

Microwave enhanced-sorption of dyestuffs to dual-cation organobentonites from water

Jiwu Li^{a,b}, Lizhong Zhu^{a,*}, Weijian Cai^b

^a Department of Environmental Sciences, Zhejiang University, Hangzhou, Zhejiang 310028, China

^b Zhejiang Gongshang University, Hangzhou, Zhejiang 310035, China

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Abstract

The microwave enhanced-sorption of dyestuffs such as Neutral Red S-BR, Neutral Dark Yellow GL and Acid Blue B onto organobentonites from water was investigated. The decolorization rates of various dyestuffs by organobentonites were increased from 18.0% to 71.8%, the saturated sorption capacity of Neutral Red S-BR and Acid Blue B were increased 83.9% and 76.3% by microwave irradiation, respectively. The value of the microwave enhanced-sorption parameter R_m increased in the following order: Neutral Red S-BR > Acid Blue B > Neutral Dark Yellow GL, which corresponded with their aqueous solubility. The zeta potentials of particles were decreased greatly by microwave, which is very significant for improving both sorption of dyestuffs to organobentonites from water and the separation of the adsorbents from treated water.

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1. Introduction

Organic dyestuffs are common contaminants in wastewaters generated from textile and dye producing industries. Many dyestuffs are considered as the priority and hazardous pollutants in wastewater. Attempts have been made to remove dyestuffs from water by adsorption, chemical flocculation, biological degradation and chemical oxidation [1]. Adsorption is the popular method in which activated carbon is a common adsorbent. The adsorption of organic compounds on activated carbon is partly controlled by physical interactions, including size exclusion and microporosity effects [2,3]. However, because of its relatively high cost, there have been attempts to utilize low cost and naturally occurring adsorbents for the removal of contaminants from wastewater [1,4,5].

In recent years, there has been an increasing interest in utilizing natural or modified bentonite materials for the removal of some organic pollutants, such as phenol and benzene [4–7], 8-hydroxyquinoline [8], polycyclic aromatic hydrocarbons [9,10], *p*-nitrophenol [11] and dyes [12–16] from aqueous solutions.

Owing to the hydrophilic environment at surface, natural bentonite is not an effective adsorbent for organic compounds in water although it's high surface area. As is well known, simple ion-exchange reactions can significantly modify the surface properties of natural bentonite. When organic cations (cationic surfactants) of the form $[(CH_3)_3NR]^+$, where R is an alkyl hydrocarbon, occupy the exchange sites of bentonite clay, the surface properties transform from hydrophilic to hydrophobic [17–19]. Organobentonites were found to be effective for removing organic contaminants from wastewater treatment [20–22]. However, the colloidal suspension of organobentonites forms in processing of sorption of organic pollutants in water, separating small powdered organobentonites particles from treated water may be very awkward.

Generally, the stability of colloid is destroyed by the conventional thermal processing method, which is lower energy efficiency and longer heating time. Microwave is electromagnetic waves in the frequency range from 300 MHz to 30 GHz. Microwave generators usually work at 2450 or 915 MHz. Microwave processing offers several advantages over the conventional thermal processing method. These advantages include fast, selective and volumetric heating, high controllability and economical way [23–25]. Microwave has widely been applied for heating or digesting in chemistry and material synthesis

* Corresponding author. Tel.: +86 571 88273733; fax: +86 571 88273450.
E-mail addresses: lijw258@sina.com, zlz@zju.edu.cn (L. Zhu).

[24–29]. Those studies have demonstrated that microwave can accelerate reaction velocity and reduce the activation energy of reaction system [23]. However, little information on using microwave irradiation (MWI) to enhance sorption of organic compounds onto organobentonite and separation of small powdered particles from treated water is available.

The main objective of this study was to explore the possibility of using microwave for enhancing sorption of three dyestuffs and separation of small powdered organobentonite particles from treated water. To the end, mechanisms for microwave affecting the sorption properties of dyestuffs onto organobentonites from water were discussed.

2. Material and methods

2.1. Materials

Bentonite used was primarily Na⁺-montmorillonite from Lin'an Zhejiang, China. Its cation-exchange capacity (CEC) was 0.746 mmol/g of clay. Cetylpyridinium chloride (CPC), tetramethylammonium bromide (TMAB) used were of analytical grade. Neutral Dark Yellow GL (NDYGL) (color index: Acid Yellow 128), Neutral Red S-BR (NRS-BR) (color index: Acid Red 362) and Acid Blue B (ABB) (color index: Acid Blue 45) were of industrial grade. These dyestuffs are widely used to

Table 1

The molecular weight and the aqueous solubility of dyestuffs at 25 °C

Dye	Molecular weight (g/mol)	Aqueous solubility (g/l)
NRS-BR	932	140
NDYGL	485.5	35
ABB	474	55

dyeing for wool, polyamide fibre and cotton in textile industry. The molecular weight and the aqueous solubility of dyestuffs are presented in Table 1. Structures of surfactant and dyestuff are presented in Fig. 1.

2.2. Preparation of organobentonites

Comparing with other organobentonites, 60CPC/40TMAB-bentonite (abbreviation as 60CPC/40TMAB) is good adsorbents with higher removal rates of organic compounds from water [7,20]. Thus, the 60CPC/40TMAB was synthesized by reacting bentonite with solutions containing cationic surfactants CPC and TMAB. The 60CPC/40TMAB identified an organobentonites had 60% of its cation-exchange capacity satisfied by cetylpyridinium cations and an additional 40% satisfied by tetramethylammonium cations. This was done as follows: a total of 20 g of previously dried bentonite was mixed with 200 ml of solutions of

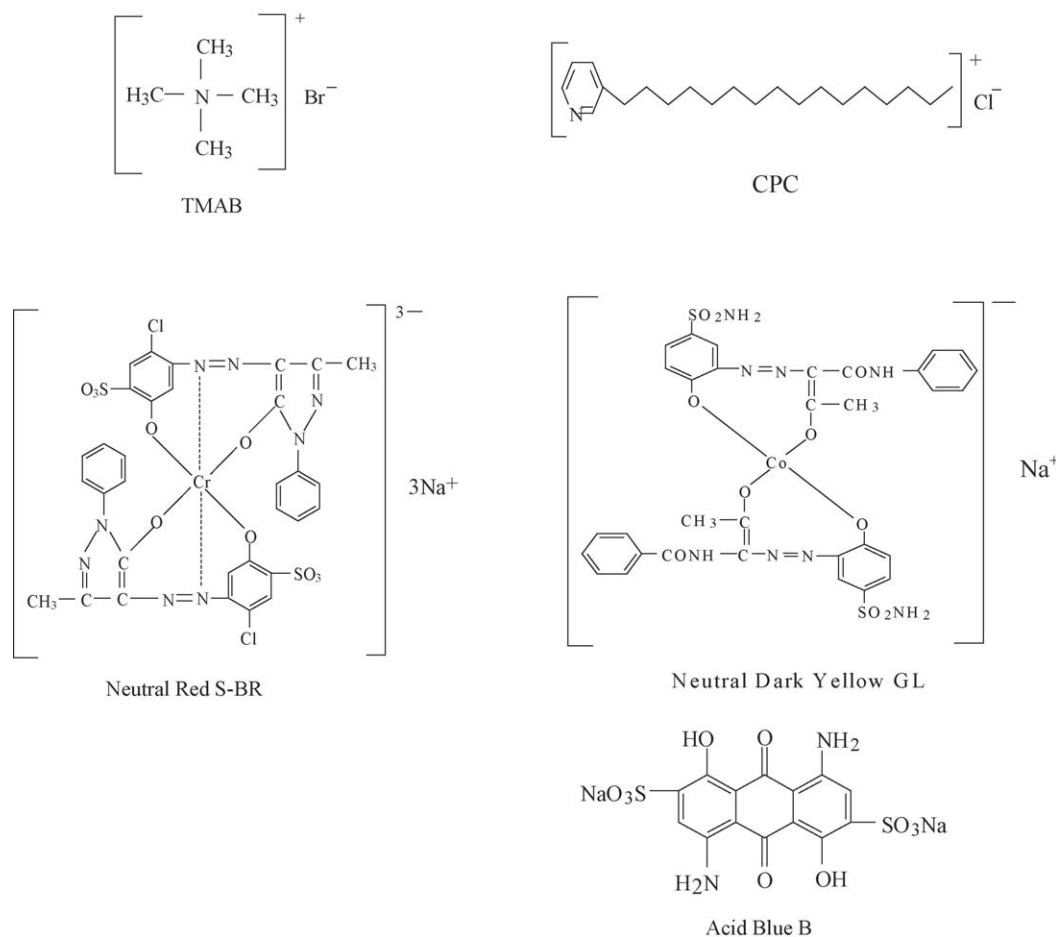


Fig. 1. Structures of cationic surfactant and dyestuff.

CPC (60% CEC) and TMAB (40% CEC). The mixture was subjected to mechanical stirring for 2 h in water bath at 60–70 °C. The final bentonite was separated from water by vacuum filtration and washed twice by deionization water. The samples were dried at 80 °C and activated for 1 h at 105 °C, and mechanically ground with a mortar and pestle to less than 100 mesh. The organic carbon content of the prepared organobentonites was determined by LECO CS-344 type carbon-sulfate analyzer.

2.3. X-ray diffraction analysis

The interlayer spacing of natural bentonite and organobentonites were examined by X'PertMPD type X-diffraction analyzer with Cu K α radiation, operated at 40 kV, and at the condition of 65% relative humidity and 22 °C.

2.4. Analytical methods

A suspension containing 50 mg organobentonites with 50 ml of 25 mg/l concentration of dyestuffs was prepared and irradiated by microwave for certain time, and then centrifuged for 10 min. The upper portions of dilute aqueous were analyzed by UV–vis spectroscopy using a UV-2450 spectrophotometer. The maximum absorption wavelengths of NRS-BR, NDYGL and ABB were 520, 410 and 560 nm, respectively. The detected limit was 0.05 mg/l.

2.5. Batch settling experiment

A 0.3 g sample of organobentonites was mixed with 50 ml deionization water in 150 ml Erlenmeyer flask with glass caps for 1 min, and then was irradiated by microwave for 1 min. The mixture was put into 50 ml colorimetric tube for settling process. The concentration of organobentonite in the suspension was determined by a UV-2450 spectrophotometer with 10 mm cells at the max absorption wavelength of 650 nm [6]. A calibration curve of the absorbance versus organobentonites concentration was obtained. Sample was taken at a series of settling time (at 5, 10, 15, 20, 25, 35, 45 and 60 min), and the absorbance was determined. The removal percentage of organobentonites was calculated.

2.6. The zeta potential measurement

A suspension containing 0.1 wt.% bentonite or organobentonites with 50 ml of 25 mg/l concentration of dyes was prepared. The mixture was conditioned by magnetic stirring for 5 min, and irradiated by microwave for 1 min. After settling for 20 min to stability, the upper portion of dilute fine particles suspension was taken for the zeta potential measurement.

The zeta potential measurement was carried out using a Zetaphometer (JS94G⁺). The computerized operating system allowed an accurate positioning of camera view field at a stationary layer to achieve accurate measurement of electrophoretic mobility. About 0.5 ml of the prepared suspension was used to fill the electrophoresis cell. Through the laser-illuminating and video-viewing system, the movement of particles in the station-

ary layer was traced. The captured images were then analyzed by the built-in imaging processing software, the zeta potential were determined. In this study, environmental temperature was maintained at 25 °C.

2.7. Sorption experiment of dyestuff

In conventional sorption experiment, a total of 50 mg of organobentonites was combined with 50 ml of 25 mg/l concentration of dyestuffs in 150 ml Erlenmeyer flask with glass caps. The initial pH of dye solution was ranged from 6.0 to 6.6. The flasks were shaken for 30 min at 25 °C on the gyratory shaker (power 750 W) at 150 rpm. After centrifuged, the dyestuff concentration in the aqueous phase was determined. The decolorization rates of dyestuffs from water by organobentonites or bentonite were calculated using the following relationship:

$$\text{Decolorization rate} = \frac{100(C_i - C_f)}{C_i} \quad (1)$$

where C_i and C_f are the initial and final concentration (in mg/l) of dyestuff, respectively.

In MWI sorption experiment, a total of 50 mg of organobentonites was combined with 50 ml of 25 mg/l of dyestuffs in 150 ml glass reactor in microwave cavity. The initial pH of dye solution was ranged from 6.0 to 6.6. The mixed dye solution with organobentonites or bentonite was irradiated for 5 min to sorption equilibrium by microwave (power 700 W) at 80 °C. After being irradiated, the mixed dye solution with organobentonites or bentonite was cooled in room temperature (25 °C), then centrifuged. The dye concentration in the aqueous phase was determined. The decolorization rates of dyes from water by organobentonites or bentonite were calculated using Eq. (1).

The losses of the organic compounds by photochemical decomposition, microwave decomposition, volatilization and sorption to the Erlenmeyer flask were analyzed by contrasting to the blank experiment, the results of which were found to be negligible.

3. Results

3.1. Structure of Organobentonite

Fig. 2 shows XRD patterns for organobentonites and natural bentonite. It indicates that the interlayer spacing of organobentonites and natural bentonite are 1.877 and 1.538 nm, respectively. The organic carbon content of organobentonites and natural bentonite are 9.39% and 0.21%, respectively. The primary silicate in the organobentonites corresponds to a d_{001} layer spacing of 1.877 nm, which represents an increase of approximately 0.339 nm from the Van Der Waals height in the original Na⁺-montmorillonite. This indicates cationic surfactant had entered into interlamellar of the bentonite by cation exchange.

3.2. The zeta potential results

In general, the zeta potential measurement of the solid–liquid interface can be used to study the nature of a solid surface. The

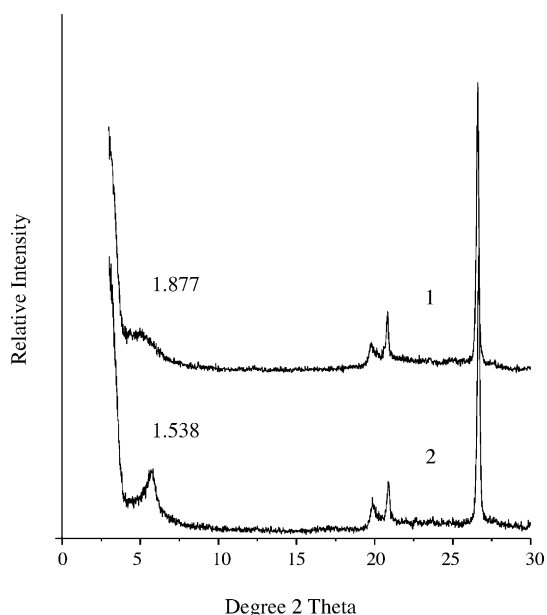


Fig. 2. XRD patterns for (1) organobentonites and (2) natural bentonite.

electrokinetic measurements are relative values for the semi-quantitative estimation of surface charge and sorption. Stability and coagulation of the colloidal dispersion are considered in terms of electrokinetic data. The zeta potential measured was shown in Table 2. It can be seen that the zeta potential of bentonite or organobentonites with dyes were decreased greatly by microwave irradiation, e.g. the zeta potential of bentonite decreased from -12.6 to -4.11 mV, the zeta potential of organobentonites with ABB decreased from -7.58 to -1.88 mV. The diminishing of the zeta potential with microwave irradiation can be partially accounted for by decreasing ionic strength of suspension and compression of electrical double layer of particles in a suspension. This means that the settleability and coagulation of bentonite or organobentonites with dyes are improved greatly by MWI.

3.3. The decolorization rates of dyestuff from water

The decolorization rates of the various dyestuffs by organobentonites and natural bentonite with MWI or conventional sorption method are listed in Table 3. It indicates that (a) the decolorization rates of dyestuffs by organobentonites are obviously higher than that of by the natural bentonite; (b) comparative with conventional sorption, the decolorization rates of NDYGL, ABB and NRS-BR by organobentonites with MWI are

Table 2
The zeta potential of bentonite or organobentonite with dyestuffs by microwave irradiation (mV)

Clay type	Additives	pH	No microwave	Microwave
Bentonite	None	9.3	-12.6	-4.11
Organobentonite	None	7.9	-3.52	-1.72
Organobentonite	NRS-BR	6.6	-5.68	-1.88
Organobentonite	ABB	6.4	-7.58	-1.35
Organobentonite	NDYGL	6.0	-6.31	-3.62

Table 3

The decolorization rates of dyestuffs onto organobentonite and natural bentonite from water by MWI and conventional sorption (%)

Dye	Organobentonite		Bentonite	
	Convention	MWI	Convention	MWI
NRS-BR	79.5	97.5	7.82	8.36
NDYGL	26.5	98.3	3.81	17.6
ABB	66.3	100	62.5	78.1

increased 71.8%, 52.4% and 18.0%, respectively, which due to the electrical double layer are compressed partially and the zeta potentials of particles are diminished by microwave irradiation (Table 2).

4. Discussions

4.1. Effects of microwave on the settling of organobentonite

Relationship of the removal rates of organobentonites from solution with the settling time was shown in Fig. 3, which indicated that solid removal rates of organobentonites from solution are increased greatly by MWI. The settling time for 50% solid removal rates of organobentonites with and without MWI are less, 5 and 20 min, respectively. The observed flocculation behavior of particles can be attributed to microwave-thermal and electromagnetic field. It indicates that the settleability and coagulation of organobentonites can be improved greatly by MWI, which is valuable for separating powered organobentonites particles from treated water.

4.2. Effects of microwave on sorption of dyestuffs onto organobentonite from water

Fig. 4 shows the sorption isotherms of three kinds of dyes onto organobentonites and natural bentonite, which drawn for

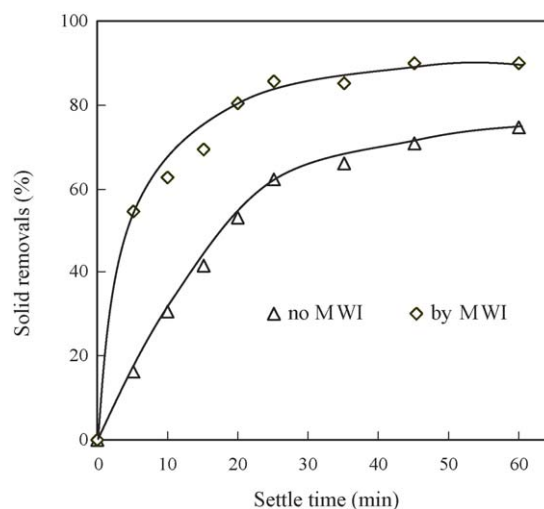


Fig. 3. The relationship between the removal rates of organobentonite from solution and the settling time.

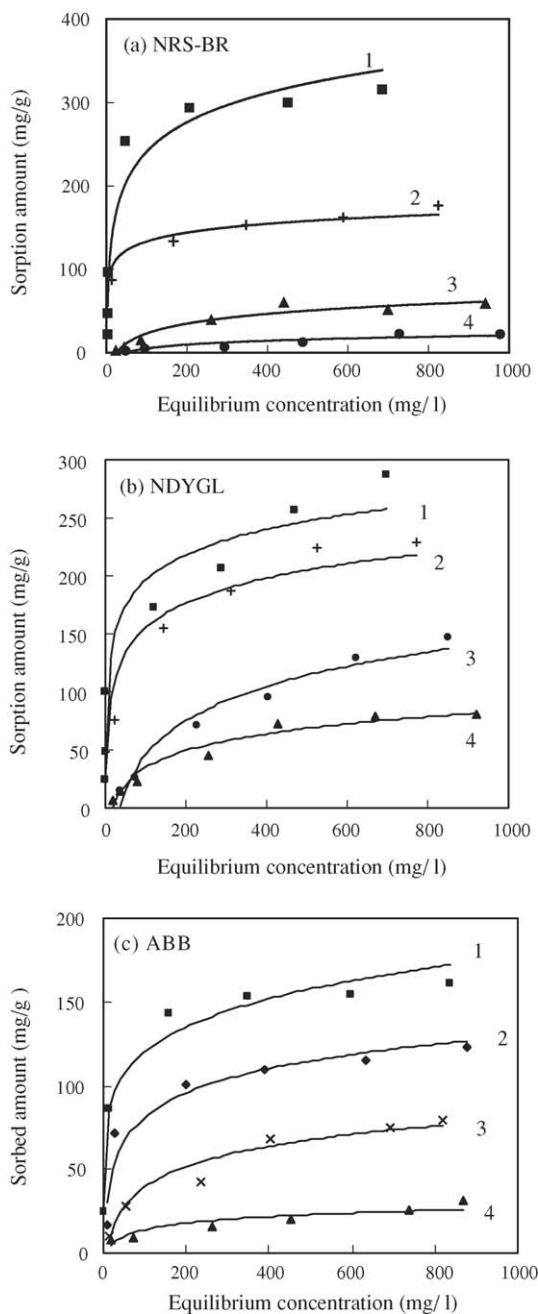


Fig. 4. Sorption isotherms of NRS-BR (a), NDYGL (b) and ABB (c) by (1) organobentonite with MWI, (2) organobentonite with convention, (3) bentonite with MWI and (4) bentonite with convention.

the samples namely natural, irritated by MWI at 25 °C and pH 6.0–6.6. These sorption isotherms were fit to the Langmuir model (Table 4). All correlation coefficient r^2 values for the Langmuir isotherm model were larger than 0.92. It indicates that the saturated sorption capacity of three kinds of dyes onto organobentonites were higher than that of the natural bentonite. The saturated sorption capacity of NRS-BR, NDYGL and ABB onto organobentonites was increased from 175.4 to 322.6, 238.1 to 277.8 and 123.5 to 217.4 mg/g, respectively.

Table 4

The Langmuir parameters for sorption of dyestuffs onto organobentonite by MWI and conventional sorption

Dye	Convention			MWI		
	Q_m (mg/g)	K_d (l/g)	r^2	Q_m (mg/g)	K_d (l/g)	r^2
NRS-BR	175.4	0.0369	0.9590	322.6	0.0475	0.9359
NDYGL	238.1	0.0202	0.9213	277.8	0.0315	0.9528
ABB	123.5	0.0330	0.9391	217.4	0.0193	0.9932

To demonstrate the effects of microwave enhanced-sorption of dyestuffs onto organobentonites, the parameter R_m is introduced with:

$$R_m = \frac{Q_M}{Q_C} \tag{2}$$

where Q_M and Q_C are the total sorption amount of dyestuffs onto organobentonites with MWI and conventional sorption method, respectively. R_m is microwave enhanced-sorption parameter. Fig. 5 shows the relationship between the parameter R_m values and the equilibrium concentration of the dyestuffs. The value of the parameter R_m is higher than 1, indicating that the enhanced-sorption by MWI. The value of the parameter R_m increase in the following order: NRS-BR > ABB > NDYGL, which corresponds with their aqueous solubility (Table 1). The value of the parameter R_m of dyestuff increases with increasing of aqueous solubility of dyestuff. The higher aqueous solubility of dyestuff is, the higher value of the microwave enhanced-sorption parameter R_m is.

The Langmuir sorption isotherm was examined with dyes under investigation according to the following equation [30–32]:

$$\frac{C_e}{Q} = \frac{1}{K_d Q_m} + \frac{C_e}{Q_m} \tag{3}$$

where Q_m is a constant related to maximum amount of sorption solute (mg/g), K_d is the sorption intensity or the Langmuir coefficient (l/g), Q is the amount sorption on solid at equilibrium (mg/g) and C_e is the equilibrium solution concentration (mg/l).

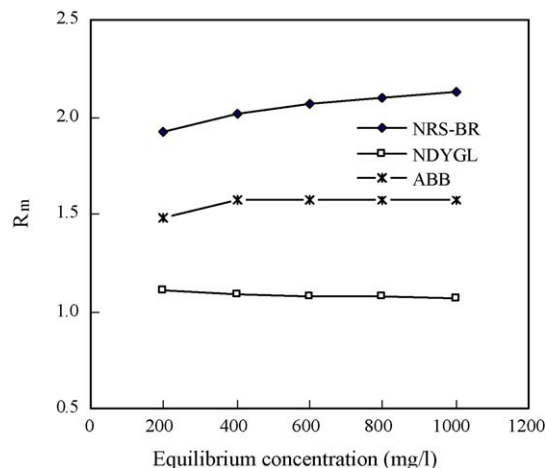


Fig. 5. The relationship between the R_m values and the equilibrium concentration of the dyestuffs.

The data obtained are well described by the Langmuir sorption isotherm when plotted according to Eq. (3). The values of Q_m and K_d (Table 4) are determined from the intercept and the slope using non-linear least-squares regression method.

4.3. Mechanisms of microwave enhanced-sorption

The sorption properties of organobentonite were depended greatly on the characteristics of the exchange organic cations. Bentonite modified with small quaternary ammonium cations exhibit mainly adsorption, i.e. non-linear isotherms, strong solute uptake and competitive. Bentonite modified with quaternary ammonium cations of large alkyl groups display mainly partition, i.e. linear isotherms, lower solute uptake and non-competitive [7,17,18,20]. The differences between the two groups of organobentonites were attributed to different sorption mechanisms. So both adsorption and partition should occur to sorption of organic compounds to a dual-cation organobentonites containing short- and long-alkyl groups.

Microwave enhanced-sorption strongly depends on the microwave-, thermal and selectivity [23–26]. The temperature of mixed solution of dyestuffs and organobentonites rises while using microwave irradiation, the aqueous solubility of dyestuffs increase, and the enthalpy of system increase and the activation energy of reaction system reduce. The polar organic molecules will be rearranged by the electromagnetic field, the molecular movement will be ordering. This accelerates the reaction velocity of sorption and the mass transfer, improves partition and result in increasing the sorption capacity of dyestuffs onto organobentonites, e.g. the sorption capacity of NRS-BR onto organobentonites increases from 175.4 to 322.6 mg/g. Moreover, the zeta potentials of particles are reduced and the electrical double layer are compressed partially by microwave, e.g. the zeta potential of bentonite decreased from -12.6 to -4.11 mV, the zeta potential of organobentonite with ABB decreased from -7.58 to -1.88 mV, which is benefit to the settling and sorption of dyestuffs onto organobentonites from water.

5. Conclusions

Comparative with the conventional sorption, microwave enhanced-sorption improved significantly the decolorization rates of dyestuffs by organobentonites from water. The value of the microwave enhanced-sorption parameter R_m increases in the following order: NRS-BR > ABB > NDYGL, corresponding with their aqueous solubility. The zeta potential of particles is decreased and the electrical double layer is compressed partially by MWI, which promoted both the sorption of dyestuffs onto organobentonites and the separation of the adsorbents from water. The high decolorization rates of the various dyestuffs by organobentonites with MWI, good settleability and coagulation may have important potential application in dyestuff wastewater treatment.

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