Physicochemical Characterization of a Clay from Sergipe State and its Potential as an Adsorbent

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Water treatment is fundamental to removing industrial dyes, such as methylene blue, and to mitigate their severe environmental impacts. In this context, adsorption stands out as a promising solution due to its efficiency and low cost. The use of clays, especially regional ones, is particularly strategic as it utilizes a local and abundant resource to develop a sustainable, accessible treatment technology that adds value to the region's economy. Thus, this work aims to characterize a regional clay with possible application in the adsorption process. Through the analysis performed, the presence of quartz, calcite, hematite, and feldspar was verified via XRD, in addition to clay minerals such as kaolinite and montmorillonite, which are also part of its composition. The clay minerals are the main components in the adsorption process. From FTIR, it was possible to observe the presence of the 3695 cm^{-1} band, characteristic of kaolinite clays, in addition to confirming the presence of characteristic bands related to the lamellar structure of clays containing tetrahedral and octahedral layers, such as the bands at 984, 914, 796, and 691 cm^{-1}. Through TGA and DTG, the decomposition of kaolinite was noted at temperatures of 480 °C and 680 °C, along with a total mass loss of 1%. With SEM, a low agglomeration of particles was observed due to the low concentration of montmorillonite in its composition; thus, less agglomerated structures tend to have a larger surface area and more free active sites for the adsorption process. And, through BET, it was found that this clay is microporous (Dp = 1.56 nm and Vp= 0.047 cm³/g) and has a high surface area (S_BET = 36.75 m²/g), and high inter-pore area, in addition to exhibiting a type IV isotherm with H3 hysteresis, typical of materials with a lamellar structure. Furthermore, preliminary adsorption tests validated this potential, achieving a maximum removal of 89.39% of the dye using a 0.3 g mass.

Keywords: Regional Clay. Characterization. Adsorption. Blue Methylene.

Abbreviations: XRD, X-ray Diffraction. FTIR, Fourier-Transform Infrared Spectroscopy. TGA, Thermogravimetric Analysis. MEV, Scanning Electron Microscopy. BET, Brunauer, Emmett and Teller Method.

Water is an essential element for life, but only 0.01% of the Earth's water is accessible for human needs. This scarcity is worsened by pollution resulting from excessive and uncontrolled human activities in sectors such as transportation, industry, agriculture, and urban development. Industrialization, in particular, has caused environmental degradation, with many factories lacking proper wastewater treatment facilities. Among these pollutants, dyes are some of the most common industrial contaminants.

Received on 26 October 2025; revised 17 January 2026.
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J Bioeng. Tech. Health 2026;9(2):135-141

These highly toxic organic molecules are used in the textile, food processing, paper, and leather industries, with approximately 8,105 tons of synthetic dyes produced each year [1].

Therefore, treating water contaminated with organic dyes like methylene blue is necessary. Among various treatment methods, adsorption has gained popularity for removing organic pollutants because of its simplicity, speed, environmental friendliness, and low-cost adsorbents. It is important to note that the efficiency of this process depends on several factors, including the affinity of the adsorbent surface for the pollutant, pH, temperature, adsorbate concentration, and the specific surface area of the adsorbent [2].

In adsorption, a variety of materials are used to remove pollutants from water, especially dyes. Some of these include layered double hydroxides and minerals found in clays, which are considered promising adsorbents due to their high adsorption capacity, thermal stability, and surface chemical properties [3]. Clays, such as kaolinite, are naturally abundant, low-cost materials that have controlled porosity and are non-toxic [4].

Kaolinite consists of a tetrahedral silicon layer (SiO_2) and an octahedral aluminum hydroxide layer ($Al_2(OH)_6$). These layers are linked by hydrogen bonds between oxygen atoms and hydroxyl groups, preventing expansion or contraction upon contact with water [5,6]. The isomorphic substitution of Si^{4+} by Al^{3+} in the silica layer of kaolinite creates a negative charge, which serves as an active site for adsorption [4].

Research shows that kaolinite effectively removes dyes like methylene blue (MB), with notable adsorption capacities. This is due to hydrogen interactions between the oxygen in kaolinite and the nitrogen atoms of the MB dye. Therefore, the accessibility of active sites on the clay surface is essential for efficient removal of organic pollutants [4].

Regional development relies on the demand for local products [7], through strategies that highlight a region's resources. For example, Geographical Indication (GI) links product quality to its origin, supporting the local economy [8], and this same idea applies to using regional clays to develop sustainable and affordable technologies.

In this context, finding low-cost materials for water treatment, such as clays, becomes vital, especially for dye removal through adsorption. However, understanding a material's characteristics is necessary before applying it to ensure performance. The main goal of this work is to characterize a regional clay from a municipality in Sergipe to gather the essential information for evaluating, in future studies, the true potential of this clay as an adsorbent for methylene blue dye.

Materials and Methods

Material

The material used in this study was a sample of regional clay from a municipality in the state of Sergipe.

Methods

The regional clay sample was analyzed using X-ray Diffraction (XRD) at the Department of Physics at the Federal University of Sergipe to identify its crystalline phases. The scan range was from 1° to 60°, conducted at a speed of 2°/min.Fourier-Transform Infrared Spectroscopy (FTIR) was employed to identify functional groups within the spectral range of 650 cm^{-1} to 4000 cm^{-1}, using equipment located at the Laboratory of Food Research at the Institute of Technology and Research, Tiradentes University. Thermogravimetric Analysis (TGA) and its derivative (DTG) were performed to assess mass loss as a function of temperature; these tests were carried out under a N2 atmosphere with a flow rate of 50 mL/min, at a heating rate of 10°C/min, over a temperature range of 25 to 900°C. Scanning Electron Microscopy (SEM) was used to examine the morphology at an acceleration voltage of 10 kV and a magnification of 130x.

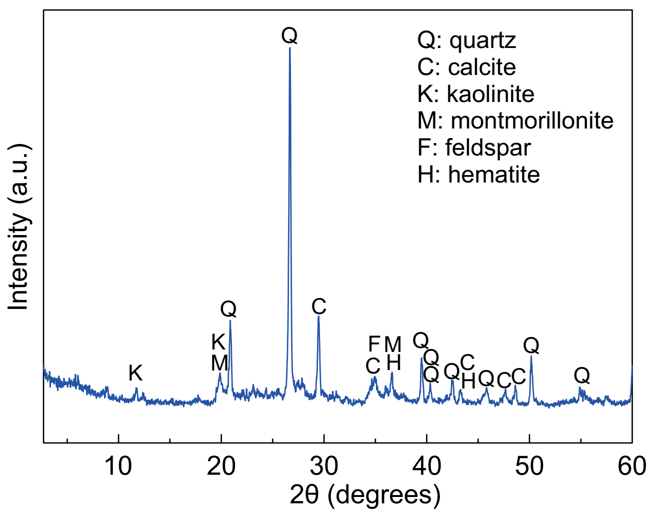
The TGA and SEM instruments are housed at the Center for Colloidal Systems Studies. N2 adsorption-desorption isotherms (BET method) determined the textural properties, with nitrogen at 77 K (- 196°C). Prior to analysis, samples were pre-treated at 200 °C under vacuum for 2 hours to remove moisture or organic residues. This equipment belongs to the Laboratory of Materials Synthesis and Chromatography at Tiradentes University. Preliminary batch adsorption tests were performed by varying the clay mass (0.1, 0.2, and 0.3 g) in 50 mL of methylene blue solution (initial concentration 5 mg/L) at 25 °C and 175 rpm. The contact time was fixed at 60 minutes, a time longer than the equilibrium time preliminarily determined to be (~20 min), thus ensuring the process reached completion. After filtration, the equilibrium concentrations (Ce) in the filtrate were determined by UV-Vis spectrophotometry (at $\lambda max \approx 664$ nm). The removal efficiency (%R) and the adsorption capacity (qe, mg/g) were also determined.

Results and Discussion

X-ray Diffraction (XRD)

The crystalline phases present in the regional clay were identified according to the Inorganic Crystal Structure Database (ICSD) and are presented in Graphic 1. The analysis revealed the presence of quartz, calcite, feldspar, and hematite, as well as the clay minerals montmorillonite and kaolinite.

Graphic 1. XRD diffractograms for regional clay.



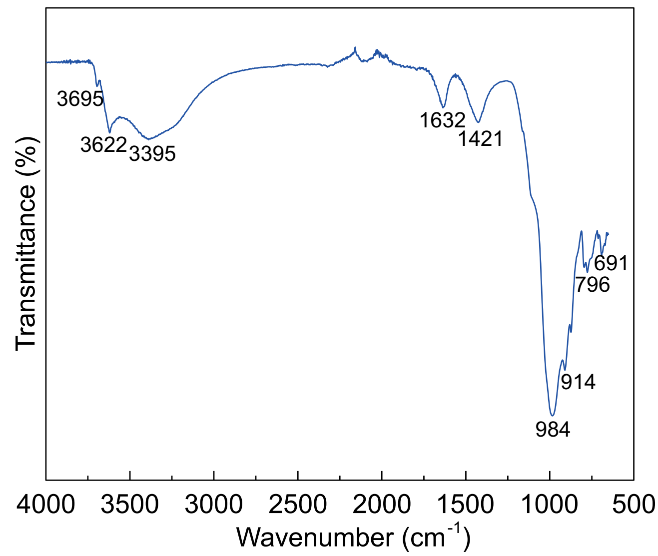
The roles of the identified minerals in adsorption vary: quartz is considered inert due to its stable crystalline structure, while feldspar can release competing alkaline and alkaline-earth cations. In contrast, hematite and calcite provide active sites that enhance adsorption [9,10]. The clay minerals, kaolinite and montmorillonite, are particularly important to the process due to their good ion exchange capacity [10].

Fourier-Transform Infrared Spectroscopy (FTIR)

The FTIR spectrum of the regional clay is shown in Graphic 2.

A band is observed at 3695 cm^{-1}, which is attributed to the stretching vibration of O-H groups present in the composition of kaolinitic clays. The bands at 3622 and 3395 cm^{-1} are related to the symmetric and asymmetric stretching of hydroxyl functional groups from water present in the clay lamellae [11].

Graphic 2. FTIR spectrum of regional clay.



The band at 1632 cm^{-1} is attributed to the typical O-H bending of constitutional water molecules adsorbed on the phyllosilicate surface. The band at 984 cm^{-1} refers to the asymmetric stretching of the internal O-Si-O and O-Al-O tetrahedra. The band at 914 cm^{-1} is attributed to the O-H bending of the carboxylic acid group, and together with the band at 796 cm^{-1}, it refers to the Al-Mg-OH stretching, confirming the presence of quartz. The band at 691 cm^{-1} is related to Si-O stretching [12,13].

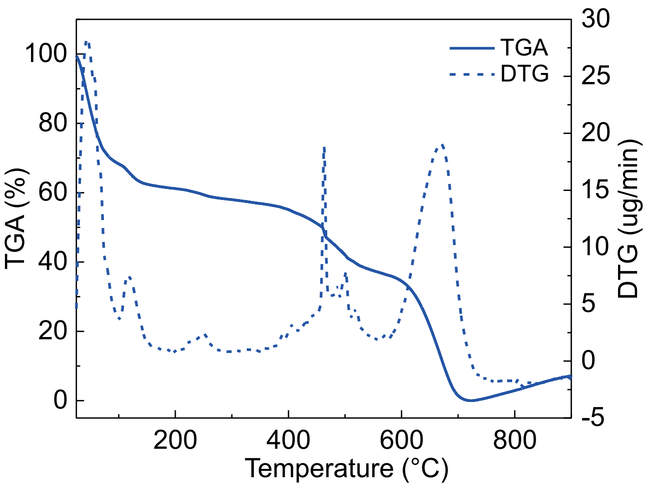
In addition to the cited bands, the presence of a band at 1421 cm^{-1} is noted, which is related to the presence of carbonate. It is worth noting that the hydroxyl groups and the negative surface charge of the silicate structures are fundamental for attracting and binding cationic contaminants, thereby enhancing the adsorption process [14].

Thermogravimetric Analysis (TGA)

The curves presented in Graphic 3 are associated with the thermogravimetric analysis (TGA) and its derivative (DTG).

The DTG analysis reveals three main stages of mass loss. The first stage, up to 150 °C, shows

Graphic 3. TGA and DTG curves of regional clay.



~38% loss attributed to physically absorbed water [15]. The second stage, occurring between 150 °C and 500 °C, corresponds to ~20% mass loss from interlayer water, and includes a distinct endothermic event at 480 °C due to the dehydroxylation of kaolinite into metakaolinite.

A third mass loss of ~41% is observed between

500 °C and 700 °C, which is related to the thermal decomposition of $CaCO_3$ into CaO and CO_2 [16]. Therefore, these results suggest that the sample contains a considerable amount of kaolinite.

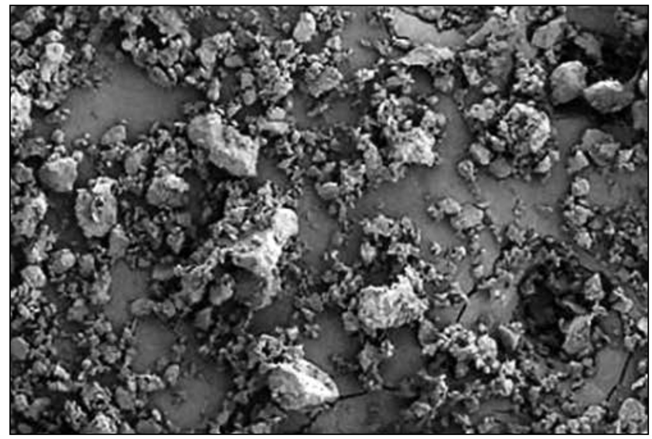
Scanning Electron Microscopy (MEV)

Figure 1 shows the particle arrangement of the analyzed regional clay.

Analyzing Figure 1, a low agglomeration of particles can be observed, which correlates with its low concentration of montmorillonite, thus being a characteristic composition of kaolinite-rich clays. This contrasts with montmorillonite clays, where higher particle agglomeration is often associated with high adsorption capacity due to a lamellar structure with swelling properties [17].

The tendency of particles, such as those in montmorillonite, to form stacked agglomerates limits access to their internal surfaces, thereby

Figure 1. SEM micrograph.



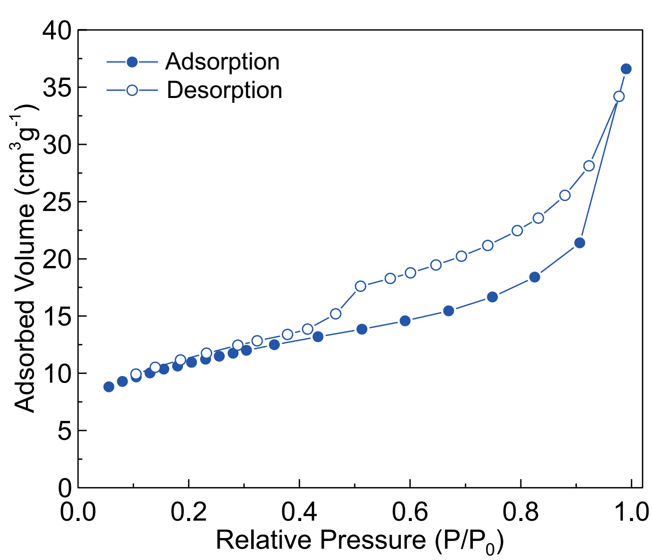
reducing the available surface area — a critical factor for an efficient adsorption process [18].

Brunauer, Emmett and Teller Method (BET)

Graphic 4 shows the adsorption and desorption isotherms for the regional clay. The isotherm is classified as type IV with H3 hysteresis, according to the 1985 IUPAC classification.

This isotherm classification is characteristic of materials with a hysteresis loop, which arises from

Graphic 4. PGraphic 4. N_2 adsorption-desorption isotherm of the regional clay.



differences between the adsorption and desorption mechanisms. In other words, N_2 molecules remain bound to the adsorbent during desorption. This H3-type hysteresis is also representative of materials with slit-shaped pores (parallel plates), a structure commonly found in clays due to their layered nature [19].

Based on the textural properties, the clay is considered microporous (< 2 nm), with a pore diameter (DP) of 1.59 nm and a pore volume (VP) of 0.047 cm^3/g. In microporous materials, the large internal surface area, combined with overlapping potential energy fields, intensifies the interaction forces between the adsorbent and the adsorbate. This effect, resulting from the small pore size, promotes a superior adsorption capacity, which is particularly effective at low concentrations compared to materials with larger pores [20].

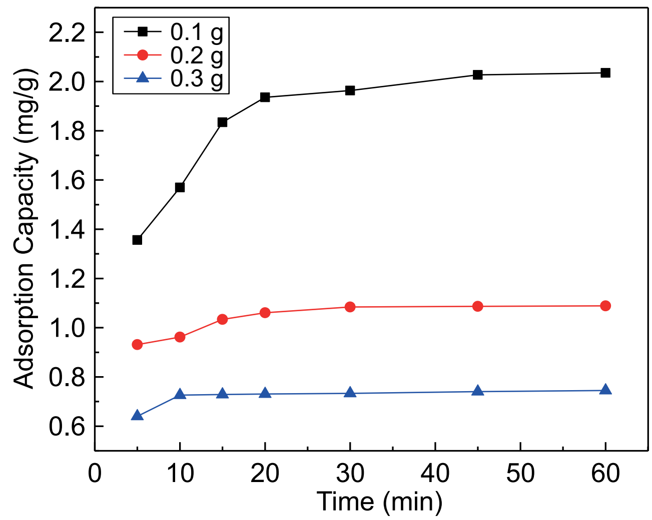
Furthermore, the specific surface area (SBET) was determined to be 36.746 m^2/g. This high surface area suggests that the material has a considerable number of active sites available for the adsorption process [21]. This result is also significantly higher than a value reported in the literature (9.51 m^2/g) [20], which is nearly four times lower than the value obtained in this work.

Preliminary Adsorption Tests

To evaluate the effect of the adsorbent mass, preliminary tests were conducted using 0.1, 0.2, and 0.3 g of clay, all at a fixed contact time of 60 minutes. This duration was adopted to ensure complete equilibrium, as preliminary tests confirmed that the slowest condition (0.1 g) reached its plateau at 45 minutes. Graphic 5 presents the resulting equilibrium adsorption capacity (qe) as a function of the adsorbent mass.

The mass assay results demonstrated the classic opposing trends. The total removal efficiency (%R) rose from 81.41% at 0.1 g to 87.11% at 0.2 g and reached a maximum of 89.39% at 0.3 g, which is attributed to the greater availability of active sites. Conversely, the adsorption capacity decreased from 2.035 mg/g at 0.1 g to 1.089 mg/g at 0.2 g

Graphic 5. Effect of contact time and adsorbent mass on the adsorption of methylene blue.



and a minimum of 0.745 mg/g at 0.3 g, indicating lower active site saturation at higher masses.

This promising performance in natura (89.39% removal and qe up to 2.035 mg/g) is notably superior to the theoretical maximum adsorption capacity of 0.907 mg/g reported by Shikuku and colleagues 2021 [23] for a natural kaolinite. This difference can be attributed to the Sergipe clay's significantly higher specific surface area (36.75 m^2/g vs. 14.616 m^2/g) and its mixed mineralogy. Furthermore, this cost-effective in natura approach is validated by Del Sordo Filho and colleagues 2021 [24] who concluded that activation did not significantly improve adsorption for Brazilian kaolinite, making the raw material the preferable cost-benefit application.

Conclusion

This work successfully characterized a regional clay from the state of Sergipe, evaluating its potential for application as an adsorbent. XRD and FTIR analyses confirmed a composition rich in clay minerals such as kaolinite and montmorillonite, in addition to accessory minerals.

The presence of hydroxyl groups, fundamental for adsorption, was evidenced. The morphology observed by SEM, with low particle agglomeration, suggests good accessibility to active sites, which was corroborated by the textural properties obtained from BET analysis. This analysis revealed a considerable specific surface area (36.75 m²/g) and a microporous structure with a type IV isotherm, typical of lamellar materials, indicating a high adsorptive potential.

This potential was validated by the preliminary adsorption tests, which demonstrated fast kinetics, equilibrium reached at 45 minutes for the limiting condition, and a high removal efficacy, achieving 89.39% of the dye using a 0.3 g mass. Therefore, it is concluded that the studied regional clay possesses promising characteristics for use as an alternative, low-cost adsorbent. For future work, isotherm studies, varying the initial dye concentration, are recommended to determine the material's maximum adsorption capacity, as well as expanding its application to the removal of other priority contaminants, such as heavy metal ions.

Acknowledgement

We thank to Tiradentes University (UNIT) and the Institute of Technology and Research (ITP) for the opportunity and infrastructure that made this work possible. My gratitude is also extended to their laboratories— the Laboratory of Food Research (LPA), the Center for Colloidal Systems Studies (NUESC), and the Laboratory of Materials Synthesis and Chromatography (LSINCROM)—as well as to the Department of Physics at the Federal University of Sergipe (UFS) for carrying out the characterizations. This study was financed in part by Coordenação de Aperfeiçoamento de Pessoal de Nível Superior [CAPES]—Finance Code 001 and Conselho Nacional de Desenvolvimento Científico e Tecnológico [CNPq].

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