Research Article

Preparation and Characterization of Montmorillonite Intercalation Compounds with Quaternary Ammonium Surfactant: Adsorption Effect of Zearalenone

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Montmorillonite (Mt) was used as the original material to prepare intercalation compounds with quaternary ammonium surfactant (QAS). The adsorption of zearalenone (ZEA) onto Mt and organomodified Mt was investigated in vitro. Effects of QAS in binding ZEA were studied. By the method of intercalation with dioctadecylmethylbenzylammonium chloride (DOMBAC), the sample exhibited the highest adsorption rate of ZEA (93.2%) which was much higher than that of Mt (10.5%). Several methods were adopted to characterize samples, including XRD, TG/DSC, N₂ adsorption/desorption, and FTIR. Adsorption isotherm parameters were obtained from Langmuir and Freundlich and the adsorption data fitted better to Langmuir. All results indicate that organomodified Mt has great potential to be a high-performance material to control ZEA contamination.

1. Introduction

Many types of clay minerals such as montmorillonite (Mt), sepiolite, and kaolinite have shown high adsorption capacity. Mt, a clay mineral with 2:1 layered structure, can hold some cations between its layers. Substitution of Si⁴⁺ with Al³⁺ in tetrahedral sheets and Al³⁺ with Mg²⁺ in octahedral sheets make the lattice a net negative charge which is usually balanced by cations of Li⁺, Na⁺, and Ca²⁺ located between the layers. These cations can be easily replaced by other organic or inorganic cations under certain conditions, which endow Mt with some special properties [1]. Mt has been widely used in industry and agriculture, particularly in adsorption of mycotoxins. According to the Food and Agriculture Organization (FAO), 25% of the world's cereal grain production is contaminated by mycotoxins [2]. Zearalenone (ZEA), along with others most of mycotoxins, commonly found in animal feeds, can cause serious health problems in livestock [3, 4]. Zearalenone is most notorious for its

effects on precocious development of mammae and other estrogenic effects in young gilts [5]. The chemical structure of ZEA is presented in Figure 1 [6]. It has been reported that natural Mt are effective in adsorbing aflatoxins in vitro and in vivo [7, 8]. However, their hydrophilic negatively charged surfaces are less effective in binding other mycotoxins, which are more hydrophobic, such as ZEA. According to some literatures, organic modification of clays with organic cations, surfactants, can result in high affinity for in vitro adsorption of hydrophobic ZEA [4–9].

The quaternary ammonium surfactant (QAS) is the most commonly used organic modifier, which can enhance the lipophilicity of Mt. However, QAS contains many different kinds such as the types with short carbochain, medium carbochain, long carbochain, and double carbochain and the type with functional group of benzyl. In this study, Mt intercalation compounds with QAS were prepared and characterized; the adsorption effect of ZEA onto adsorbents was compared. The objective of the research is to investigate

FIGURE 1: The molecular structural formula of ZEA.

the adsorption of ZEA onto Mt intercalation compounds with QAS and explore the optimum QAS in binding ZEA. To the best of our knowledge similar research has not been reported.

2. Materials and Methods

2.1. Preparation and Characterization of Mt. Mt, the original materials supplied by Sanding Company, consists of a lot of Mt and a small amount of quartz, according to powder X-ray diffraction (XRD) analysis shown in Figure 2. The primary ion in the exchangeable position is sodium and the cation exchange capacity of Mt is 1.44 mmol/g [10]. Mt was dried at 80°C to constant mass and then milled to less than 45 μm . QAS, supplied by Daochun Company, includes dodecyltrimethylammonium chloride (DTAC), octadecyltrimethylammonium chloride (OTAC), behenyltrimethylammonium chloride (BTAC), dodecylmethylbenzylammonium chloride (DDBAC), octadecylmethylbenzylammonium chloride (ODBAC), and dioctadecylmethylbenzylammonium chloride (DOMBAC), and the corresponding organomodified Mt is MDTAC, MODAC, MBTAC, MDDBAC, MODBAC, and MDOMBAC, respectively. The molecular structural formulas of QAS are shown in Table 1.

For preparation of organomodified Mt, which was synthesized by interaction (cation exchange) of Mt with OAS, Mt reacted with 1.44 mmol/g QAS at 90°C in distilled water, mixing speed 200 rpm for 1 h, according to the procedure described previously [11]. When the reaction was over, the suspensions were centrifuged and washed by distilled water for several times and dried at 80°C to constant mass and then milled to less than 45 μm . The effect of different kinds of QAS on the adsorption of ZEA was investigated according to the adsorption capacity of ZEA.

Samples were characterized by X-ray powder diffraction (XRD) analysis, thermogravimetric/differential scanning calorimetry analysis (TG/DSC), N_2 adsorption/desorption, and Fourier transform infrared spectroscopy (FTIR) analysis. XRD analysis was performed on a Rigaku D/max-3Bx diffractometer with $\text{Cu K}\alpha$ radiation, in the range of 5–70° 2θ and at a step of 0.02°. TG/DSC analysis was performed on a STA 409 thermal analysis instrument, from 25°C (room temperature) to 1200°C and with heating rate of 10°C/min. Brunauer-Emmet-Teller (BET) specific surface area (S_{BET}), total pore volumes (V_{total}), and average pore diameter (D_p) of the samples were measured from N_2 adsorption/desorption isotherms at 77 K, using an automatic specific surface area

M: Montmorillonite

Q: Quartz

FIGURE 2: XRD patterns of (a) Mt and (b) MDOMBAC.

measuring equipment (ASAP, 2020M, USA) after a degassing under vacuum for 8 h at 110°C. Infrared spectra were recorded by means of Thermoelectron FTIR spectrometer (Avatar370) in the range of 4000–400 cm^{-1} .

2.2. Adsorption Experiments. The method of enzyme linked immunosorbent assay (ELISA) was adopted to measure the amount of ZEA which was purchased from Sigma-Aldrich Co., and ELISA kits were purchased from R-Biopharm Co. Firstly, ZEA stock solution (4 $\mu\text{g/mL}$) was prepared, then 10 mg of organomodified Mt was added to tube that was filled with 10 mL ZEA solution (4 $\mu\text{g/mL}$) whose pH was adjusted to 2 by adding 0.1 M phosphate buffer as required. After 1 h reaction at 37°C in temperature-controlled shaking water bath pot at a shaking rate of 120 rpm, the tube was centrifuged for 10 min at 5000 rpm and then the amount of ZEA remaining in the supernatant layer of suspension was analyzed with the method of ELISA. The adsorption capacities were calculated from the difference between initial and equilibrium concentrations of ZEA. For isotherm study, Mt (10 mg) was added to 10 mL ZEA solution (pH = 2, 37°C) at different concentrations (0.25, 0.5, 1.0, 2.0, 4.0, and 6.0 $\mu\text{g/mL}$ resp.) and so was organomodified Mt but with increased concentrations of ZEA solution (1.0, 2.0, 4.0, 6.0, 8.0, and 10.0 $\mu\text{g/mL}$, resp.) due to its higher adsorbance of ZEA. The adsorption data were fitted to the models of Langmuir and Freundlich isotherm.

3. Results and Discussion

3.1. Effects of QAS. As can be seen from Table 1, Mt is quite ineffective in adsorbing ZEA (10.5%) due to its poor lipophilicity and different kinds of QAS can draw significantly different influences on the adsorption of ZEA. For QAS without benzyl, the type with medium carbochain is beneficial to the adsorption of ZEA because MOTAC obtains the maximum adsorbance of ZEA (45.8%), which is higher than that of MDTAC (24.7%) and MBTAC (36.5%). The

TABLE 1: Effects of QAS on the adsorbance of ZEA on adsorbents.

Samples	QAS	Molecular structural formula of QAS	Initial amount of ZEA (μg)	Adsorption rate (%)
Mt	—	—	40	10.5
MDTAC	DTAC	$\begin{array}{c} \text{CH}_3 \\ \\ \text{H}_3\text{C}-\text{N}^+-\text{C}_{12}\text{H}_{25}\text{Cl}^- \\ \\ \text{CH}_3 \end{array}$	40	24.7
MDDBAC	DDBAC	$\begin{array}{c} \text{CH}_3 \\ \\ \text{C}_6\text{H}_5-\text{CH}_2-\text{N}^+-\text{C}_{12}\text{H}_{25}\text{Cl}^- \\ \\ \text{CH}_3 \end{array}$	40	50.8
MOTAC	OTAC	$\begin{array}{c} \text{CH}_3 \\ \\ \text{H}_3\text{C}-\text{N}^+-\text{C}_{18}\text{H}_{37}\text{Cl}^- \\ \\ \text{CH}_3 \end{array}$	40	45.8
MODBAC	ODBAC	$\begin{array}{c} \text{CH}_3 \\ \\ \text{C}_6\text{H}_5-\text{CH}_2-\text{N}^+-\text{C}_{18}\text{H}_{37}\text{Cl}^- \\ \\ \text{CH}_3 \end{array}$	40	69.1
MBTAC	BTAC	$\begin{array}{c} \text{CH}_3 \\ \\ \text{H}_3\text{C}-\text{N}^+-\text{C}_{22}\text{H}_{45}\text{Cl}^- \\ \\ \text{CH}_3 \end{array}$	40	36.5
MDOBAC	DOBAC	$\begin{array}{c} \text{C}_{18}\text{H}_{37} \\ \\ \text{C}_6\text{H}_5-\text{CH}_2-\text{N}^+-\text{C}_{18}\text{H}_{37}\text{Cl}^- \\ \\ \text{CH}_3 \end{array}$	40	93.2

result is attributed to that the lipophilicity of MOTAC with longer carbochain is higher than that of MDTAC, but when the carbochain is too long, it is hard for the intercalation reaction which is adverse to the organic modification [12]. Moreover, the influence of intercalation reaction on pore size of adsorbent has two completely opposite effects [13]: the “pore blockage” effect, organic molecules fill pores and result in the decrease of pore size and S_{BET} , and the “pore prop” effect, macromolecular organics insert into the interlayer and cause the distraction of aluminosilicate layer and the

increase of pore size and S_{BET} . Hence, the molecule sizes of QAS have great influences on the absorption capacity of adsorbents. Meanwhile, benzyl group has great influences on the adsorption of ZEA, which can significantly increase the adsorbance of ZEA on adsorbents. Herein, the adsorbance of ZEA on MDDBAC (50.8%) and MODBAC (69.1%) is higher than that of MDTAC and MOTAC, respectively. It can be attributed to that the critical effect of benzyl ring into the interlamellar space of Mt greatly enhances the penetration of QAS, which results in a better effect of modification; besides,

FIGURE 3: TG/DSC curves of Mt and MDOBAC.

benzyl is beneficial to the increase of lipophilicity [14]. Furthermore, MDOBAC has higher adsorption capacity of ZEA (93.2%) than that of MODBAC, which is the maximum in all of the organomodified Mt, indicating that optimal QAS with double medium carbochain and benzyl group does favor the adsorption of ZEA onto adsorbent. The result obtained shows that QAS plays an important role in binding ZEA and it can be attributed to the physicochemical property of QAS which has influences on the dispersion and lipophilicity of organomodified Mt [15–17].

3.2. XRD Characterization. XRD patterns reveal the microstructure variety of samples. The comparative XRD patterns of Mt and MDOBAC are presented in Figure 2. It shows that the d_{001} values of Mt and MDOBAC are 1.26900 and 1.93526 nm, respectively, indicating that Mt is intercalated by dioctadecylmethylbenzylammonium (DOMBA) ions because the radius of DOMBA ions is larger than that of sodium ion [18]. Besides, there is no significant difference between their patterns, indicating that the basic structure of Mt is not changed. By the way of organic intercalation, MDOBAC can obtain greater surface area and become more porous theoretically, which can improve its adsorption ability of ZEA greatly [19]. Therefore, the adsorption capacity of ZEA on MDOBAC is much higher than that of Mt.

3.3. TG/DSC Characterization. The TG/DSC curves of Mt and MDOBAC are shown in Figure 3. The DSC curves of Mt show that the first endothermic valley appears at 98.3°C for the loss of the physically adsorbed water and the second endothermic valley appears at 153.9°C corresponding to the loss of the interlayer water. About 5.3% of weight loss is shown in TG curve of Mt. With the increase of temperature, the third endothermic valley appears at 689°C with 5.26% of weight loss, which is due to the dehydroxylation. Comparatively, the TG/DSC curves of MDOBAC show that there are only one endothermic valley at 73.1°C for the loss of physically adsorbed water and interlayer water, because of the less moisture content caused by hydrophobicity after organic intercalation. Between 400 and 700°C, a great exothermic

TABLE 2: The structure characteristics of Mt and MDOBAC.

Sample	S_{BET} (m^2/g)	V_{total} (cm^3/g)	D_p (nm)
Mt	41.217	0.087	7.854
MDOBAC	147.745	0.276	9.121

peak appears due to the burning of organism and then a lot of heat releases and about 43.73% of weight loss occurs, which is close to the adding quantity of DOMBAC. When the temperature rises continuously, the decomposition of MDOBAC occurs with 1.32% of weight loss [20, 21]. The results obtained show that the dosage of DOMBAC is proper and the structure of MDOBAC is stable below 400°C, confirming that DOMBA ions added are bound in the interlayer space of MDOBAC [22].

3.4. N_2 Adsorption/Desorption. The S_{BET} , V_{total} , and D_p of Mt and MDOBAC were measured, and all of the data are summarized in Table 2. The N_2 adsorption/desorption isotherms and pore size distribution curves of samples are shown in Figure 4. It shows that isotherms exhibit Type II behaviors according to the IUPAC classification [23], and the D_p of samples was about 7–9 nm, characteristics of major mesoporous adsorbents, seen from the formed hysteresis loop which fits to the fourth type of hysteresis loop (H4), indicating slit pores formed from layer structure [23]. The main differences obtained from the isotherms are that the S_{BET} and V_{total} of MDOBAC are significantly larger than that of Mt, indicating the different pore structure of MDOBAC with more mesoporous. It can be attributed to the “pore prop” effect; meanwhile, the incorporation of DOMBA ions and Mt in the interlayer area forms a more porous intercalation compound [24]. The results demonstrate that, with the special pore structure formed by intercalation, MDOBAC obtains a higher adsorption capacity of ZEA than that of Mt.

3.5. FTIR Characterization. The FTIR spectra of Mt and MDOBAC are shown in Figure 5. The spectra of Mt shows that the stretching vibration band of Al–OH–Al group at octahedral layer is at about 3668 cm^{-1} and its bending vibration band is at 915 cm^{-1} . The band at around 3430 cm^{-1} is the stretching vibration of interlayer water molecule and at 1639 cm^{-1} is the bending vibration. The two bands are observed at 1077 cm^{-1} and 1031 cm^{-1} in the Si–O–Si stretching vibration region and the former is stronger; the band at 463 cm^{-1} is the bending vibration of Si–O–Si. The weak band at 525 cm^{-1} is bending vibration of Si–O–Al and the band at 834 cm^{-1} is due to stretching vibration of Al^{IV} tetrahedra, when replacing Si with Al [25]. After intercalation with DOMBAC, three new bands are observed at 1473 cm^{-1} , 2921 cm^{-1} , and 2852 cm^{-1} which are bending vibration, asymmetrical stretching vibration, and symmetrical stretching vibration of CH due to the introduction of DOMBA ions [26]. These results suggest that Mt and DOMBA ions have been combined together, which results in more active sites on MDOBAC and higher adsorption capacity of ZEA.

FIGURE 4: The N_2 adsorption/desorption isotherms of (a) Mt and (b) MDOMBAC and the insert figures are pore size distribution curves.

FIGURE 5: FTIR spectra of Mt and MDOMBAC.

3.6. Adsorption Isotherms. It is important to analyze the isotherm data so as to investigate the mechanism of ZEA adsorption by Mt and MDOMBAC, and the results can help realize better design to remove ZEA from the foods for human and animal. Therefore, two main models of adsorption isotherm were analyzed at pH = 2, Langmuir and Freundlich, respectively [27, 28].

The Langmuir adsorption isotherm assumes that adsorption occurs at specific homogeneous sites inside the adsorbent. The adsorption data were fitted to the linear form of Langmuir adsorption model (1) and the adsorption isotherm was obtained by plotting the particular adsorption (C_e/C) against the equilibrium concentration of ZEA (C_e). C_e is the equilibrium concentration of ZEA which means the final concentration (mg/L) of ZEA in the solution after adsorbed, C_m is the maximum adsorbance of the sorbent, C is the

amount of ZEA adsorbed by the adsorbent at equilibrium, and K is a Langmuir constant related to the adsorption energy:

$$\frac{C_e}{C} = \frac{1}{C_m K} + \frac{C_e}{C_m}. \quad (1)$$

Secondly, the adsorption data were fitted to the linear form of Freundlich adsorption model:

$$\ln C = \ln K_f + \frac{1}{n_f} \ln C_e, \quad (2)$$

where K_f is Freundlich constant related to adsorption intensity and n_f is constant related to adsorption intensity as well; C and C_e denote adsorbance of ZEA and equilibrium concentration of ZEA, respectively. Freundlich equation is an exponential variation in site energies and it is assumed that the adsorbent has heterogeneous energy distribution of active sites. Theoretically, Freundlich model is suitable for use with heterogeneous surfaces and with this expression; an infinite amount of adsorption can occur.

The absorption isothermal curves (Figure 6) and all isotherm values (Table 3) display that the data of Mt fit to the Langmuir model much better ($R^2 = 0.9997$), whereas the Freundlich model is less appropriate ($R^2 = 0.8817$); meanwhile, comparing the R^2 values of MDOMBAC, the Langmuir model obtains much better fit too, indicating that isotherms of both Mt and MDOMBAC are consistent with the Langmuir model and conform to monolayer adsorption over a homogenous adsorbent surface. The results show that organic intercalation does not change the adsorption mode. The C_m values of Mt and MDOMBAC shown in Table 3 present that the adsorbance of ZEA on MDOMBAC is higher than that of Mt; as the type of interlayer ions is the key factor for the adsorption [29, 30], it indicates that interlayer ions of DOMBA in MDOMBAC have stronger interaction with ZEA than that of sodium ions in Mt.

FIGURE 6: Adsorption isotherms of ZEA onto (a) Mt and (b) MDOBAC.

TABLE 3: Isotherm parameters for ZEA adsorption onto Mt and MDOBAC.

Sample	Langmuir			Freundlich		
	C_m (mg/g)	K (L/mg)	R^2	K_f (mg/g)	n_f	R^2
Mt	0.4529	4.1156	0.9997	0.3140	3.8610	0.8817
MDOMBAC	5.0761	6.6555	0.9989	3.497	1.8392	0.8199

4. Conclusions

The final results demonstrate the adsorption of ZEA onto Mt and organomodified Mt. By the method of intercalation with DOMBAC, an effective adsorbent of MDOBAC for removing ZEA was prepared, which exhibited the highest adsorption rate of ZEA (93.6%). Mt and MDOBAC were characterized by several methods, including XRD, TG/DSC, N_2 adsorption/desorption, and FTIR. According to the analysis of results, it can be concluded that the higher adsorption capacity of ZEA on MDOBAC mainly lies in the factors of greater surface area, higher porosity, much more active sites, and hydrophobicity. Determination of ZEA adsorption isotherms for Mt and MDOBAC shows that the ZEA adsorption follows the Langmuir isotherm model and the maximum adsorbance of ZEA on adsorbents was estimated to be 5.0761 mg/g.

Generally speaking, in the practical application, detoxification treatments with MDOBAC are technically and economically reliable, including (a) the elimination of ZEA, (b) not having any secondary pollution, (c) the protection of nutritive value, and (d) the antibacterial activity. Meanwhile, this adsorbent is cost effective and has high removal efficiency for ZEA from aqueous and organic solutions. Furthermore, the adsorbent is adaptable and stable when applied in different environments, which are the most critical properties for the removal of ZEA with variant concentrations in natural contaminated food. Hence, in order to prevent the hazardous effects of ZEA, Mt intercalation compound with

DOMBAC can be used as the potential adsorbent to prevent mycotoxicosis.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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