

Structural and Adsorptive Properties of Sapropel-Based Sorbents: Effect of Mechanical and Thermal Activation

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ABSTRACT: This study presents the results of investigations on the preparation of highly adsorptive sorbents based on sapropel. It was established that mechanical and thermal treatments significantly influence the structure and porosity characteristics of the material. Mechanical grinding led to a marked increase in total pore volume as particle size decreased, which in turn enhanced the number of adsorption centers and overall sorption capacity. Thermal activation under controlled conditions revealed that a temperature of 350 °C provides an optimal balance between BET surface area and mechanical stability. Based on the data illustrated in Figures 1 and 2, it was confirmed that both mechanical and thermal processing are crucial steps for developing efficient sorbent materials. The resulting sapropel-based sorbents demonstrate high potential for use in industrial wastewater treatment, nutrient retention in agronomic applications, and catalytic processes.

KEYWORDS: Sapropel, sorbent, mechanical grinding, thermal activation, BET surface area, pore volume, adsorption capacity, purification technology, industrial wastewater, natural adsorbent.

1. INTRODUCTION

The preparation of sorbent materials based on natural raw materials, especially the activation and structural modification of dense organo-mineral substances like sapropel, has become one of the key directions in modern ecotechnology. Various studies have demonstrated that in order to convert sapropel into an effective sorbent, its physical and chemical structure must be purposefully modified—especially through methods such as thermal activation and mechanical grinding, which are considered highly effective.

Sapropel is a mixture of organic and mineral substances that accumulates at the bottom of water bodies and is characterized by a high content of organic matter and mineral inclusions. The presence of humic and fulvic acids, calcium, magnesium, iron and silicon oxides, as well as carbohydrates and proteins in its composition makes it a promising material for use as a sorbent. However, due to its low porosity and surface area in its natural form, the sorption capacity of raw sapropel is limited. Therefore, special treatments are required to optimize its structure for sorptive applications.

Mechanical grinding serves as the initial stage of sapropel activation, reducing particle size and increasing the specific surface area. Grinding within the 50–600 µm range directly affects the pore volume. In described studies, sapropel particles up to 600 µm in size exhibited very low total pore volume—approximately 0.03 cm³/g. However, when ground to 50 µm, this value increased up to 0.26 cm³/g, indicating an 8.6-fold growth. This reflects a significant expansion of porosity, an increase in the number of active sorption sites, and a broader spatial structure.

In addition to mechanical grinding, thermal activation plays a vital role by altering the internal organic and mineral structures of sapropel and unlocking its surface area and pore network. Experimental investigations conducted at different temperatures (250 °C–450 °C) revealed that a BET surface area of 39.49 m²/g is achieved at 350 °C, where the formation of mesopores is observed. This condition ensures that the sorbent possesses maximum adsorption activity. As the temperature increases up to 450 °C, the BET surface area reaches 45.39 m²/g, but temperatures above 400 °C may cause structural degradation. Various studies suggest that the optimal activation temperature lies in the range of 350–370 °C, as this range offers both mechanical stability and high adsorption efficiency (Ivanets et al., 2017; Bagreev et al., 2001).

The changes occurring during this process are associated with the formation of active molecular sites, decomposition of organic matter, and the preservation of the crystallinity of mineral components such as calcite, dolomite, and hematite. According to X-ray diffraction (XRD) analyses, sapropel activated at 350 °C retains well-defined crystalline phases of calcite, magnetite, and muscovite, which contribute to the material's chemical stability and

reusability. Moreover, the combined presence of mesopores and micropores enhances the speed and efficiency of the adsorption process.

Other studies have shown that such activated sapropel-based sorbents exhibit high efficiency in removing heavy metal ions such as Pb^{2+} , Cu^{2+} , Cd^{2+} , and Zn^{2+} from aqueous media. For instance, Visentsev and co-authors (2018), comparing composite sorbents based on bentonite and hydroxyapatite, noted that sapropel's high density of active sites and well-developed porosity enable highly selective adsorption of metal ions. Furthermore, the modification of sapropel with flocculants can further enhance its activity and application range.

Overall, research on improving the sorption capacity of sapropel through mechanical grinding and thermal activation establishes the foundational methods for preparing effective sorbents. Activation at 350 °C combined with grinding to particle sizes below 50 μm ensures an optimal balance of pore volume, active site density, and surface area. This makes sapropel-based sorbents a modern and economically viable solution for the environmentally safe treatment of industrial wastewater.

II. RESEARCH METHODOLOGY.

Within the scope of this study, the mechanical and thermal processing techniques used to prepare sapropel as an adsorbent with high adsorption activity were investigated step by step. These processing stages — particle size reduction and thermal activation — are crucial in determining the material's surface area, pore volume, and internal structural parameters.

Initially, sapropel samples were collected in their natural state and dried at 105 °C for 5 hours. The dried samples were then subjected to mechanical treatment using a planetary soil grinder and separated into fractions of 600, 400, 200, 100, and 50 μm . Each fraction was sieved and stored in airtight containers. To evaluate the effect of grinding degree on porosity, the total pore volume (cm^3/g) of selected samples was determined using the porometry method.

To monitor the physical and structural changes, data were collected on the opening of sorption-active centers, the development of pore networks, and the overall porosity composition in each size fraction. Special attention was paid to the evolution of micro- and mesopores using gas adsorption techniques. The results of the total pore volume for each fraction are illustrated in Figure 1.

Fractions with particle sizes below 200 μm were subjected to thermal activation. This process was carried out under a nitrogen atmosphere using standard laboratory muffle furnaces. The activation was performed at temperatures of 250, 300, 350, 400, and 450 °C. Each sample was treated at the specified temperature for 2 hours under uniform conditions.

The specific surface area (m^2/g) of the activated samples was determined using the Brunauer–Emmett–Teller (BET) method. For this purpose, nitrogen (N_2) adsorption–desorption isotherms were measured with a dedicated instrument. These analyses provided direct insight into the number of opened pores on the surface and the development of active adsorption sites. The relationship between temperature and surface area is shown in Figure 2.

Microscopic and macroscopic structural changes were observed in all mechanically ground and thermally activated samples. In addition to total pore volume and surface area, indicators such as the types of porosity (micro-, meso-, and macropores), structural stability, and the degree of formation of adsorption centers were recorded. The relationship between structural features and adsorption functionality was also critically analyzed.

III. RESULTS OBTAINED

The porosity and adsorption capacity of sapropel-based sorbents are closely related to the degree of mechanical processing, particularly the grinding level, which significantly influences the total pore volume. Figure 1 clearly illustrates the variation in total pore volume (cm^3/g) for sapropel samples ground to different particle sizes. The histogram presents data corresponding to particle fractions of 600, 400, 200, 100, and 50 μm , arranged from left to right, showing the respective total pore volumes for each fraction. These data provide a solid scientific basis for the conclusion that grinding sapropel can effectively enhance its sorption properties.

In the sample with a particle size of 600 μm , the total pore volume was the lowest, recorded at just 0.03 cm^3/g . This value reflects a state in which the particles remain relatively large, and the material retains its compact and dense structure. At this stage, the porous structure remains largely unopened, and the sorption-active centers are not yet activated. Such coarse-fraction sapropel exhibits not only low porosity but also reduced adsorption capacity, making it unsuitable for effective application in technical purification processes.

When the particle size was reduced to 400 μm , the pore volume doubled to 0.06 cm^3/g . This increase suggests that the grinding process begins to open up the structure, allowing the development of porosity and the initial activation of internal adsorption centers. However, it is likely that the core-shell structure remains partially intact, meaning that not all

active centers are fully accessible. Nevertheless, this fraction represents a notable improvement in performance compared to the 600 μm material.

Further reduction to a particle size of 200 μm resulted in a total pore volume of 0.12 cm^3/g , indicating another twofold increase. At this stage, the mechanical disintegration of the sapropel structure is more pronounced, and valuable mineral, humic, and bituminous components contribute to the formation of increased surface area and the opening of pores. As a result, the effective surface area of the material likely increases significantly due to this mechanical activation. Consequently, this fraction can be considered moderately effective in terms of its capacity to adsorb contaminants from aqueous media.

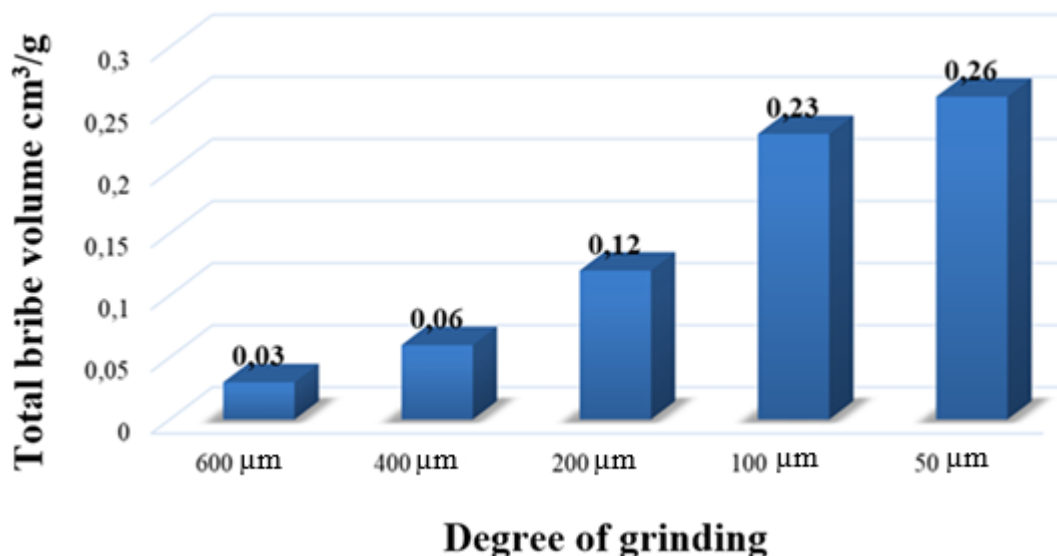


Figure 1. Influence of mechanical grinding degree on the total pore volume of sapropel

When the grinding size is reduced to 100 μm , the total pore volume increases sharply, reaching 0.23 cm^3/g . This indicates that nearly all adsorption centers within the sapropel structure have been activated, and fully developed pores accessible to the internal surfaces have formed. This value is nearly double that of the 200 μm fraction. As the degree of grinding increases, the multilayered structure is disrupted, resulting in a multiple-fold increase in both porosity and surface area. Therefore, materials with this level of structural activation are highly effective for the treatment of industrial wastewater containing elevated concentrations of salts and ionic pollutants.

The highest pore volume is observed in the 50 μm fraction, where it reaches 0.26 cm^3/g . This represents nearly a ninefold increase compared to the 600 μm fraction. At this level of grinding, sapropel transforms into an ultrafine-structured activated sorbent. Due to the reduced particle size, the surface energy becomes significantly elevated, allowing rapid and efficient adsorption of ions, organic compounds, and colloidal pollutants from aqueous environments. Furthermore, sapropel in this fraction is capable of forming nanolayers, which can further enhance purification efficiency.

Based on the analysis, a directly proportional relationship is observed between the degree of mechanical grinding and the total pore volume. This highlights the importance of selecting an optimal grinding size in the preparation of sorbents intended for water treatment applications. At the same time, the diminishing difference in pore volume between the 100 μm and 50 μm fractions suggests the presence of a practical limitation, wherein further grinding yields reduced incremental benefits. Recognizing this effect is critical to avoid unnecessary costs. As such, the 100–50 μm particle size range can be considered optimal for effective adsorption and purification processes.

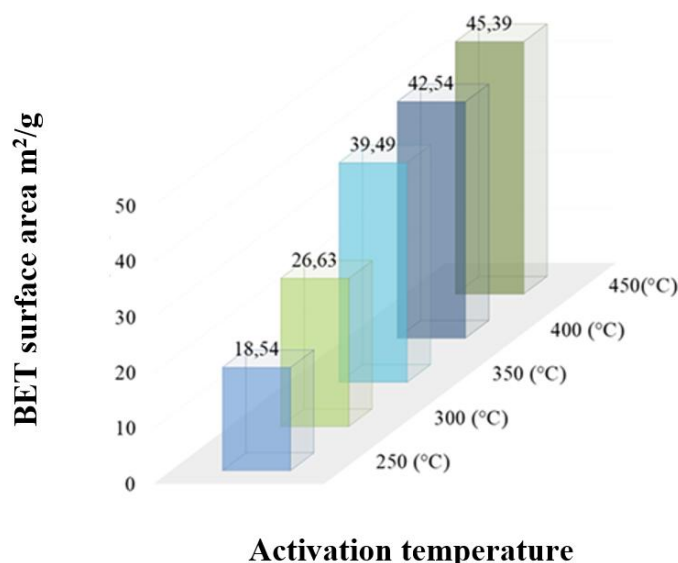


Figure 2. Effect of thermal activation temperature on the surface area of mechanically processed sapropel

One of the key parameters determining the adsorption capacity of sapropel-based sorbents—particularly those derived from natural materials like bentonite or sapropel—is the specific surface area. The influence of activation temperature on surface area, as shown in Figure 2, was determined using the Brunauer–Emmett–Teller (BET) method. As the figure indicates, increasing the activation temperature leads to a significant rise in the specific surface area. This process is explained by the opening of internal pores, decomposition of organic matter, and the formation of active adsorption sites within the skeletal structure of the material.

At 250 °C, the BET surface area reaches 18.54 m²/g, indicating low surface activity. At this temperature, only physically bound moisture is removed from the sorbent, and no significant structural transformations occur. Consequently, active centers are not yet fully developed, and many pores remain partially closed. As a result, the material remains ineffective for adsorption-based purification applications at this stage.

As the activation temperature increases to 300 °C, the BET surface area rises to 39.49 m²/g. At this point, thermal decomposition of organic components begins, and both mesopores and micropores within the mineral skeleton expand, contributing to a sharp increase in surface activity. More active centers are formed, and the number of adsorption sites increases, making the material effective for capturing a wide range of ions, colloidal species, and organic contaminants.

At 350 °C, the surface area reaches 42.54 m²/g—an optimal value for sapropel-based adsorbents. At this temperature, the pore structure, surface centers, and mineral skeleton remain stable. In other words, the mechanical integrity of the sorbent is preserved, while its adsorption performance remains high. Thus, 350 °C can be considered the optimal temperature for thermal activation. At this stage, a balance is achieved where both the surface area and pore system are fully developed.

Further increasing the temperature to 400 °C results in a surface area of 45.39 m²/g, indicating peak surface activation. However, at this stage, partial structural degradation may occur within the mineral skeleton due to the complete decomposition of residual organics. While the number of active sites increases, large pores may become unstable due to washout or re-collapse, potentially limiting the sorbent's reusability.

At 450 °C, a similarly high surface area of 45.39 m²/g is maintained. However, the BET surface area achieved at such high temperatures may not directly correspond to effective adsorption capacity. This is because the dispersion of the pore structure at extreme temperatures may lead to pore collapse or fusion, and mechanical weakness may cause the material to disintegrate quickly. In other words, high activation does not always guarantee structural durability.

IV.CONCLUSION

The mechanical and thermal treatment processes used in the preparation of sapropel-based sorbents have a direct influence on their structural properties, adsorption performance, and practical applicability. The data shown in Figure 1 demonstrate that as particle size decreases through grinding (from 600 µm to 50 µm), the total pore volume of sapropel increases significantly. For example, the pore volume increases from 0.03 cm³/g at 600 µm to 0.26 cm³/g at 50 µm. This directly enhances the material's ability to adsorb contaminant ions from aqueous environments. The formation of micro-

and mesopores due to mechanical fragmentation plays a critical role in the development of effective adsorption centers and ultimately determines the efficiency of water purification.

Figure 2 further illustrates the importance of thermal activation temperature during sapropel processing. Between 250–300 °C, surface area increases gradually, while a peak value of 42.54 m²/g is achieved at 350 °C, representing maximum functional activation. At this point, the active adsorption sites are fully developed, mesopores are well-formed, and the structural stability of the material is maintained. Although surface area continues to increase at 400–450 °C, partial structural degradation becomes more likely. Therefore, 350 °C is identified as the optimal temperature for achieving a balance between mechanical strength and adsorption capacity.

In summary, the combination of fine mechanical grinding (to fractions of 50–100 µm) and thermal activation around 350 °C allows for the production of high-porosity, high-surface-area sapropel-based sorbents. These materials demonstrate strong scientific and practical potential for use in industrial wastewater treatment, enhanced nutrient retention in agricultural systems, and catalytic applications.

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