiment, the TOC concentration in wastewater decreased significantly from 92.2 mg/l to 18.5 mg/l, which is lower than the discharge standard (20 mg/l) of dye wastewater in China (GB 4287–1992, China). Further increasing fly ash dosage



(a) fly ash (b) MW-FA

Fig. 8. SEM photographs of (a) fly ash and (b) MW-FA.

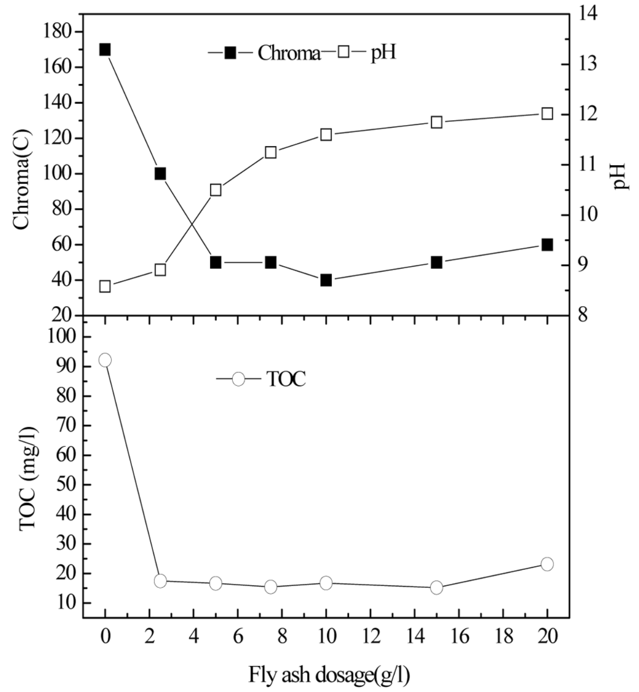


Fig. 9. Chroma and TOC removal of real dye wastewater along with fly ash dosage.

from 2.5 to 15.0 g/l almost had no effect on TOC removal. However, when 20.0 g/l of fly ash was introduced, the TOC concentration in wastewater presented a slightly upward trend. It was inferred that some unburned carbon particles in MSWI fly ash dissolved into the wastewater. However, the exact reason is not clear to us at the present, thus further study is required.

4. Conclusions

Simultaneous wastewater decolorization and MSWI fly ash dechlorination were evaluated in the MSWI fly ash wet-pretreatment process by using dye wastewater as washing agent. During the pretreatment process, the chlorine content in MSWI fly ash was reduced significantly from 10.95 ± 0.53% to 0.44 ± 0.06% and met the control objective (1%) of raw materials substitution for cement in China. Additionally, the soluble species of heavy metals in MSWI fly ash can also be removed effectively and the residual TCLP values could meet the US EPA leaching standards. MSWI fly ash shows good ability to remove color from dye wastewater and the MB adsorption on fly ash followed Langmuir model with the monolayer adsorption capacity of 10.20 mg/g. These results indicate that using MSWI fly ash to treat dye wastewater is a promising method which can realize MSWI fly ash dechlorination and dye wastewater decolorization simultaneously.

Symbols

C_e — MB equilibrium concentration, mg/l
K — Langmuir equilibrium constant
Q_0 — Amount of adsorbate required to form a monolayer
q_e — Adsorbed amount of MB per specified amount of adsorbent, mg/g

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