

Removal of Tetracycline by Adsorption on Natural Bentonite from Aqueous Solutions

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ABSTRACT

Natural bentonite's potential as a sustainable solution for removing environmental contaminants makes it a promising candidate for addressing agricultural water quality challenges. This study evaluates whether natural bentonite can adsorb tetracycline (TC) from water-based solutions, offering a viable approach to improving water quality in agricultural settings. Under conditions of pH 7, 80 mg/L of tetracycline, 1 g/100 mL of natural bentonite, a 120-minute reaction period, 200 rpm of agitation, and a temperature of 25°C, the bentonite effectively adsorbed tetracycline. Analytical techniques, including Fourier-transform infrared (FTIR) spectroscopy, the Brunauer-Emmett-Teller (BET) method, and field emission scanning electron microscopy (FESEM), were used to characterize the bentonite, revealing a surface area of 37.13 m²/g. The isotherm study showed that the sorption of tetracycline onto NB fitted the Langmuir isotherm model with coefficient of determination (R²) equal to 0.961. Our findings underscore the applicability of natural bentonite for mitigating antibiotic contamination in wastewater.

Key words: Adsorption, Bentonite, Clay, Tetracycline.



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INTRODUCTION

Environmental pollution is due to human, agricultural and industrial activities. A number of toxic organic compounds have been discovered in water resources from human waste and the discharges of pharmaceutical factories, health centers and veterinary centers as a result of their improper usage, improper disposal, and improper intake as a remedy for viral diseases including colds, flu, and the like (Ortúzar et al, 2022). These compounds pose a major risk to human health and the environment when discharged directly into water resources, causing persistence and biological accumulation. (Aftab et al, 2023). Tetracycline is an antibiotic with great activity against microorganisms and it is broad-spectrum. It is used to enhance the efficiency of nutrition and increase and stimulate the growth rate. Its antimicrobial properties make it useful in the treatment of illnesses caused by bacteria and fungi. So, it's one of the best

antibiotics out there (Ajemba, 2012). There have been findings of tetracycline in several water sources, including groundwater, surface water (such as rivers and lakes), sediment water, potable water, and wastewater. Extensive research on tetracycline removal methods in water-based systems has included sophisticated oxidation techniques. (Al-Ajeel et al, 2014) Natural bentonite is a rock or clay deposit. It forms from weathering of volcanic ash. It is mainly composed of the clay mineral montmorillonite with general formula of (Na, Ca)_{0.33}(Al, Mg)₂Si₄O₁₀(OH)₂• (H₂O)_n (up to 80 wt.%), Bentonite (montmorillonite) is white to pale green and, with exposure, darkens in time to yellow, red, or brown (Berhe et al, 2024) It has permanent negative charges that increase owing to the in the isomorphous substitution of Al⁺³ for Si⁺⁴ in the tetrahedral layer and Mg⁺² for Al⁺³ in the octahedral layer. This negative charge is balanced by the presence of exchangeable

cations (Na^+ , Ca^{2+} , etc.) in the lattice structure (Ikhtiyarova et al, 2012). The results showed that TC was partially mined, which led to the generation of by-products with less toxicity than TC and low degradability (Al-Hassan et al, 2023). Moreover, through the pH range of 2-12, the ozonation of tetracycline in aqueous solution was investigated, where it produced secondary products resistant to ozonation through aromatic rings and dimethylamine group, where the decomposition rate of tetracycline was moderate, ranging from (40-60%), for a specific period of time (Hammad Khan et al, 2010). The absorption process is one of many options for cleaning up wastewater, where it is recently gained popularity due to its many desirable qualities, such as being easy to implement, inexpensive, very effective, and producing no harmful by-products. (Algarni and Al-Mohaimeed, 2022). This technique for separation made use of a wide range of adsorbents, from simple minerals to complex nanoparticles. A variety of carbon compounds have been developed and used for TC adsorption in several recent studies. According

to the findings, chemical and physical interactions are responsible for the absorption. Clay minerals used as natural adsorbents have several characteristics. They are chemically stable, have a large specific surface area, are wet table, swell, and have a negatively charged surface. Additionally, their cation exchange capacity is high (Alshammari et al, 2020). This helps in absorbing a wide range of contaminant. Calcium montmorillonite were used to remove TC at $\text{pH}=7$ for natural and catalyzed bentonite. The high surface area of natural bentonite was due to the interference of iron oxide (hydroxide) in the spaces between the plates in montmorillonite. More recent research has shown that TC-bentonite interactions and adsorption processes in neutral and acidic environments are very common (Althobaity et al, 2022). The aim of this study is to remove residual tetracycline in aqueous solutions and to study the effect of experimental conditions such as pH, contact time, mixing rate, initial concentration of pollutants and sorbent dosage on the adsorption of tetracycline using natural bentonite.

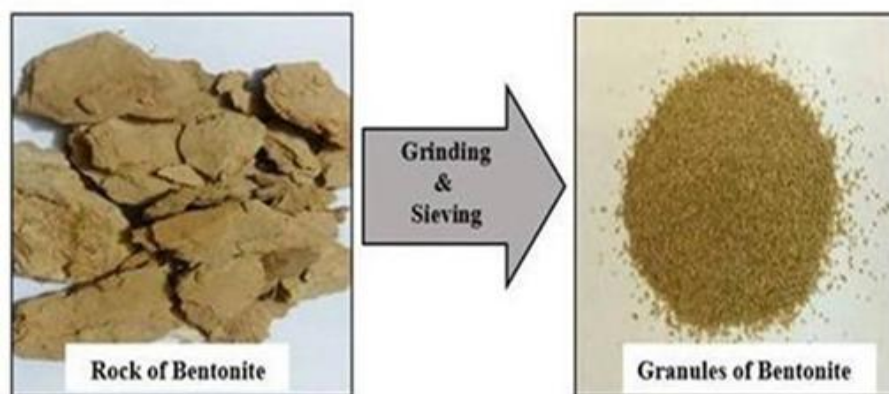


Figure 1. The natural bentonite before and after grinding

MATERIALS AND METHODS

The bentonite used in this investigation was a locally sourced, naturally occurring calcium base. Particles of rock show in figure (1) were acquired from the General Company for Geological Survey and Mining in Baghdad.

The first stage included many washes with distilled water, drying at 90°C , and crushing the rocks into various sizes. subsequently to create bentonite granules of $250\text{-}500\ \mu\text{m}$, the material was passed through sieves made of Retch, a German brand.

Table 1. displays the results of analyses conducted on the physical and chemical properties of naturally occurring bentonite by the State Company of Geological Survey and Mining.

COMPOSITION	VALUE WT (%)
Silica (SiO ₂)	57.05
Aluminum (Al ₂ O ₃)	13.5
Ferric oxide (Fe ₂ O ₃)	4.5
Magnesium oxide (MgO)	2.35
Calcium oxide (CaO)	8.56
Sodium oxide (Na ₂ O)	1.04
Loss on ignition (LOI)	13

RESULTS AND DISCUSSION

Characterization of reactive materials

Figure (2) shows the Fourier transform infrared spectra of untreated bentonite (NB) and bentonite treated after tetracycline adsorption over the 400-4000 cm⁻¹

wavelength range. An absorption peak at 3633 cm⁻¹ is associated with the (Al-O-Si) bond's bending vibration. The (Al-O-Al) and (Si-O-Si) bond stretching vibrations in bentonite are reflected by the extremely strong band in the 911–1071 cm⁻¹ range (Ao et al, 2019).

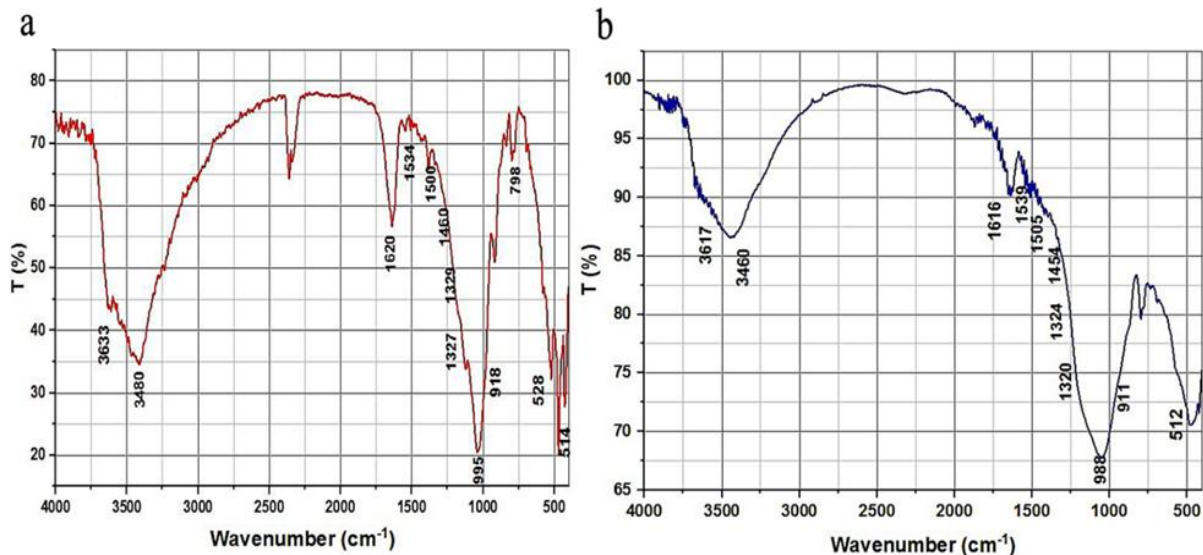


Figure 2. FTIR spectra of untreated bentonite (a) and treated bentonite (b) after TC adsorption (species) and the negative charge of the bentonite surface, the band (N-H) shifted from 1327 cm⁻¹ in NB to 1320 cm⁻¹ after adsorption TC (pH 7). Coordination complexes containing divalent cations that are very polarizable are thought to be the main mechanism by which this negative charge is stabilized. It is suggested that external complexes are forming to coordinate the water domain of the Ca⁺² cation with the TCH⁻ species, as the band (O-H) inter laminar water moved from (1620-1616) cm⁻¹. When it comes to organic chemical adsorption on bentonite, these findings are supported by other investigations.

SEM analysis: The examined the exterior morphology and composition of both untreated and treated after adsorption TC scanning electron microscopy (SEM) both prior to and

during tetracycline adsorption (Fig. 3). The original bentonite's surface morphology underwent noticeable changes, as seen. The surface morphology of NB is large, aggregated, very smooth, and fluffy, in contrast to the rough surface of NB after tetracycline adsorption (Corbett and Mullassery, 2015.). The reason for this is because when the cationic pollutant is adsorbed onto natural bentonite, some of its foliated structure is lost in the insertion process. The micrograph of the natural bentonite also revealed distorted sections, which may have resulted from the decrease of certain crystalline domains inside the particles. Following adsorption, the basal spacing of

(NB) grew from 34.65 nm to 52.44nm (Faisal and Naji, 2019).

BET analysis: It is crucial to know the precise surface area in order to estimate the capacity to absorb chemicals. The BET study relied on it to determine the precise surface area of unprocessed bentonites. In contrast to the 37.13 m²/g surface area of untreated bentonite, the BET following tetracycline adsorption on bentonite (PH 7) reduced it to 31.15 m²/g. These BET decrees are a result of the tetracycline absorbed and volume-blocking filling of natural bentonite's porosity (Faisal and Naji,2019).

Energy-dispersive X-Ray (EDX)

The test generates elemental compositional graphs for NB before and after adsorption of tetracycline as shown in Fig (4).

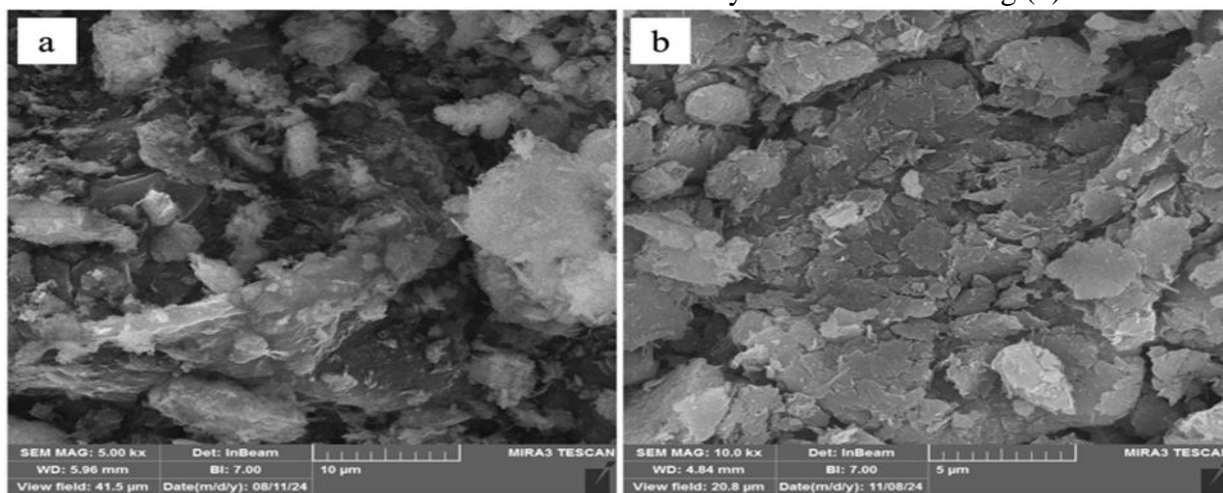


Figure 3. SEM images of Natural bentonite (before and after Tetracycline adsorption)

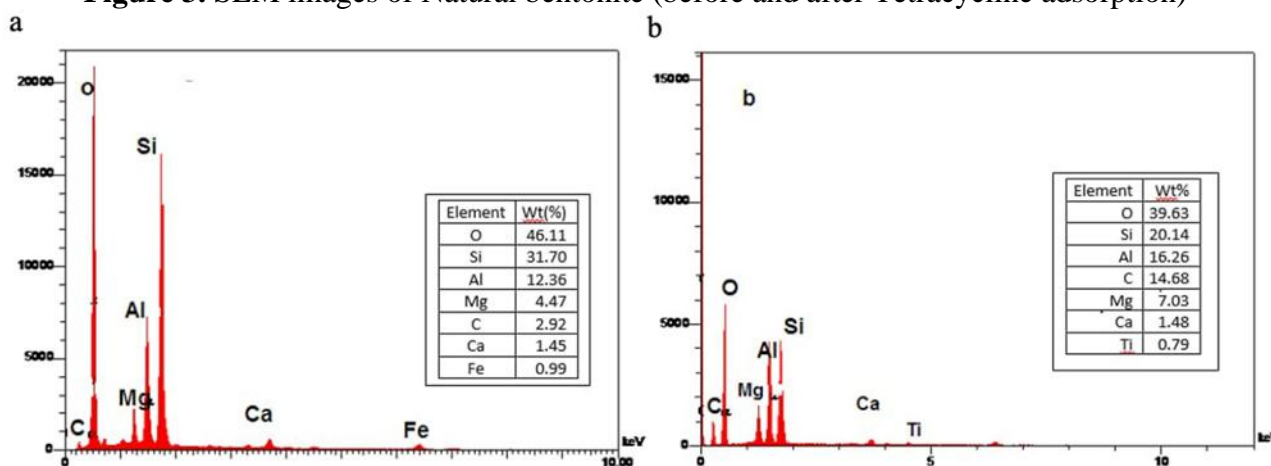


Figure 4. EDX spectra for NB before and after Tetracycline adsorption

Figure 4 (a) shows that the natural bentonite materials (NB) and Fig (b) natural bentonite after adsorption that were used to adsorption (TC). This shows that the contaminant (tetracycline) has interacted.

Batch mode sorption experiments

Using a thermostatic shaker (Edmund Buhler SM25, Germany) to mix a certain amount of NB with 100 mL of synthetic solution (with a various initial concentration), we conducted batch removal experiments to study the adsorption of tetracycline

(TC) by NB under various conditions. Batch studies were conducted with varying quantities of sorbent (0.01-1.5 g/100 mL), agitation speeds (0-250 rpm), starting pH values (ranging from 2 to 12), and contact times (ranging from 0 to 120 minutes) for practical reasons. The solution was taken from each flask in a fixed volume (100 mL). To separate the adsorbent from the water, it was filtered using filter paper of the Whatman No. 40 type. A dual beams UV-visible spectrophotometer (PG Tools, Model UV T80, England) was then used to evaluate the filtered solution at λ max in order to determine the impurity concentration. To determine the quantity of contaminants adsorbed onto natural adsorbent (NB), q_e (mg/g), the mass balance Equation 1 (Faisal et al,2022) was used.

$$q_e = \frac{(C_0 - C_e) V}{m} \quad (1)$$

This equation takes the following values: q_e , the quantity of total organic carbon (TC) adsorbed per unit mass of adsorbent (mg/g), the volume of the TC solution V (L), the initial and equilibrium concentrations of TEC (mg/L) are C_e (mg/L) respectively and m the mass of the adsorbent (mg). Using Equation 2. (Huang et al.2017), the removal efficiency (R%) of TEC/NB at time (t) was determined

$$R (\%) = \left(\frac{C_0 - C_e}{C_0} \right) * 100 \quad (2)$$

Results and discussion (Batch experiments).

Equilibrium time: The contact duration in batch tests should be set to a constant value to guarantee that the concentrations achieve equilibrium. Batch experiments were conducted at 25°C using 0.5g of natural bentonite (NB) applied to 100 mL of contaminant solution (Huang et al, 2017). The impact of contact time on Tetracycline removal is shown in Fig (5). The figure clearly shows that as the contact time rose, the proportion of Tetracycline removed from the solution increased as well. Evidently, the sorption rate was high in the beginning but decreased later on because there were less sorption sites on the adsorbent's surface, the sorption process was probably slower (Jeeva and Zuhairi,2018; Jeeva et al, 2018). Natural bentonite was able to remove around 62% percent of the tetracycline after 120min of contact. However, concentrations of this pollutant remained generally consistent during contact times longer than 2 hours. This indicates that, up until 3-4 hours later, the quantities of residual contaminants did not vary noticeably (Jin et al.2014; Lan et al, 2019).

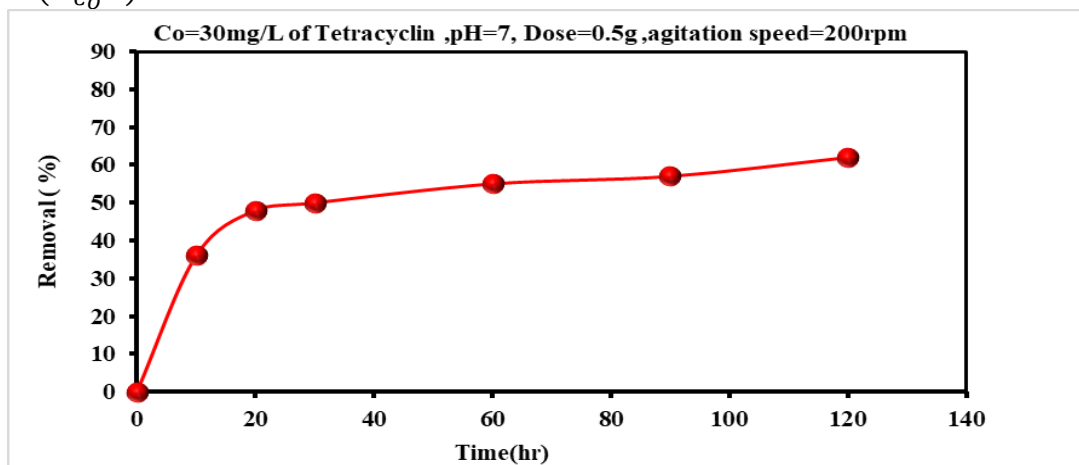


Figure 5. Removal efficiency of Tetracycline by natural bentonite (NB).

Initial pH of the solution

Tetracycline adsorption onto natural bentonite was studied in aqueous solutions with pH ranging from 2 to 12 (Fig. 6). As the pH climbed from 2 to 12, the adsorption removal of tetracycline on natural bentonite increased from 38 % to 58% respectively. Two major processes might be used to explain the tetracycline adsorption onto the adsorbent at

various pH settings. The cation-exchange process is one of them; in this case, protonated functional groups from tetracycline replaced low-valence cations in bentonite (Leyva-Ramos et al., 2021; Mahdi and Najji, 2016). Because tetracycline positive charge is most apparent in solutions with low pH. According to this happens because its carboxyl (pKa: 2.68), amine (pKa: 7.49), and phenolic

hydroxyl (pKa: 9.63) functional groups get ionized (Masood and Shahadh, 2022; Masoud, 2017). Nevertheless, when the pH of the solution rose, more tetracycline molecules were adsorbed onto the adsorbent by the

partition mechanism; this is because tetracycline becomes neutral at higher pH levels (Mohammed et al.2023; Mohammed and Salim, 2023).

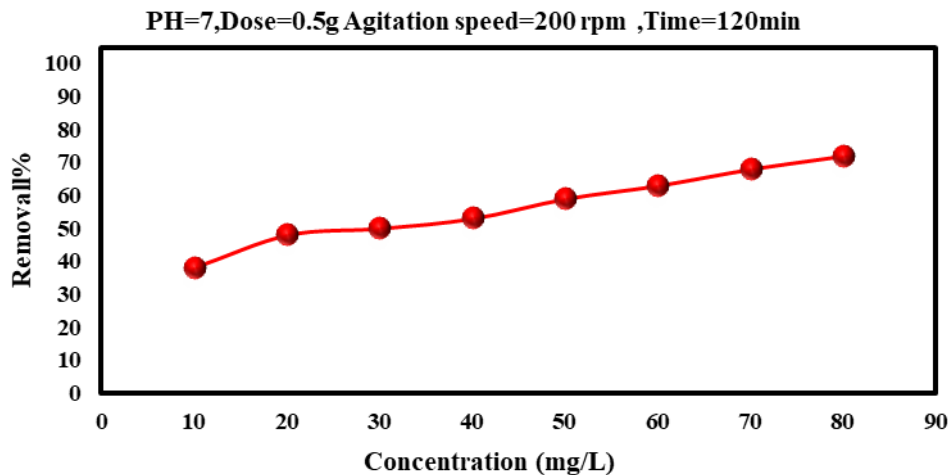


Figure 7. Impact of starting concentration on natural bentonite's ability to remove tetracycline

Natural bentonite dosage

For batch testing at 25°C, we varied the quantity of adsorbent from 0.01 to 1.5 g added to 100 mL of Tetracycline solution, while maintaining the other parameters constant: $C_0=80\text{mg/l}$, $\text{pH}=7$, shaking speed=200 rpm, and contact time=120min. This allowed us to study the dependency of Tetracycline sorption on adsorbent dose. Fig. 8 shows the relationship between the quantity of adsorbent and the effectiveness of removing tetracycline. For a constant starting concentration of contaminants, it is clear that increasing the

adsorbent dose from 0.01 to 1.5 g improves removal efficiency. Given that there are more active sites available with a larger concentration of adsorbents in the solution, this result was anticipated (Al-Shami & Al-Tamimi, 2022; Raza et al,2016). It is implied that maximum adsorption occurs at a particular dose of 1g/100ml because the amount of Tetracycline bonded to the adsorbed and the concentration of TC in the solution remain constant even after the adsorbent dosage is added.

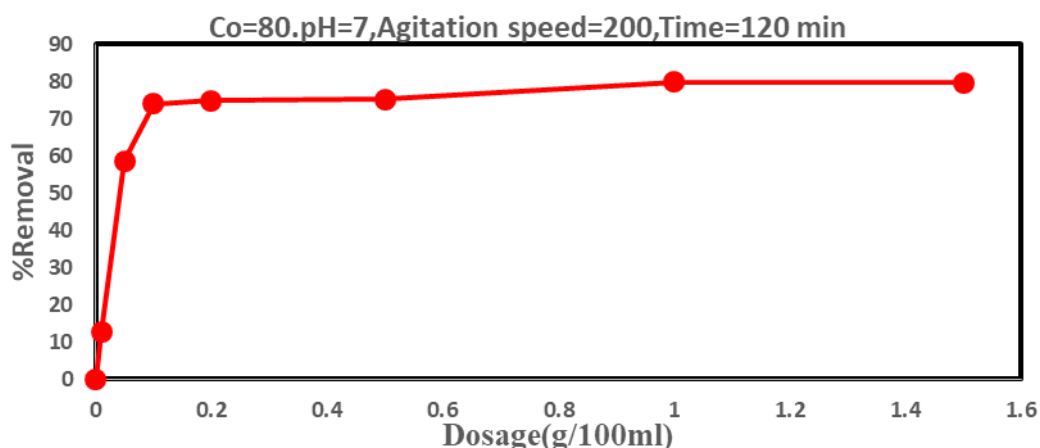


Figure 8. Effect of natural bentonite a dosage on removal efficiency of Tetracycline

Agitation speed: We kept all other parameters set to the optimal values found in the preceding phases and varied the agitation speed from 0 rpm (no shaking) to 250 rpm to

see how it affected the tetracycline removal efficiency. Fig. 9 indicates that natural bentonite removed about 75% of the tetracycline prior to shaking, and that the

absorption of tetracycline rises as the shaking rate increases. As the agitation speed was raised from zero to 200 rpm. At this point, almost 74.5% of the Tetracycline was removed by the natural bentonite (Tiwari et al.2019; Wang et al,2019). One possible explanation for these findings is that raising the agitation speed enhances the diffusion of Tetracycline towards the adsorbent surface. Sorbate ions are more effectively transferred to the sorbent sites when Tetracycline in solution makes appropriate contact with the binding sites. Fig. 9 further demonstrates that the optimal agitation speed for reaching equilibrium was

200 rpm. This rate of Tetracycline uptake should guarantee that all sites are accessible for amoxicillin uptake, which means that greater uptake rates may be achievable (Wei et al,2023). Nevertheless, the binding capacity was much enhanced and the process moved along quickly due to the increased availability of functional groups on the surface of the adsorbent. These groups are necessary for the interaction between the adsorbent and Tetracycline. Thus, the equilibrium time is a crucial characteristic for a cost-effective system to remediate polluted water.

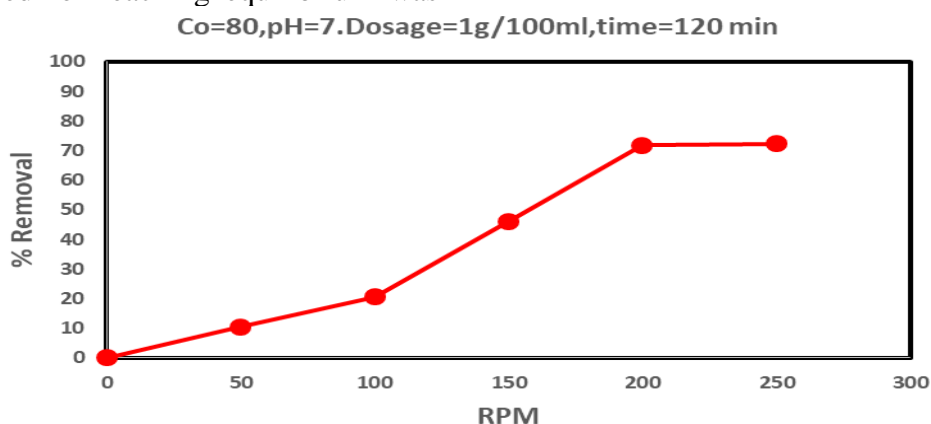


Figure 9. Speed of agitation affects the efficacy of natural bentonite in removing tetracycline

Sorption isotherms Sorption isotherms are mathematical models that show the distribution of the sorbate species among the liquid and the sorbent, based on a set of assumptions about the heterogeneity or homogeneity of the sorbents, the type of coverage, and the probability of interaction between the sorbate species (Wilkinson, et al 2017). These isotherms determine the connection between the contaminant absorption per unit mass of sorbent, q_e , and the equilibrium sorbate concentration in the bulk solution phase, C_e as a result, we used Excel 2010 to find the empirical coefficients (Table 2) for every model by analyzing the linear plot's slope and intercept. When it came to Tetracycline sorption on natural bentonite,

the Langmuir isotherm model clearly gave the greatest correlation (Xu et al, 2021). As a result, we will adapt Tetracycline on natural bentonite in continuous mode and utilize the Langmuir isotherm model to explain the contaminant sorption in the partial differential equation that governs the transport of a solute undergoing equilibrium sorption via natural bentonite. Recalculating the sorbed amounts using the equilibrium level quantities and Langmuir parameters to confirm the accuracy of the Langmuir model is both intriguing and essential (Fig.10). The tetracycline Langmuir isotherm model, on the other hand, had solid fitness between predicted and experimental values (Yang et al, 2020; Yang et al, 2019).

Table 2. Sorption isotherm constants with coefficients of determination Tetracycline by natural bentonite

Model	Parameters	Natural Bentonite
Langmuir	q_m (mg/g)	33.898
	b (l/mg)	0.042
	R^2	0.961
Freundlich	K_F (mg/mg) (l/mg) ^{1/n}	6.902
	N	2.803

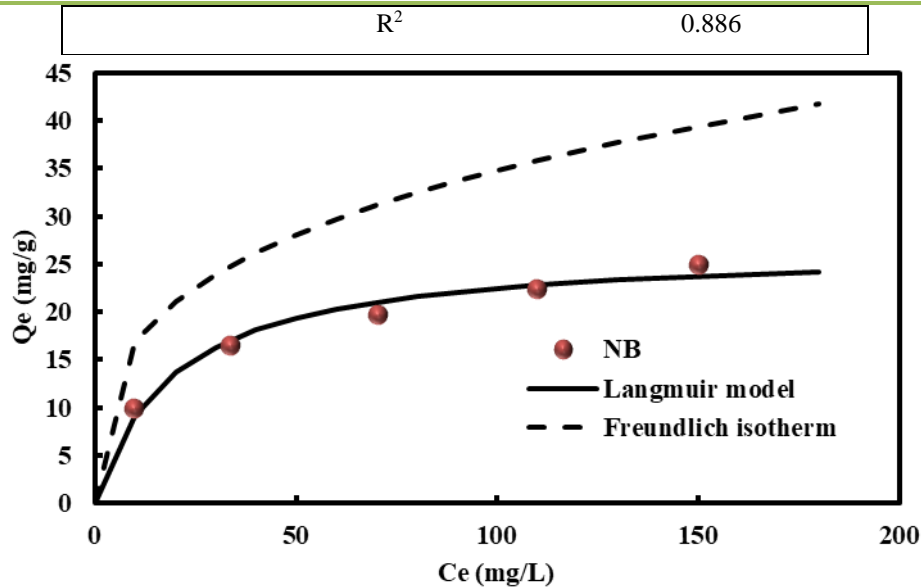


Figure 10. The Langmuir isotherm model's q_e values for tetracycline by natural bentonite are compared to the experimental data

CONCLUSION

This study proves that natural bentonite is a very efficient and environmentally friendly decontamination for water contaminated with tetracycline. This ubiquitous clay has demonstrated that it can take on the formidable challenge of reducing antibiotic runoff from agricultural areas at an optimum pH and at ambient temperature. A surface area and physical structure that is confirmed by the FTIR imaging and FESEM, explains why; the clay environment is ideal, with its "sticky" quality, for TC to adhere. The data is well correlated with Langmuir isotherm model, indicating that the tetracycline is able to form a consistent and predictable layer on the bentonite surface. In summary, these findings underscore the potential use of natural bentonite as a powerful and cost-effective solution for wastewater purification and the overall environmental well-being.

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CONFLICT OF INTEREST

The authors declare that there are no conflicts of interest. No source of funding outside of this research or financial or personal relationships that influenced the work reported in this paper were identified.

AUTHOR/S DECLARATION

This is an original manuscript which is not published or under review in any other journal. All the authors contributed and approved the final version, and cited all the sources and confirmed that all the results are accurate.

AUTHOR'S CONTRIBUTION STATEMENT

All the authors developed this study and prepared the manuscript.

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إزالة النترات سايكلين عن طريق الامتزاز على البنتونيت الطبيعي من المحاليل المائية

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المستخلص

إن الإمكانيات التي يتمتع بها البنتونيت الطبيعي كحل مستدام لإزالة الملوثات البيئية تجعله مرشحاً واعداً لمعالجة تحديات جودة المياه الزراعية. تهدف هذه الدراسة إلى تقييم قدرة البنتونيت الطبيعي على امتزاز النترات سايكلين (TC) من المحاليل المائية، مما يوفر نهجاً فعالاً لتحسين جودة المياه في الاستخدامات الزراعية. تحت ظروف تتضمن درجة حموضة 7، وتركيز نترات سايكلين يبلغ 80 ملغ/لتر، و 1 غرام/100 مل من البنتونيت الطبيعي، وفترة تفاعل لمدة 120 دقيقة، وسرعة تحريك تبلغ 200 دورة في الدقيقة، ودرجة حرارة تبلغ 25 درجة مئوية، أظهر البنتونيت فعالية كبيرة في امتزاز النترات سايكلين. تم استخدام تقنيات تحليلية، منها التحليل الطيفي بالأشعة تحت الحمراء (FTIR)، وطريقة برونور-إيميت-تيلر (BET)، والمجهر الإلكتروني الماسح ذو الانبعاث الميداني (FESEM)، لتوصيف البنتونيت، حيث أظهرت النتائج مساحة سطحية قدرها 37.13 م²/غرام. أظهرت دراسة الأيسوثرم أن امتصاص النترات سايكلين على NB زود نموذج لنكميور الأيسوثرم بمعامل تحديد R² يساوي 0.961. تؤكد النتائج التي توصلنا إليها إمكانية تطبيق البنتونيت الطبيعي للتخفيف من التلوث بالمضادات الحيوية في مياه الصرف الصحي.

الكلمات المفتاحية: الامتزاز، بنتونايت، الطين، نترات سايكلين.