Performance assessment of adsorbents based on natural bentonite for removal of cationic dye

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Abstract
The development of safe and cost-effective methods for the treatment of dye polluted wastewater has been a great concern. Herein, Adsorption of methylene blue from aqueous solutions onto modified clay has been investigated. Three adsorbants were prepared through the treatment of a natural Algerian bentonite (NAB) by sodium (Na-Clay), calcium (Ca-Clay), and sulfuric acid solution (Hs-Clay), respectively. The accomplished materials was performed by X-ray diffraction and Fourier transforminfrared (FTIR).
The effects of differents process parameters such as contact time, pH of dye solution, initial concentration of dye, and adsorbent amount on the removal efficiency of methylene blue (MB) from aqueous solution were evaluated by batch experiment. The maximum adsorption capacity of 220.28 mg/g reached by Na-Clay. Furthermore, all the prepared adsorbents also displayed a prominent efficiency in removal of cationic dyes.
The findings reveal the feasibility of facile clay modification to be used as a potential and rapid low cost adsorbent for removal of cationic dyes from wastewater.
Keywords: clay, water pollution, adsorption, methylene blue, isotherms
Kulcsszavak: agyag, vízszennyezés, adszorpció, metilénkék, izotermák

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1. Introduction

Water pollution has become a serious global problem due to rapid industrialization and various human activities. Dyes effluents, originating from dyeing, textile, leather, paper and other related industries, are one of the most severe environmental pollutants because of their high toxicity to plants [1], aquatic life [2–4] and human beings [5–7]. Consequently, it is of significance to treat colored wastewater prior to their discharge into natural environment. For this reason, many attempts such as membrane separation, flocculation precipitation, oxidation, photocatalytic, degradation, microbial degradation, and adsorption have been studied for the removal of dyes from wastewater [8]. Among them, adsorption has been widely regarded as one of the most efficient treatments for dyes in wastewater due to its cost effective operation, simple and diverse design, and high efficiency [9–10].

Number of natural adsorbents have been used for the removal of pollutants from water [11]. Utilization of clays as adsorbents could bring great economical and environmental benefits to wastewater industries compared to synthesized materials [12]. Previous works reported that the utilization of clays as adsorbents was efficient for the removal of dyes [13; 15].

In order to improve the adsorption properties of raw bentonite, the modification of mineral clay was attractive for researchers, numerous studies have investigated MB removal from aqueous solution, using clay modified with dodecyl sulfobetaine surfactant [15] poly(acrylic acid-co-2-acrylamido-2-methylpropanesulfonic acid) hydrogel nanocomposite [16], Fe_3O_4 [17]. Another potential candidate for a MB sorbent is sodium or calcium-exchanged and acid-activated bentonite, where their preparation is relatively inexpensive and easy compared with that of other modifications of bentonite which can cause secondary pollution due to the leaching of modifying agents.

The main objective of this paper was to obtain several low-cost and very efficient adsorbents based on local clay which is a bentonite, a clay mineral of the smectite group having silica tetrahedral sheets layered between alumina octahedral sheets. The adsorbent materials realized were characterized and used in investigated to remove methylene blue from synthetic wastewater. The effects of initial pH value, adsorbent dosage, initial MB concentration, and contact time were checked. Furthermore, adsorption isotherms were investigated.

2. Experimental

2.1 Materials and methods

All the reagents were of analytical grade and used as received. A stock solution of dye having a concentration of 1000 mg/L was prepared by dissolving appropriate amount of MB dye powder in 1 L deionized water and then it was diluted to the desired concentration of each experiment. The molecular formula of MB dye is $C_{16}H_{18}ClN_3S$ with the molecular weight of 319.851 g/mol.

Our supports have been characterized by X-ray diffraction and The Fourier transform infrared (FTIR). A Phillips Analytical X-ray spectrometer (PW 1830) using CuKα radiation was used to characterize the adsorbents. FTIR spectra of the adsorbents were taken with a Perkin Elmer 1600 Spectrometer (range 4000–400 cm^{-1}). The UV/VIS (V-670) spectrophotometer was used to measure the concentration of MB dye in solution at wave length of λ_{max} = 665 nm. The pH solution was adjusted using reagent grade HCl and NaOH.

2.2 Adsorbents

Natural bentonite (NAB) from Maghnia deposit in west of Algeria, was used without any chemical pretreatment to produce three modified clay adsorbents. Na-Clay and Ca-Clay were obtained by treating the NAB sample for four hours with a solution of NaCl (0.5 M) and a solution of $CaCl_2$ (0.5 M), respectively. The mixtures were filtered, washed with distilled water until Cl^- ions were not detected by the silver nitrate test and then air-dried at room temperature. Afterwards, NAB was treated under reflux for two hours with an aqueous H_2SO_4 solution (10 wt. %). The suspension obtained was filtered and the solid resulting, named (Hs-Clay), was washed with distilled water until it was free of SO_4^{2-} ($BaCl_2$ test) and then air-dried at room temperature.

2.3 Adsorption experiments

All the adsorption experiments were conducted in triplicate under the same conditions. The effect of various parameters on the adsorption behaviour of the adsorbents (NAB, Na-Clay, Ca-Clay and Hs-Clay) were studied. The experimental variables examined were contact time, bentonite concentration in suspension, pH of suspension and MB concentration. Suspensions of solid adsorbents and MB adsorbate solutions were obtained by contacting 0.1 g of dry NAB, Na-Clay, Ca-Clay and HS-Clay solid adsorbents with 100 mL of 80 mg/L of MB solutions separately in sealed Erlenmeyer flasks. The mixtures obtained were magnetically stirred at room temperature at different time intervals (0–120 min). After that, the flasks were centrifuged and the concentration of MB in aqueous solution was determined by UV–VIS spectrophotometer. The effect of pH solution on MB adsorption was investigated in a pH range between 3 and 10. In these cases, the pH value of the solution was adjusted to the desired value by adding HCl or NaOH as required. Batch sorption experiments were also conducted to reveal the effect of adsorbent the amount on the removal of MB from aqueous solution. The effect of adsorbent dose was varied from 0.25 g up to 4.0 g. To establish the adsorption isotherms, this study was carried out with different initial MB concentrations varying from 5 mg/L to 280 mg/L. The adsorbent MB uptake, per unit mass of clay at time t, q_t (mg/g) was evaluated by the following equations:

$$q_t = (C_0 - C_t)\frac{V}{m} \quad (1)$$

$$q_e = (C_0 - C_e)\frac{V}{m} \quad (2)$$

Where q_t and q_e (mg/g) are adsorption amount of the dye at time t and equilibrium, C_0, C_t and (mg L^{-1}) are the concentration of MB solution at initial, time t and equilibrium, respectively, V (L) is the volume of MB solution and m (g) corresponds to dosage of adsorbent.

3. Results and discussion

3.1 Characterizations

Fig. 1 presents the X-ray diffraction patterns of natural bentonite (NAB), Na-Clay, Ca-Clay and Hs-Clay samples. The X-ray diffraction pattern of NAB shows many diffraction peaks from montmorillonite, which is characterized by four peaks: one located at 15.61 Å (d_{001}) and the other three at 4.49 Å (d_{020}), 3.79 Å (d_{004}), and 1.50 Å (d_{060}). Some peaks were identified as impurities (Quartz, calcite, cristabolite, hematite and dolomite). The results relating to the identification of the minerals present in NAB are summarized in the *Table 1*.

Clay, Basal spacing (Å)	Impurities, Basal spacing d (Å)				
Montmorillonite	Quartz	Cristabolite	Dolomite	Calcite	Hematite
15.61 (001)	3.36	3.23	2.22	3.04	2.71
4.49 (020)	1.54			2.29	1.87
3,79 (004)	1.38			1.91	1.60
2.56 (200)					
1.50 (060)					
1.69 (009)					

Table 1 Intereticular distances of different NAB minerals (Å) according to X-ray diffraction analysis
1. táblázat Különböző NAB ásványok interetikuláris távolságai (Å) röntgendiffrakciós elemzés szerint

Compared with NAB clay, as shown in *Fig. 1*, the basal spacing (d_{001}) of Na-Clay decreases from 15.61 Å to 12.91 Å as a consequence of the ion exchange process. Similar observations have also been reported by other authors [18]. In contrast, the basal spacing d_{001} of Ca-Clay and Hs-Clay expanded to 15.75 Å and 16.19 Å, respectively. Indicating that ion exchange did take place.

The FTIR spectra of samples NAB, Na-Clay, Ca-Clay and Hs-Clay are depicted in Fig. 2. The absorption band observed at 3510 cm^{-1} is attributed to hydroxyl group vibrations of Mg–OH–Al, Fe–OH–Al and Al–OH–Al units in the octahedral layer, typical of montmorillonite [19, 20]. The very broad vibration band at 3388 cm^{-1} is due to the O–H stretching vibration of the interlayer silanol (Si–OH) groups and also to the HO–H vibration of the interlayer water adsorbed silica surface involved in hydrogen bonding linkages. The absorption band at 1632 cm^{-1} is attributed to the vibration of physisorbed water molecules. The band near 1100 cm^{-1} is attributed to stretching vibration of the Si–O–Si groups of the tetrahedral layer. The broad band at 977 cm^{-1} is related to the stretching vibrations of the Si–O groups. It is observed that the samples display all the absorption bands of the parent NAB material, indicating that the layered structure is preserved after modification.

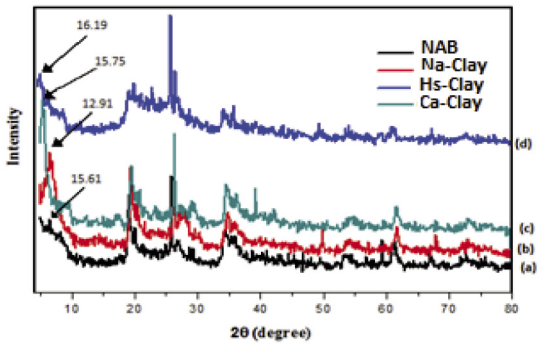

Fig. 1 X-ray diffraction patterns of NAB (a), Na-Clay (b), Ca-Clay (c), and Hs-Clay (d) samples
1. ábra NAB (a), Na-agyag (b), Ca-agyag (c) és Hs-agyag (d) minták röntgendiffrakciós mintázata

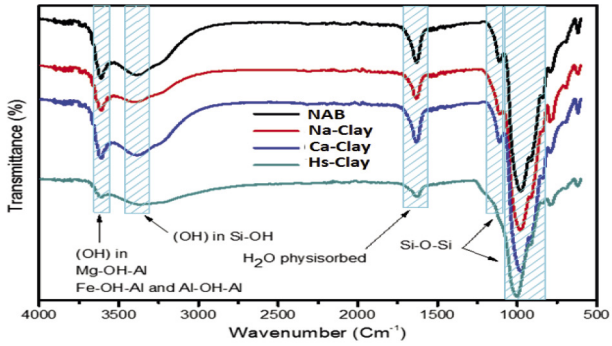

Fig. 2 FTIR spectra of NAB (a), Na-Clay (b), Ca-Clay (c), and Hs-Clay (d) samples
2. ábra NAB (a), Na-agyag (b), Ca-agyag (c) és Hs-agyag (d) minták FTIR spektruma

3.2 Adsorption

3.2.1 Effet of contact time

It is known that the performance of dye removal from aqueous solution is significantly affected by contact time. As shown in *Fig. 3*, it was apparent that the adsorption capacity rapidly increased at beginning and then gradually slowed down until equilibrium. This may be ascribed to the higher MB concentration and abundant free adsorption sites available at initial adsorption phase [22].

It is noted that a contact time of only 10 min is sufficient to reach equilibrium when Na-Clay and Ca-Clay adsorbents are used, while a contact time of 40 min is necessary in the case of Hs-Clay solid.

3.2.2 Effect of pH

The influence of pH solution on the removal of MB by modified bentonite was investigated to gain further insight into the adsorption process. *Fig. 4* shows the qe of NAB, Na-Clay, Ca-Clay and Hs-Clay in various pH solutions. The absorption capacity of Hs-Clay for MB was much lower than that of Na-Clay and Ca-Clay. While the qe values of Na-Clay were greater than those of Ca-Clay in the range pH 3–10. On the other hand, the qe values of adsorbents increased with the increase of pH solution, which also could be explained by the electrostatic interaction of positive charges of MB with the negative charges of clay, which were enhanced at higher pH values, leading to greater q_e values. The adsorption was not drastically affected by pH, suggesting the presence of other interactions (such as hydrophobic interaction) between MB and adsorbents Similar observations have also been reported by other authors [15]. At the low pH, excess H^+ ions have resulted in the formation of a positive charge on the adsorbent's surface, and it reduces the attractive force between the adsorbent and the cationic MB dyes [23, 24]. The maximum adsorption percentage was observed at pH=7. The same trend has been supported by the literature for MB sorption [24, 25]. Therefore, pH=7 value was selected as optimum pH for MB removal from aqueous solution.

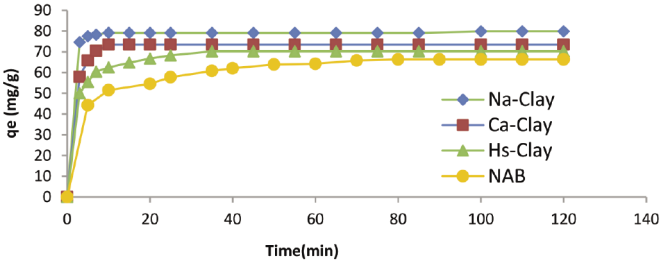

Fig. 3 Kinetics of MB removal onto NAB, Na-Clay, Ca-Clay, and Hs-Clay adsorbents
3. ábra Az MB eltávolításának kinetikája NAB, Na-agyag, Ca-agyag és Hs-agyag adszorbenseken

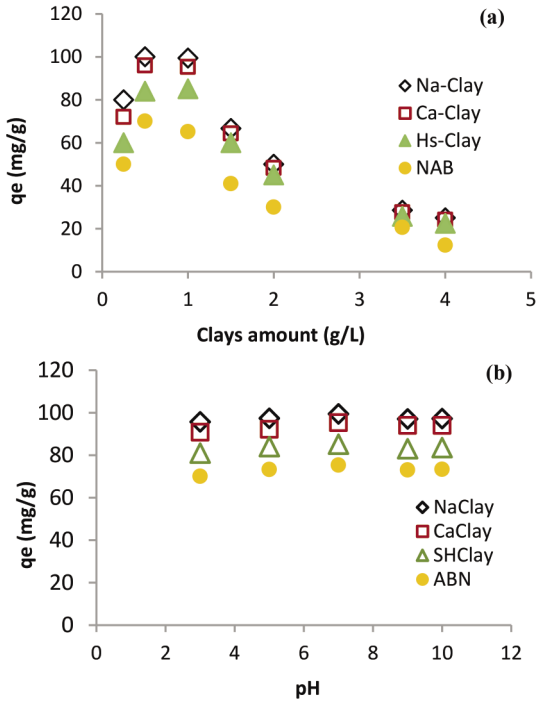

Fig. 4 Effect of Clays amount (a), and pH (b) on sorption of MB onto NAB, Na-Clay, Ca-Clay and Hs-Clay adsorbents
4. ábra Az agyagok mennyiségének (a) és a pH-értéknek (b) a hatása az MB NAB, Na-agyag, Ca-agyag és Hs-agyag adszorbenseken történő szorpciójára

3.2.3 Effect of adsorbent dose

Adsorbent doses are optimized by introducing the various amounts of NAB, Na-Clay, Ca-Clay, and Hs-Clay (0.25 g/L to 4.0 g/L) in the MB dye solution. Maximum amount of dye removal is observed at 1 g/L (fig.4). In contrast, a very slight decrease in removal percentage has been noticed with an increase in the adsorbent dosage. Initially, the removal percentage increases with the adsorbent dosages, as many

active sites are available in the adsorbents. At a high dose, It could be seen that the adsorption capacity of Na-Clay, Ca-Clay, Hs-Clay and NAB decreased with increasing adsorbent dose. These results could be due to the agglomeration of the active sites on the adsorbent surface and the heightened diffusion path length responsible for the reduction in the adsorption capacity [26].

3.2.4 Effect of dye concentration

The adsorption isotherms of MB onto NAB, Na-Clay, Ca-Clay and Hs-Clay are shown in *Fig. 5*. It appears in all cases that the adsorption loading of MB (mg/g) at equilibrium qe increases until a certain limit when increasing the initial concentration of the MB solution; afterward, it remains constant (*Fig. 5*). The adsorbed amounts are 82.35, 220.28, 217.50, 117.14 mg/g respectively for ANB, Na-Clay, Ca-Clay and Hs-Clay. The maximum adsorption capacity is observed in the case of Na-Clay adsorbent. This is attributed to the enhanced swelling property of Na-Clay [26].

Comparison of maximum sorption capacity for MB of different adsorbents

Adsorption capacities (q_m) of various adsorbents towards methylene blue dye as reported in literature were presented in *Table 2*.

A comparison between this work and other reported data from the literature shows that modified bentonite is a better adsorbent for methylene blue compared to other adsorbents. Therefore, it could be safely concluded that the Na-Clay, Ca-Clay and Hs-Clay adsorbents have a considerable potential for the removal of methylene blue from an aqueous solution.

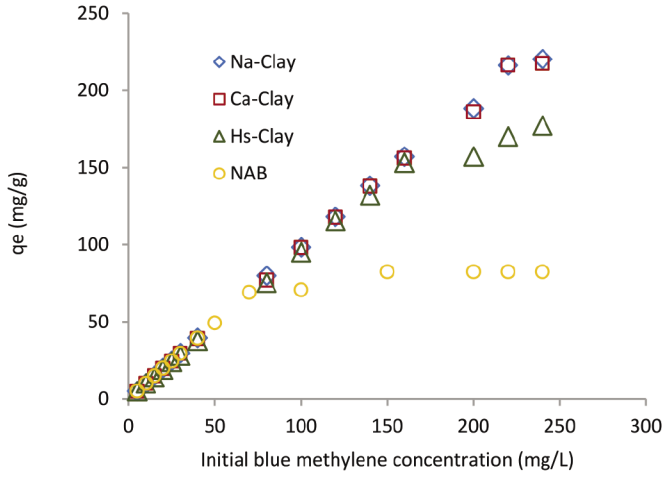

Fig. 5 Isotherms of MB adsorption onto NAB, Na-Clay, Ca-Clay and Hs-Clay adsorbents
5. ábra Az MB adszorpció izotermái NAB, Na-agyag, Ca-agyag és Hs-agyag adszorbenseken

4. Conclusions

In this study, the low-cost modified clays were prepared with different cations, and their performances of the adsorptive removal of MB from wastewater were investigated. All the

Material	pH/ Optimal pH	Contact Time/ equilibrium time (min)°	Concentration (mg/L)	Amount adsorbent	q_m (mg/g)	Ref.
Hydrochloric Acid-modified Rectorite	2-12 / 7	5–90 / 30	20–100	1–8 g/L	37.00	[2]
Black cumin seeds	1-9 / 4.8	5–120 / 20	20–100	0.1 g / 50 ml	16.85	[4]
magnetic halloysite–chitosan nanocomposites	5-11 / 8	0–1440 / 15	20–200	25 mg / 25 ml	50.37	[6]
Purified clay (Na-TIFL)	No indicated	60 / 10	80–200	1 g/L	82.00	[13]
Natural clay	3–12 / 7	0–250 / <50	9–300	1 g/L	100.00	[14]
Dodecyl sulfobetaine surfactant-modified montmorillonite	2–11 / 5	0–120 / 60	0–350	0.05 g /50 mL	254.00	[15]
Poly(AA-co-AMPS)/ montmorillonite nanocomposite hydrogel	2–13 / 10	600	10–150	20–120 mg	215.00	[16]
Fe3O4/activated montmorillonite nanocomposite	3–11 / 7.37	0-60 /	100–250	0.625–2.5 g/L	106.38	[17]
Eco-friendly polyvinyl alcohol/ carboxymethyl cellulose hydrogels reinforced with graphene oxide and bentonite	2-10 / 8	0–250	0–250	30 mg / 20mL	172.14	[21]
Acid-Treated Eucalyptus Leaves	2–10 / 8	5–360	10–300	1–10 g/L	194.34	[22]
NAB	3-10 / 7	0–120	5–280	0.25–4.0 g/L	82.35	This work
Na-Clay	3-10 / 7	0–120	5–280	0.25–4.0 g/L	220.28	This work
Ca-Clay	3-10 / 7	0–120	5–280	0.25–4.0 g/L	217.50	This work
Hs-Clay	3-10 / 7	0–120	5–280	0.25–4.0 g/L	117.14	This work

Table 2 Comparison of the maximum adsorption capacity of MB on some natural and synthetic adsorbents from aqueous solution
2. táblázat Az MB maximális adszorpciós kapacitásának összehasonlítása egyes természetes és szintetikus adszorbenseken, vizes oldatból származó MB esetében

results demonstrate that the three adsorbents can effectively remove cationic dye pollutants, represented by methylene blue. The Na-Clay showed superior adsorption performance compared to Ca-Clay and Hs-Clay. The effects of contact time, initial metal concentration, pH value, and adsorbent mass on the adsorption process were discussed. The results of this study indicate that a simple modification of clay is a reusable adsorbent for the fast and highly efficient removal of MB from aqueous solutions.

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