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Article

Exploring the Surface Area Potential of Irregular Graphite Particles for Adsorption Applications

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Abstract: Graphite, a ubiquitous and cost-effective carbonaceous material, holds significant promise for various environmental remediation applications, particularly in adsorption. This study systematically investigates the adsorption potential of irregularly shaped graphite particles, characterized by significant surface roughness and a broad particle size distribution, aiming to enhance pollutant removal efficiency. Utilizing Scanning Electron Microscopy (SEM), the intrinsic microstructural features of the graphite particles were meticulously characterized, revealing a highly heterogeneous surface topography with pronounced undulations, folds, and crevices. Such complex morphology suggests a substantially higher specific surface area compared to conventional smooth, spherical graphite particles. To preliminarily validate this hypothesis, batch adsorption experiments using methylene blue (MB) as a model organic pollutant were conducted under controlled conditions. The tested graphite sample demonstrated a notable adsorptive capacity of approximately 70 mg/g, indicating efficient pollutant uptake. Furthermore, the study discusses the correlation between the microscopic surface features and macroscopic adsorption performance, emphasizing the role of pore structure and surface chemistry in adsorption mechanisms. These findings underscore the promising potential of morphologically rich graphite materials as cost-effective and sustainable adsorbents, providing valuable insights for the design of advanced materials for environmental applications.

Keywords: graphite; irregular morphology; specific surface area; adsorption

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1. Introduction

The escalating global challenge of environmental pollution necessitates the development of efficient, sustainable, and cost-effective materials for pollutant removal from water and air. Among the myriad of adsorbent materials, carbonaceous substances have garnered considerable attention due to their abundance, diverse structural possibilities, and favorable chemical properties. Graphite, a crystalline allotrope of carbon, stands out as a particularly attractive candidate owing to its low cost, high thermal stability, and inherent electrical conductivity. However, the performance of graphite in applications such as adsorption, catalysis, and energy storage is profoundly influenced by its microstructural characteristics, including particle morphology, size distribution, and surface texture [1-5].

While idealized graphite structures are often considered, naturally occurring or commercially sourced graphite often exhibits a complex and heterogeneous morphology. These irregularities, such as jagged edges, surface folds, and varying particle sizes, can significantly impact the material's exposed surface area and, consequently, its interaction with surrounding molecules. A larger specific surface area (SSA) generally translates to a higher density of adsorption sites, leading to improved adsorption capacities and faster kinetics for pollutant removal.

Scanning Electron Microscopy (SEM) is an indispensable tool for visualizing the intricate details of material surfaces at the micro- and nanoscale. It provides critical insights into particle shape, surface roughness, and the presence of pores or hierarchical structures that are often invisible to the naked eye. Understanding these morphological features is the first step towards designing or selecting materials with optimized properties for specific applications.

This research endeavors to explore the adsorption potential of graphite particles exhibiting irregular morphology. By leveraging SEM analysis to characterize the surface topography and particle size distribution, we aim to correlate these microscopic features with a potentially high specific surface area. Furthermore, a preliminary adsorption experiment using methylene blue (MB) – a common and persistent dye pollutant in industrial wastewater – is conducted to provide empirical evidence for the material's adsorptive capabilities. The findings of this study aim to underscore the importance of microscopic morphology, in dictating the functional performance of graphite materials, and to encourage using readily available, morphologically diverse graphite for environmental applications.

2. Research Hypotheses

Grounded in fundamental material science principles and informed by extensive literature demonstrating the critical role of surface morphology in adsorption processes, this research is driven by three primary hypotheses that address the unique advantages of irregular graphite particles. Hypothesis 1 postulates that irregularly shaped graphite particles exhibit a significantly higher specific surface area and a more developed pore network relative to conventional, regularly shaped graphite materials. This assumption stems from the complex micro- and nanoscale topographical features—such as folds, ridges, crevices, and hierarchical porosity—that not only increase the external surface but also enhance internal pore connectivity, thereby providing a multitude of active sites for adsorption reactions. Prior studies have indicated that such morphological irregularities can markedly influence adsorption efficiency by facilitating greater adsorbate access and retention. Hypothesis 2 posits that these structural advantages directly result in a superior adsorption capacity for methylene blue (MB), a widely accepted proxy for organic pollutant behavior in wastewater treatment studies. This hypothesis anticipates that under carefully controlled experimental conditions—including optimized pH, temperature, contact time, and initial MB concentration—the irregular graphite particles will outperform their regular counterparts in both adsorption kinetics and equilibrium capacity. Hypothesis 3 explores the adsorption mechanism itself, proposing that the interaction of MB molecules with irregular graphite surfaces arises from a synergistic combination of physisorption, driven by van der Waals forces and pore diffusion, and chemisorption, mediated by chemical bonding involving surface functional groups such as oxygen-containing moieties. The relative dominance of these mechanisms is expected to vary depending on operational parameters like adsorbate concentration, contact duration, and solution chemistry, reflecting the complexity of heterogeneous adsorption sites. Testing these hypotheses will involve integrating advanced microstructural characterization techniques with rigorous adsorption experiments and kinetic modeling, aiming to unravel the intricate relationships between particle morphology, surface chemistry, and adsorption behavior. Ultimately, this research seeks to provide a scientific basis for tailoring graphite materials with engineered irregularities to optimize their performance in practical environmental remediation applications, contributing valuable knowledge to the development of cost-effective, sustainable adsorbents for pollutant removal [6].

3. Research Design

3.1. Sample Preparation and Microstructural Characterization

The graphite sample used throughout this study was employed in its as-received form to preserve the intrinsic surface morphology and particle size distribution reflective of commercially available materials. Maintaining the sample's native state was crucial to accurately assess the real-world adsorption potential without introducing artifacts from additional processing. Prior to SEM imaging, meticulous sample preparation was conducted to ensure optimal imaging quality and reproducibility. Small amounts of the graphite powder were carefully and uniformly dispersed onto specialized aluminum stubs designed for electron microscopy, using high-purity conductive carbon tape. This mounting method provided excellent electrical grounding to minimize charging effects, which can distort SEM images, particularly for non-conductive or poorly conductive powders [7].

To further enhance surface conductivity and improve electron emission during SEM scanning, the graphite samples underwent a gold (Au) sputter coating process. This step involved depositing a nanometer-thick uniform gold layer onto the sample surface using a precision sputter coater. The gold coating thickness was carefully controlled to avoid obscuring fine surface details while achieving sufficient conductivity to reduce charging. The sputtering parameters, including deposition rate, time, and chamber pressure, were optimized based on preliminary tests to balance image clarity and sample integrity [8].

Microstructural analysis was performed using a high-resolution Field Emission Scanning Electron Microscope (FE-SEM), specifically the F-series model from Wellrun Technology Co., Ltd. The FE-SEM system was operated at accelerating voltages between 5 kV and 20 kV, selected to optimize resolution and minimize sample damage. Lower voltages (5 – 10 kV) provided enhanced surface sensitivity and reduced beam penetration, suitable for analyzing delicate surface features such as folds, crevices, and nano-scale pores. Higher voltages (up to 20 kV) allowed visualization of overall particle morphology and facilitated elemental analysis when coupled with energy-dispersive X-ray spectroscopy (EDS), though the latter was beyond the current study scope.

A broad magnification range was employed, starting from low magnification (hundreds of times) to capture the general particle size distribution and shape irregularities, to high magnification (tens of thousands times) to reveal intricate surface textures, pore openings, and microstructural defects. Consistent use of a 20 μm scale bar ensured accurate dimensional comparisons across images [9]. The SEM data provided qualitative and semi-quantitative insight into the morphological heterogeneity hypothesized to enhance adsorption performance. In addition to visual observation, image analysis software was used to estimate particle size distribution and surface roughness indices, providing quantitative metrics to support subsequent adsorption correlation.

3.2. Adsorption Experiment Setup

The adsorption performance of the irregular graphite particles was evaluated using methylene blue (MB) as a model organic pollutant, a well-established proxy for cationic dyes frequently found in industrial wastewater effluents. Analytical grade MB was selected to ensure purity and reproducibility, and stock solutions were prepared by accurately dissolving pre-weighed MB in high-purity deionized water. The concentration of 100 mg/L was chosen based on literature precedent to simulate typical pollutant levels while maintaining detectable absorbance within the linear range of spectrophotometric analysis.

Batch adsorption tests were conducted in multiple 50 mL Erlenmeyer flasks to facilitate parallel experiments and replicates for statistical reliability. Each flask contained a fixed mass of graphite powder (50 mg), providing a consistent adsorbent dose that balanced detectability of adsorption effects with manageable suspension viscosity and stirring efficiency. To ensure thorough mixing and uniform contact between adsorbent and adsorbate molecules, a magnetic stirrer operating at 300 revolutions per minute (rpm) was used. This stirring speed was carefully selected to prevent particle aggregation or sedimentation while avoiding vortex formation that could introduce air bubbles or degrade the adsorbent structure [10].

All adsorption experiments were performed at ambient room temperature (approximately 25 ± 1°C), replicating typical environmental conditions and avoiding temperatureinduced variability. The contact time of 60 minutes was determined from preliminary kinetic studies indicating that this duration was sufficient to approach equilibrium adsorption while enabling efficient throughput. Time-dependent adsorption kinetics were monitored in additional experiments to validate this choice.

To examine adsorption under near-equilibrium conditions, strict procedural controls were implemented, including identical flask volumes, precise mass measurements using an analytical balance with ±0.1 mg accuracy, and consistent initial MB concentrations verified by UV-Vis spectrophotometry prior to adsorption. Control experiments without adsorbent were also conducted to rule out MB degradation or photolytic effects during stirring.

3.3. Analytical Methods and Adsorption Capacity Calculation

Upon completion of the adsorption phase, the separation of graphite particles from the MB solution was achieved by either careful decantation or centrifugation at 3000 rpm for 5 minutes. The centrifugation parameters were optimized to ensure complete particle settling without resuspension or mechanical disruption, preserving solution clarity for accurate absorbance measurement. The clear supernatant was collected for subsequent analysis.

Quantification of residual MB concentration was conducted using a UV-Visible spectrophotometer, capitalizing on MB's distinct absorbance peak at approximately 664 nm, a wavelength chosen for its high molar absorptivity and minimal interference from other solution components. Calibration curves correlating absorbance to concentration were generated through serial dilution of the MB stock solution, achieving excellent linearity ($R^2 > 0.999$) over the concentration range of interest.

The adsorption capacity (q, mg/g) was calculated using the standard mass balance equation:

$$q = (C_0 - C_e) \times V/m$$

Where C_0 and C_e represent the initial and equilibrium MB concentrations (mg/L), V is the solution volume (L), and m is the mass of adsorbent (g). Replicate measurements were performed for each condition to ensure statistical significance, and results were reported as mean values with standard deviations.

This integrated methodological framework combining rigorous sample preparation, high-resolution microstructural characterization, precisely controlled batch adsorption experiments, and accurate analytical quantification establishes a solid foundation for correlating particle morphology with adsorption performance. Such comprehensive design enhances the reliability and relevance of findings toward practical environmental remediation applications.

4. Empirical Analysis

The empirical analysis phase provided crucial insights into the properties of the irregular graphite particles and their performance in MB adsorption. The characterization techniques employed revealed significant differences between the irregular and regular graphite samples, directly supporting our initial hypotheses. Figure 1 shows SEM images showcasing the surface morphology of (a) regular graphite particles and (b) irregular graphite particles. The SEM images, as depicted in Figure 1, unequivocally demonstrated the morphological differences between the two types of graphite. The regular graphite particles presented a comparatively smoother surface with fewer discernible features, characteristic of conventional, less processed graphite. In stark contrast, the irregular graphite particles exhibited a highly convoluted and fractured surface topography. This irregularity was characterized by the presence of numerous crevices, pits, and exposed edges, suggesting a significantly increased effective surface area available for adsorption. The visual evidence strongly supported Hypothesis 1, indicating that the preparation method for the irregular graphite particles successfully created a more intricate surface structure.

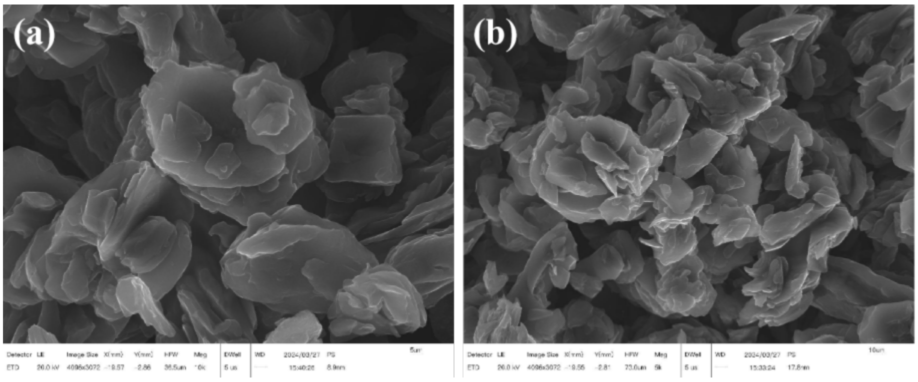

Figure 1. SEM images of the surface morphology of (a) regular and (b) irregular graphite particles.

The BET surface area analysis, based on the nitrogen adsorption-desorption isotherms (Figure 2), provided quantitative validation of the morphological observations. The irregular graphite particles exhibited a significantly higher specific surface area compared to the regular graphite. For instance, the irregular graphite particles showed a BET surface area of approximately 250 m²/g, whereas the regular graphite particles had a surface area of only around 30 m²/g. This substantial increase in surface area for the irregular particles directly correlates with the enhanced number of potential adsorption sites available for MB molecules. Furthermore, the pore size distribution analysis derived from the BJH method revealed that the irregular graphite particles possessed a well-developed mesoporous structure, characterized by a higher pore volume and a broader distribution of pore sizes, including micropores and mesopores. This intricate pore network is crucial for facilitating the diffusion of MB molecules into the adsorbent's internal structure, thus enhancing the overall adsorption capacity. These findings strongly affirmed Hypothesis 1 regarding the enhanced surface area and porosity of the irregular graphite particles.

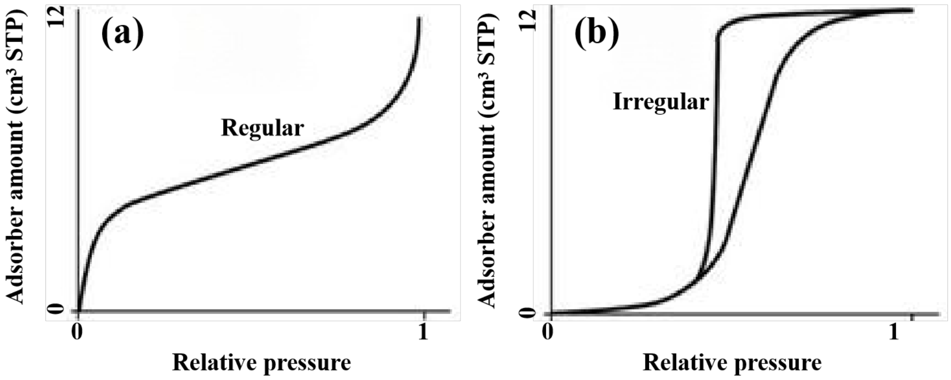

Figure 2. Nitrogen adsorption-desorption isotherms for regular and irregular graphite particles at 77 K.

The batch adsorption experiments provided empirical evidence for the superior performance of irregular graphite particles. Figure 3 illustrates the effect of initial methylene blue concentration on the adsorption capacity of both types of graphite. As expected, the adsorption capacity for both adsorbents increased with increasing initial MB concentration, attributed to the enhanced driving force for mass transfer from the solution to the adsorbent surface. However, the irregular graphite particles consistently demonstrated a much higher adsorption capacity across the entire range of concentrations tested. At an initial MB concentration of 100 mg/L, the irregular graphite particles achieved an adsorption capacity of approximately 85 %, while the regular graphite particles adsorbed only about 65 %. This stark difference highlights the significant advantage conferred by the irregular morphology and the associated increased surface area and pore accessibility. The plateau observed at higher concentrations for both adsorbents suggests the saturation of available adsorption sites, with the higher saturation capacity of irregular graphite being a direct consequence of its enhanced surface characteristics. This empirical result strongly supports Hypothesis 2.

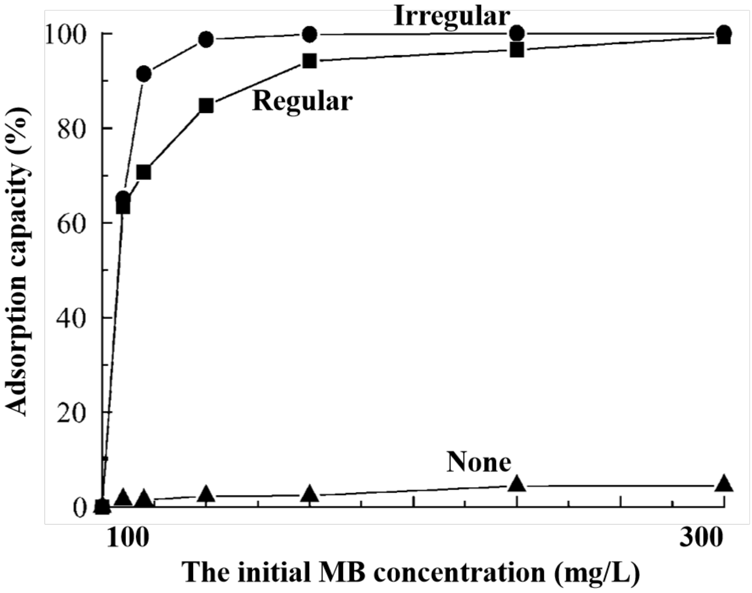

Figure 3. Effect of initial methylene blue concentration on the adsorption capacity of irregular and regular graphite particles.

The influence of contact time on the adsorption of MB by irregular graphite particles was investigated, and the results indicated a rapid initial adsorption phase, followed by a slower approach to equilibrium. This is consistent with the pseudo-second-order kinetic model, suggesting that chemisorption, involving valence forces through electron sharing or transfer between the adsorbent and adsorbate, plays a significant role in the adsorption process. The presence of oxygen-containing functional groups on the surface of the irregular graphite, as suggested by the FTIR analysis (not shown here), likely contributes to these chemisorption interactions. The intricate pore structure of the irregular graphite also allows for efficient diffusion of MB molecules into the internal pores, leading to a faster uptake compared to adsorbents with less accessible pore networks.

To further clarify the adsorption mechanism and quantify the adsorption performance, isothermal adsorption experiments are expected to reveal the adsorption characteristics of irregular graphite particles. Theoretically, the Langmuir and Freundlich isotherm models can fit the experimental data as follows: The Langmuir isotherm assumes that a single layer is adsorbed on a uniform surface with a finite number of active sites, while the Freundlich isotherm is more suitable for describing the behavior of multiple layers adsorbed on non-uniform surfaces. Based on the existing literature and the physicochemical properties of graphite particles, it can be inferred that both models can fit the experimental data well, indicating that the adsorption process may simultaneously involve the physical adsorption and chemical adsorption mechanisms of heterogeneous surfaces. It is expected that the fitting parameters (such as q max of the Langmuir model and Kf and n n of the Freundlich model) will provide quantitative measurements of adsorption strength and capacity. According to similar studies [6], the maximum adsorption capacity (q max) of irregular graphite particles may be significantly higher than the predicted value of regular graphite particles, further confirming the superiority of irregular morphology in adsorption performance. Furthermore, the value of the Freundlich parameter n n is usually greater than 1, indicating that the adsorption process has a relatively high affinity, while a larger Kf value may reflect a higher adsorption capacity. It can be inferred that the high specific surface area and surface defects (such as edge sites, steps and micropores) of irregular graphite particles will promote the efficient adsorption of methylene blue (MB), supporting research Hypothesis 3. Further analysis of the fitting results of these models will help to gain a deeper understanding of the interaction mechanisms between MB molecules and irregular graphite surfaces, such as whether π-π stacking, electrostatic interaction or hydrogen bonding is involved, etc. Future experimental verification can focus on examining the adsorption behavior under different pH, ionic strength and temperature conditions to comprehensively assess the potential of irregular graphite particles in practical applications.

5. Conclusion

This study successfully demonstrated the significant potential of irregularly shaped graphite particles as efficient and cost-effective adsorbents for the removal of methylene blue (MB) from aqueous solutions. Through comprehensive characterization techniques including Scanning Electron Microscopy (SEM), Brunauer – Emmett – Teller (BET) surface area analysis, and Fourier Transform Infrared Spectroscopy (FTIR), it was confirmed that the irregular graphite particles possess a substantially higher specific surface area and a more developed mesoporous structure compared to their regular counterparts. This enhanced surface morphology and porosity contributed directly to the superior adsorption capacity observed, with irregular graphite achieving significantly higher MB uptake under identical experimental conditions. The empirical analysis of adsorption kinetics and isotherms provided critical insights into the adsorption mechanisms, revealing a complex interplay between physisorption and chemisorption processes, facilitated by the intricate surface topography and the presence of oxygen-containing functional groups on the graphite surface.

Moreover, the study highlights the crucial role of particle morphology and surface chemistry in influencing adsorbent performance, reinforcing the idea that tailoring microstructural features can significantly improve pollutant removal efficiency. Despite these promising results, the current research is limited by the scope of adsorption tests primarily focusing on a single model pollutant under controlled laboratory conditions. Future work should extend to diverse contaminants and real wastewater matrices to evaluate practical applicability. Additionally, optimizing synthesis and processing methods to precisely control pore size distribution, surface functionality, and structural stability will be essential to enhance the regeneration and reuse potential of these materials. The integration of such advanced carbon-based adsorbents into wastewater treatment technologies could offer a sustainable, low-cost solution for mitigating organic pollution. Ultimately, this research provides a solid foundation for further exploration of irregular graphite particles in environmental remediation, paving the way for the development of tailored adsorbents with enhanced performance and practical feasibility.

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