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TREATMENT OF MUNICIPAL LANDFILL LEACHATE WITH ORGANICALLY MODIFIED BENTONITE

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Abstract—Landfill leachate is one of the most difficult effluents with which to deal from an environmental perspective because of its concentration and complex composition, including refractory and toxic components such as heavy metals or xenobiotic organic compounds. The objective of the present study was to use organically modified bentonite (OMB) to dispose of landfill leachate >10 y old. The OMB was synthesized using a new method, which removed four steps (filtering, washing, drying, and grinding) from the traditional process. After treatment using OMB, the chemical oxygen demand concentration (COD concentration, an index of the organic pollutants in the landfill leachate, was determined using the potassium dichromate method) of the landfill leachate sample decreased from 2400 to 245 mg/L in 5 h, *i.e.* the organic pollutants reduction efficiency was as high as 90%. Gas chromatography-mass spectrometry results indicated that most of the organic compounds were removed during the process. The modified and unmodified bentonite contained in the OMB deal with the hydrophobic and hydrophilic organic pollutants, respectively, resulting in significant degradation of the leachate. The study results have provided a new cost-effective method for treatment of landfill leachate.

Key Words—Hydrophilicity, Hydrophobicity, Landfill Leachate, Organic Pollutants, Organically Modified Bentonite (OMB).

INTRODUCTION

Sanitary landfilling is the main method used internationally to dispose of municipal solid wastes. In China, nearly 85% of municipal wastes are deposited in sanitary landfills. In spite of the many advantages, sanitary landfilling inevitably produces heavily polluted landfill leachate, during and after landfilling. The components of the landfill leachate are generally very complex, including organic compounds, ammonia nitrogen, and various heavy metals, and their concentrations are sometimes extremely high. The contamination by leachate of the surrounding air, soil, surface water, and groundwater (Zhou *et al.*, 2007; Bortolotto *et al.*, 2009) poses a great threat to residents near landfill sites. Organic pollutants in the leachate are the most difficult with which to deal (Fujita *et al.*, 1996; Chian, 1997; Kjeldsen *et al.*, 2002; Leenheer and Croue, 2003). In a landfill site >10 y old, the BOD_5 (biochemical oxygen demand after 5 days) of the leachate is small and the BOD_5/COD value is <0.3, indicating that man-made biological methods are ineffective means for treating the leachate (Kjeldsen *et al.*, 2002; Ni *et al.*, 2004; Colomer Mendoza and Gallardo Izquierdo, 2009). The lack of a harmless and cost-effective treatment for landfill leachate remains a technical bottleneck in sanitary landfilling (Harmsen, 1983; Han *et al.*, 2001; Ni *et al.*, 2004; Colomer Mendoza and Gallardo Izquierdo, 2009; Gotvajn *et al.*, 2009).

Bentonite can be applied to treat medium- and old-aged landfill leachate (>10 y old) (Zhou, 2007; Lu, 2005; Lu *et al.*, 2007). Due to the very hydrophilic surface and the easily hydrated interlayer of natural bentonite (Kaufhold *et al.*, 2010), its application to the treatment of organic pollutants has been limited. Many studies have focused on methods for bentonite modification, with a view to improving its capacity to adsorb organic pollutants (Lee *et al.*, 1989, 1990; Zhu *et al.*, 1998; Sharmasarkar *et al.*, 2000). Organically modified bentonite has been applied extensively in the treatment of simulated wastewater (Han *et al.*, 2001; Ozdemir and Keskin, 2009; Erdal, 2009; Xu and Zhu, 2009) and sewage (Smith and Jaffe, 1994; Shao *et al.*, 2008; Bortolotto *et al.*, 2009). Few studies have dealt with treatment of landfill leachate (Zhou, 2007; Lu *et al.*, 2007). The large cost of making OMB limits its large-scale application in treatment of landfill leachate (Zhou *et al.*, 2007).

In the present study, a series of low-cost OMBs was synthesized to treat the organic pollutants in landfill leachate efficiently. The structure of each OMB was identified by X-ray diffraction (XRD); the composition of the bentonite was established by means of X-ray fluorescence spectrometry (XRF); and the landfill leachate samples before and after treatment were characterized by gas chromatography-mass spectrometry (GC-MS) and COD measurement.

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MATERIALS AND METHODS

Materials

Landfill leachate samples were taken from a large landfill in Beijing, China. Bentonite was obtained from Jianping County in Liaoning Province, China; the bentonite is a solid yellow powder, having an average particle size of 14.124 μm (tested by MS2000 laser granularity analysis apparatus from Malvern Co. Ltd, England, measurement range: 0.02−2000 μm); the BET surface area is 67.505 m^2/g (tested using an ASAP2010 High-Speed Surface Area and Pore Size Analyzer (Micromeritics, USA)); the swelling index is 13 mL/g (Zhou *et al.*, 2007); and the cation exchange capacity (CEC) is 0.598 mol/kg (tested by the NH_4Cl-$NH_3{\cdot}H_2O$ method, Chen *et al.*, 2000).

The organic modifier, di-octadecyl dimethyl ammonium chloride (DODMA-Cl, $(C_{18}H_{32})_2(CH_3)_2NCl)$), was supplied (purity = 70%) by the China Daily Chemical Research Institute. Alum (A.R.), used as a flocculant, was supplied by the Beijing Chemical Reagent Co., Ltd.

Techniques

The chemical composition of bentonite (dried at 80ºC for 8 h prior to testing) was measured by XRF (Thermo Corporation ARL ADVANT XP+) using an excitation current of 50 mA, an excitation voltage of 50 kV, and a sensitivity of 0.001%.

The structures of the bentonite and the OMB were analyzed using a 12 kV Rigaku-RA high-power X-ray diffractometer (XRD) (Rigaku, Japan) using CuKα radiation (λ = 1.5406 Å). The bentonite and OMB were dried at 80ºC for 8 h prior to testing. The XRD patterns were collected in the 2θ range from 1.5 to 50º2θ at a scan rate of 4º2θ/min.

The organic pollutants in the landfill leachate before and after treatment were detected using GC-MS (a system combining a GC 7890A with a MS 5975C, Agilent Technologies, USA). The organic pollutants were extracted with dichloromethane. The methods of preparing the samples and the detailed analytical procedure were in accordance with the Environmental Protection Agency of the USA (Liu *et al.*, 2003, 2010). In the GC-MS determinations, the flow rate of the carrier gas (He) was 1 mL/min and the injection port temperature was 300ºC. A quartz capillary column (30 m × 250 μm × 0.25 μm) was used. The temperature of the column was started at 35ºC and held for 5 min, raised to 40ºC at 5ºC/min and held for 2 min, raised to 150ºC at 10ºC/min and held for 3 min, and then raised to 300ºC at 10ºC/min and held for 10 min. Mass spectrometry with electron-impact ionization (electron energy, 70 eV) was performed in selected ion monitoring (SIM) mode.

The COD concentration, an index of the organic pollutants in the landfill leachate, was determined using the potassium dichromate method (Ling *et al.*, 2011). The BOD_5 was determined using an OxiTop-12 BOD analyzer (WTW, Weilheim, Germany). The ammonia concentration was determined using the Nessler's reagent spectrophotometer method (Wang *et al.*, 2010). The pH was measured using a PB-10 type pH meter (Sartorius, Goettingen Germany).

Preparation of OMB

Bentonite, DODMA-Cl, and deionized water were added to each 250 mL flask at different ratios (1 g: 10 g: 125 mL, 2 g: 10 g: 125 mL, 3 g: 10 g: 125 mL, 4 g: 10 g: 125 mL, and 5 g: 10 g: 125 mL). The mixtures were then stirred at 250 rpm at 65ºC for 3 h and aged in a closed environment at 25±5ºC for 24 h. Finally, a series of OMB materials with different organic modification degrees (referred to as OMD) was prepared.

The reaction equation between the bentonite and the organic modifier is *B*-*X* + DODMA-Cl → DODMA-*X* + *B*-Cl (*B* represents exchangeable inorganic cations in bentonite, *X* represents the exchange positions of the bentonite surface). The OMD of the OMB can be calculated using the following equation:

$$\text{OMD} = (M \times P)/(W \times C) \times 100\%$$

where *M* represents the mass ratio between the organic modifier and the bentonite, *P* represents the purity of the organic modifier, *W* represents the molecular weight of the organic modifier, and *C* represents the CEC of the bentonite. The molecular weight of DODMA-Cl is 595.5 g/mol and its purity is 70%. The CEC of bentonite is 5.98×10^{-4} mol/g (Chen *et al.*, 2000). Therefore, the OMD of the OMB depends on the mass ratio (*M*) between the organic modifier and the bentonite. For example, if *M* is 1:10, then the OMD is equal to $(0.1 \times 0.7)/(595.5 \times 5.98 \times 10^{-4}) \times 100\% = 20\%$.

An OMD of 20% indicates that 20% of the interlamellar cations in bentonite have reacted with the organic modifier. Below, the term '0.2CEC OMB' is used to denote the 20% OMD of OMB.

Treatment procedure

Most of the ammonia nitrogen in the landfill leachate was pretreated by the struvite crystallization method (Zhou, 2007). The OMB was then used to treat landfill leachate which contained a small amount of ammonia nitrogen. Several experiments showed that the following process was more effective. First, OMB (10 g/L) was added to the landfill leachate at 25±5ºC and then stirred for 1.5 h at 200 rpm. Second, alum (15 g/L) was also added to the mixture, and was stirred for 1 min at 200 rpm and then for 10 min at 100 rpm. Third, the solid and the liquid were separated by the filtration method through a Buchner funnel (Zhou, 2007). Fourth, the liquid was reprocessed twice using all of the steps above. Fifth, the solid was carried back to landfill and the liquid was recycled or reprocessed. According to previous experiments, the OMD of the OMB played a significant role in the treatment. In order to estimate the influence

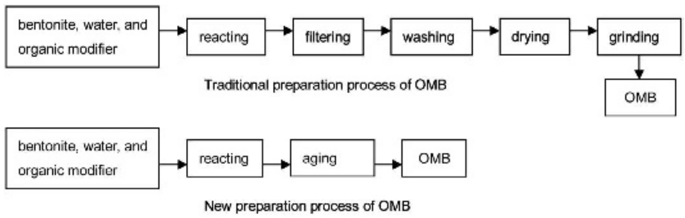

Figure 1. Comparison of traditional and new preparation processes for organically modified bentonite (OMB). The more modern version has four fewer steps (no filtering, washing, drying, or grinding).

of the OMD, five groups of OMB each with different OMDs were designed. The group OMDs were 20%, 40%, 60%, 80%, and 100%.

RESULTS AND DISCUSSION

Synthesis of OMB

The traditional method of preparing OMB is complex and costly (Lee *et al.*, 1989, 1990; Sharmasarkar *et al.*, 2000; Zhu and Chen, 2006; Zhou *et al.*, 2007; Xu and Zhu, 2009). The new process (Figure 1) eliminates the filtering, washing, drying, and grinding steps and, thus, consumes less energy and costs less to produce (Ling *et al.*, 2011).

Characterization of bentonite and OMB

The XRD pattern of the original bentonite sample was that of a typical Na-bentonite with strong lines for (001), (100), and (004), and d_{001} = 1.251 nm for smectite (Figure 2). The fraction of smectite in the bentonite was calculated to be 87%, with lesser amounts of feldspar, calcite, quartz, and pyrite also present. The chemical composition of bentonite is given in Table 1.

The OMDs of the OMBs were 20%, 40%, 60%, 80%, and 100%. The viscosity of the OMB decreased as the OMD increased, suggesting that the proportion of hydrophilic unmodified bentonite decreased and the hydrophobic modified bentonite components increased with the increase in OMD. The XRD patterns of OMB with different OMDs (Figure 3) revealed that the d_{001} value of the smectite mineral increased significantly after organic modification, indicating successful modification, *i.e.* that the long-chain molecules of the organic modifier had been inserted into the interlayer space of the smectite. The main diffraction peak (001) of the basal plane in the modified smectite of OMB shifted gradually to a lower angle as the OMD increased. The diffraction peak (001) of the original bentonite also appeared in the XRD pattern of the OMB, indicating that the OMB was a mixture containing both modified and unmodified bentonite. The organic modification had no effect on XRD peaks between 10 and 50°2θ, suggesting no reaction of the organic modifier with feldspar, calcite, quartz, or pyrite.

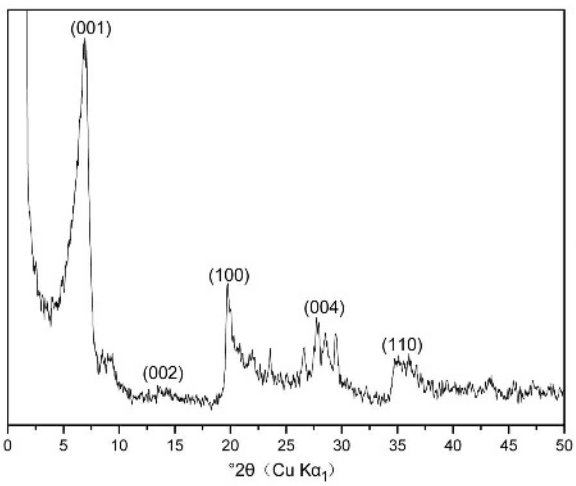

Figure 2. Random XRD pattern of the original bentonite, a typical Na-bentonite as indicated by the strong (001), (100), and (004) reflections, and d_{001} = 1.251 nm.

Landfill leachate treated by OMB

As seen from the typical physicochemical characteristics (Table 2) of the landfill leachate sample, such as the purple black color, high concentration of ammonia nitrogen, weak alkalinity, COD value between 2000 and 5000 mg/L, and small BOD_5/COD ratio (0.3), the leachate was clearly medium- and old-aged.

After pretreatment of the landfill leachate by struvite crystallization, the concentration of ammonia nitrogen was reduced from 3859 to 175 mg/L, corresponding to a removal efficiency of 95.5% (Zhou, 2007). Meanwhile, the COD decreased slightly from 2566 mg/L to 2400 mg/L. Subsequently, the leachate was treated with OMB. The COD reduction efficiency was poorest, 26.0%, (Figure 4) when the leachate was treated using the original bentonite (0% OMD of OMB). The efficiency was 89.8% when the OMD was 60%. The optimal efficiency reached 90.6% when the OMD was 80%. However, the efficiency decreased to 87.5% when the OMD was 100%.

The results demonstrate the following. (1) The original bentonite can remove some of the organic substances by adsorption. The exchangeable cations of the original bentonite were easily hydrolyzed, thus decreasing the effective CEC and adsorption performance, which also degraded the COD reduction efficiency. (2) When the OMD of the OMB was 40% or less,

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Table 1. Chemical composition of the Jianping bentonite (wt.%).

	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	TiO_2	P_2O_5	SO_3	MnO	LOI	Total
Bentonite	52.04	15.58	6.32	4.03	2.69	1.85	1.04	0.86	0.64	0.11	0.08	14.64	99.88

the OMB content was too small to treat hydrophobic organic compounds effectively such that the COD reduction efficiency was low. (3) When the OMD of the OMB was 60%, its treatment efficiency was almost the same as that of 80% OMD. Because of the cost of the organic modifier, the 60% OMD was preferred. (4) When the OMD of the OMB was 100%, the COD reduction efficiency decreased. A possible explanation for this is that as more organic modifier is added during OMB preparation, the greater the probability that some modifier fails to react with the bentonite and remains in solution to be detected by the COD measurement. (5) With the optimum OMD of 60%, the unmodified bentonite is fully utilized to remove hydrophilic organic pollutants. The unmodified bentonite fraction in the system can also react at the same time with organic compounds in the leachate, further improving the OMD of OMB itself and, consequently, enhancing its treatment capacity for the organic pollutants. The overall effect was, therefore, better when the OMD was 60%.

Mechanism of removal of organic pollutants by OMB

The XRD analysis showed that the OMB with 60% OMD was a mixture containing both modified and unmodified bentonite. The surface area and CEC of unmodified bentonite is comparatively large, so its adsorption performance is good; and because this OMB has negative charges and displays electrode performance (Li *et al*., 2007), it can absorb polar organic molecules and treat hydrophilic organic pollutants. Modified bentonite is hydrophobic so it can adsorb hydrophobic (non- or weakly polar) molecules. Moreover, the hydrophobic organic compounds could be allocated from the water phase in landfill leachate to the organic phase in modified bentonite so that a large number of hydrophobic organic compounds are treated (Chiou *et al*., 1979; Yang, 2004; Zhu and Chen, 2000, 2006). Therefore, the hydrophobic modified bentonite could eliminate the hydrophobic organic pollutants, and the hydrophilic unmodified bentonite could treat hydrophilic organic pollutants at the same time, so that the OMB could remove organic pollutants to a large extent from landfill leachate.

To further explore the mechanism of the OMB treatment of organic pollutants, the change in organic pollutants after treatment (with 60% OMD of OMB) was assessed using GC-MS (Figure 5). Landfill leachates are known to contain both hydrophilic and hydrophobic

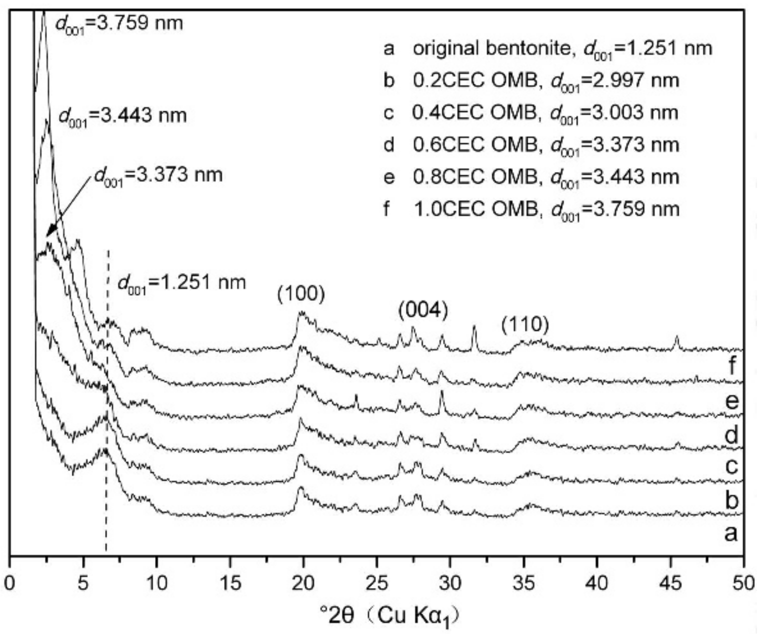

Figure 3. XRD patterns of (a) original bentonite; (b) 0.2CEC OMB (0.2CEC OMB represents the 20% degree of organic modification of the OMB, *i.e.* indicating that 20% of the interlamellar cations in bentonite reacted with the organic modifier) (c) 0.4CEC OMB; (d) 0.6CEC OMB; (e) 0.8CEC OMB; and (f) 1.0CEC OMB.

Table 2. Physicochemical characteristics of the landfill leachate (tested on 25 December 2007).

COD	BOD_5	Ammonia nitrogen	pH
2566 mg/L	450 mg/L	3859 mg/L	8.0–8.2

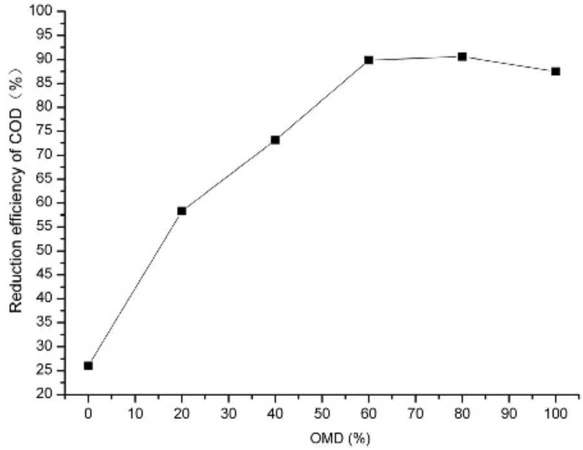

Figure 4. The COD reduction efficiency of landfill leachate as affected by the organic modification degree (OMD) of the OMB. For OMD = 60%, the COD removal efficiency = 89.8% and the material cost is lower.

organic pollutants (alkanes, alkenes, alcohols, ketones, carboxylic acid, benzene, esters, amides, pyrrole, furan ketone, pyridine, phenols, indole, hydrazone, siloxane, *etc.*, >70% of which are hydrophobic) (Liu *et al.*, 2003, 2010). Four organic compounds typical of these (Table 3) were reacted with the 60% OMD of OMB. The GC-MS test results showed that the OMB decreased significantly the amounts of hydrophilic (dibutyl phthalate and 10-methylnonadecane) and hydrophobic (cyclopropanecarboxamide) organic substances, except for propanamide, present in landfill leachate. The ineffective treatment of propanamide may be due to the fact that propanamide is amphoteric and, because of the presence of some ammonia nitrogen (175 mg/L), competitive adsorption between propanamide and ammonia nitrogen may have occurred. With OMB, ammonia nitrogen would probably be adsorbed first in a landfill leachate.

CONCLUSIONS

In the present study, a new procedure for synthesizing OMB was developed, using DODMA-Cl as the organic modifier; then it was used as an adsorbent to remove organic pollutants from landfill leachate. Compared with the traditional method of synthesizing OMB, the new method eliminated filtering, washing, drying, and grinding steps, significantly reducing the energy consumption as well as production cost. The OMB contained a mixture of bentonite surfaces which were modified and unmodified with the DODMA-Cl. During the treatment of organic pollutants, the unmodified bentonite fraction of the OMB was able to remove the hydrophilic organic pollutants, and the modified bentonite fraction of the OMB simultaneously eliminated the hydrophobic organic pollutants. The COD concentration in the landfill leachate was reduced from 2400 to 245 mg/L within 5 h.

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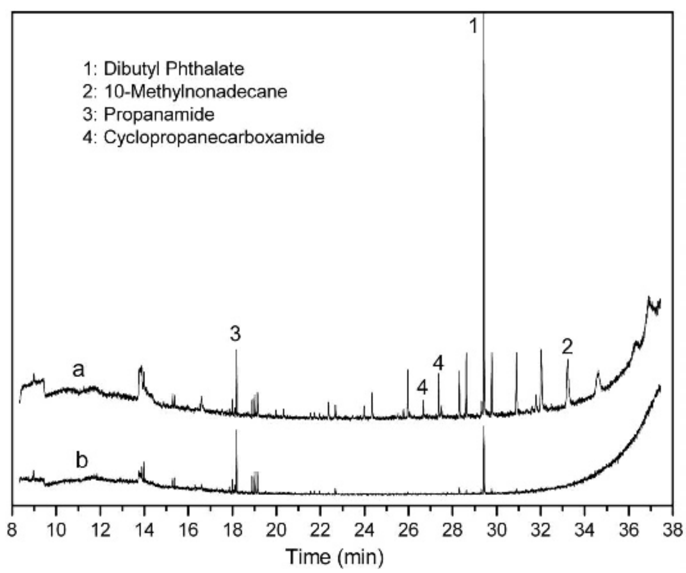

Figure 5. GC-MS analysis of landfill leachate (a) before and (b) after treatment with 0.6 CEC OMB.

Table 3. Typical organic substances and their characteristics in landfill leachate.

	CAS number	Molecular formula	Content (%)	Types
Dibutyl phthalate	000084-74-2	$C_{16}H_{22}O_4$	18.236	Hydrophobic
10-Methylnonadecane	056862-62-5	$C_{20}H_{42}$	6.887	
Propanamide	000079-05-0	C_3H_7NO	2.657	Amphoteric (Hydrophilic and Lipophilic)
Cyclopropanecarboxamide	006228-73-5	C_4H_7NO	1.187	Hydrophilic

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