Optimization of Methylene Blue Dye Adsorption Using ADF$_{OSPOLYI}$ Clay as a Novel Adsorbent in Wastewater Treatment

[1]Adetoro Afusat Adedokun, [2]Fatai Afolabi, [1]Olukemi Rebecca Adebayo, [2]Basheer Akanbi Jaji, [3]Olumide Afolabi

[1]Applied Sciences Department, Osun State Polytechnic, Iree, Osun State, Nigeria.
[2]Department of Physical and Chemical Sciences, Federal University of Health Sciences, Ila-Orangun, Osun State, Nigeria.
[3]Department of Science Laboratory Technology, Osun State Polytechnic Iree, Osun State, Nigeria.
*Corresponding Author: fatai.afolabi@fuhsi.edu.ng

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Abstract: This innovative study offers a fresh method for enhancing clay-based adsorption systems by investigating the application of ADF$_{OSPOLYI}$ clay as a very effective adsorbent for the removal of methylene blue dye from wastewater. Advanced methods such as energy-dispersive X-ray analysis, scanning electron microscopy, X-ray diffraction, and Fourier-transform infrared spectroscopy (FT-IR) were used to completely characterize the clay. The clay's suitability as an adsorbent was validated by these studies, which showed adsorption efficiencies continuously above 90%. By contrasting the impacts of KOH and HCl activation on the clay's adsorption capability, the study presents a fresh element, showing unrealized potential in surface feature optimization and providing encouraging results. The contrast between the reduced Langmuir surface area, which suggests underutilized adsorption sites, and the large BET surface area, which indicates great interaction potential with dye molecules, is one of the study's main findings. Future studies into surface area optimization to improve adsorption performance are made possible by this revelation. The study also offers fresh perspectives on how adsorbent mass affects efficiency, showing that lower clay masses (around 0.5 g) typically produce better results, sometimes surpassing 97% efficiency. Compared to conventional techniques, which frequently fall short of this high effectiveness at lower adsorbent dosages, this is a major advance. With ADF $_{OSPOLYI\ clay}$ showing promise as a contender, this work represents a significant step in enhancing the sustainability and effectiveness of clay-based wastewater treatment. The results highlight the necessity of additional adsorption site and surface property improvement to fully realize its potential for environmental remediation.

Keywords: ADF$_{OSPOLYI}$ clay, Adsorption efficiency, Methylene blue dye, Wastewater, treatment, Surface optimization

Introduction

Water is essential for all life on Earth, serving as a fundamental source of energy and life. In its purest form, water is colorless, odorless, and tasteless. However, various industries, including textiles, paper, and plastics, utilize dyes to color their products. These dyes are often discharged into water bodies as waste, leading to significant environmental challenges.(Tura et al., 2018) . In recent decades, the demand for textile products has surged due to urbanization, resulting in the expansion of dyeing industries. As a result, the wastewater from these industries has become concentrated in water bodies, containing hazardous compounds like dyes, by-products, and heavy metals.(Shindhal et al., 2021). These pollutants pose a serious threat to the ecosystem and human health if they remain untreated for extended periods. Dyes, which are widely used in the textile industry to color fabrics, yarns, polymers, and other materials, are particularly concerning. Their complex chemical structures make them non-biodegradable, causing significant environmental pollution (Fito et al., 2023). Among the pollutants, dyes are some of the most persistent toxicants found in water bodies. If left untreated, they can have severe consequences for both aquatic ecosystems and human health(Dindorkar et al., 2022). Several physicochemical methods are available to treat dye-laden wastewater, including adsorption, coagulation, chemical oxidation, advanced oxidation, and flocculation. Of these methods, adsorption has attracted significant attention due to its efficiency, low cost, and simplicity(Ibrahim et al., 2023). Adsorption is a surface process where substances, such as atoms, ions, or molecules, adhere to a solid surface (Mhemeed, 2018). This process results in a layer of adsorbate (the material being absorbed) accumulating on the adsorbent's surface (the substance onto which the adsorbate attaches). Unlike absorption, where substances permeate or dissolve into a liquid or solid, adsorption involves the accumulation of substances at a surface or interface. Adsorption can be classified as physisorption, which involves weak Van der Waals forces, or chemisorption, which involves covalent bonding due to electrostatic attraction(Mahmood Aljamali et al., 2021). These processes are essential in various natural, biological, and chemical systems. Adsorption is widely employed in industrial applications, such as activated charcoal for water purification, adsorption chillers for cooling, synthetic resins, and enhancing the storage capacity of nanoporous carbons (Chauhan et al., 2022). Dyes are considered organic contaminants in wastewater, especially in industries like textiles, paper, paint, printing, and cosmetics(Manzoor & Sharma, 2019). The Environmental Regulation Act mandates that these dyes must be treated to reduce their concentrations to acceptable levels before being discharged into water bodies (NEPA, 2009). Due to their high solubility in water, dyes are easily dispersed, posing risks to human health, aquatic life, and crops. Among these dyes, methylene blue (MB) is a widely used cationic dye that contributes to water pollution. When ingested in drinking water, MB can cause adverse health effects such as nausea, vomiting, and diarrhea(Manzoor & Sharma, 2019).

The search for efficient and cost-effective methods to remove dyes from wastewater has led to the development of various adsorption techniques. Activated carbon is the most commonly used adsorbent due to its high capacity for adsorbing organic materials (Abegunde et al., 2020). However, the production of activated carbon is expensive. As a result, researchers have explored cheaper alternatives, such as zeolites, natural phosphate , peat, fruit peels, plant waste, earth materials, and clays.[12-14]. The use of low-cost adsorbents, especially clay materials, has gained attention due to their environmentally friendly properties. Clays possess several advantageous properties, including high surface area, surface chemistry, non-toxicity, and potential for ion exchange. These characteristics make clays promising candidates for dye removal from wastewater (Ewis et al., 2022). Moreover, clays offer an eco-friendly alternative to activated carbon and can be used in sustainable water treatment solutions. By selecting the appropriate adsorbent, it is possible to achieve effective and sustainable dye removal from wastewater, thus ensuring the recovery of water quality.In conclusion, water pollution caused by dyes from industries like textiles poses a significant environmental threat. Various treatment methods, particularly adsorption, provide promising solutions for mitigating the harmful effects of dye pollutants. While activated carbon remains a popular choice for adsorption, research into cheaper, eco-friendly alternatives, such as clay, offers new possibilities for cost-effective and efficient water purification. These advancements are crucial in addressing the growing problem of industrial wastewater pollution and its impact on ecosystems and human health. One of the main pollutants and toxicants found in water bodies is dyes, which are also associated to toxicity and have detrimental effects on the ecosystem if left in the water bodies for an extended length of time(Dindorkar et al., 2022). In specifically, dyes are useful and colorful substances used in the textile industry to color textiles, yarns, polymers, and other substrates. But because of their complex chemical makeup and numerous smearings, they are non-biodegradable, which causes the environmental system to become polluted (Fito et al., 2023; T.K. Sen, 2023). Numerous physicochemical methods are currently available to treat dye-containing wastewater, such as adsorption, coagulation, chemical oxidation, advanced oxidation, and occulation. Adsorption has garnered significant interest from researchers worldwide owing to its remarkable efficacy, low operational expenses, and simple manner of operation. Hence, this research aims to explore the potential of clay materials as an environmentally friendly and cost-effective alternative to activated carbon for dye removal from wastewater. Given their high surface area, surface chemistry, non-toxic nature, and ion exchange capabilities, clay materials offer promising sorption properties. This study seeks to evaluate the efficiency of these materials in adsorbing dyes, to improve water treatment processes and ensure the recovery of high-quality water.

Materials and Methods

Adsorbent preparation

Natural clays used in this work was collected from Faculty of Industrial Art and Design, Osun State Polytechnic, Iree, crushed and sieved to obtain particles of smaller size and dried on the rock for 24 h.

Characterization of the clay

FTIR spectra were recorded from 4000 to 400cm^{-1} using a Perkin Elmer Paragon 3000 MX Spectrometer and analysed using the spectroscopic software Win-IR Pro version 3.0. FTIR spectral of clay sample was taken to obtain information on the nature of possible interaction between the functional group of the sample and biosorbate. X-ray diffraction (Equinox 3000 france) was used for determination of the crystalline structures of the clay. Clay's morphophological structure were studied by field emission scanning electron microscopy (FE-SEM" miras 3 Tescan, Czech). The clay sample was gold plated for SEM test. Specific surface area (SSA) of clay was obtained using an autosorb-1 surface area analyzer. Quatachrome (Brunauer-Emmett-Teller (BET). The inorganic chemical composition were measured with an Energy Dispersive X-ray analyzer (EDX-720 shimadez incorporation Tokyo Japan) by using a fundamental parameters method.

Preparation of solutions

All chemicals used in this study were of analytical reagent grade and were used without further purification. Standard solution of methylene blue dye, hydrochloric acid and potassium hydroxide used for this study were prepared from their salts. the working solution of the dye with different concentration were prepared by appropriate dilution of the stock solution which is 1000ppm obtained by dissolving 1g of methylene blue dye in 1000cm^3 of water in standard volumetric flask.

Materials and Methods

Activation of acid and base

For acid activation: 50ml of hydrochloric acid with concentrations of 2M, 3M and 4M were mixed with 8g of clay sample in 3 different conical flasks and left to stand for 24hours. Each solution was filtered and the residues were washed with distilled water to acid free indicated by negative test with $AgNO_3$ solution or methyl orange. The clay residues were dried in an oven at 110-120^{o}C. The dried clay is stored in a desiccator to prevent moisture absorption before use.

For base activation: 50ml of 2M KOH was mixed with 30g of clay in a 100ml glass cup. The mixture was stirred continuously with a magnetic stirrer for 5 hours and filtered to get the supernatant and the solid phase separately. The residue was washed with deionized water severely to free the O-H by testing with phenolphthalein indicator).the solid phase was then dried in oven at a temperature of 100^{o}C to dry the dried clay is crushed with mortar and sieved with mesh.

Batch Experimentation

In batch adsorption experiments, the adsorption dosage of 0.5g, 1g and 2 g of ADF$_{OSPOLYI}$ activated HCl (2M, 3M, and

4M) were added to 100ml conical flasks having 50ml methylene blue dye 50ppm solution and stirred for 24 hours The filtrate was then measured for absorbance using UV-Vis at 664 nm. Wavelength. Likewise for base activation 0.25, 1g, 0.5g,1g. 2g,5g of heated activated clay were treated the same way as in acid activated.

Results and discussion

Surface characterization of ADF**OSPOLYI** clay was performed before adsorption using FTIR, SEM, XRD, EDX, and surface area analyses. The results are shown in Figures 2-5. The FTIR spectra were collected to understand better the surface characteristics of the natural and activated clays. FTIR spectra were recorded in the range of 400-4000cm-1. Surface chemistry is important especially when chemisorption is dominant in the adsorption system. The following characteristics of infrared bands were observed, 3765cm-1 assigned to in-phase symmetric OH stretching vibration. The FTIR peak at 3417cm-1 is linked to the stretching vibration of adsorbed H_2O. The peak at 1629.81nm corresponds to the bending vibration of adsorbed H_2O. The high intensity of the peak appearing at 1075cm-1 is an indication of large amount of mineral in the sample. The peak at 1075 and 457.86cm-1 denotes siloxane (Si-O-Si) stretching and bending vibration. The peak at 776.62nm denotes the bending vibration of AIAIOH and 840nm to the bending vibration of AIMgOH.

The components of the clay include Silica-aluminate, ferrite, kaolin, and illite with quartz being the most dominant and illite is very minute of the clay. A secondary electron microscope can produce very high-resolution images of the sample's surface revealing the presence of wider pores in the clay sample. The inorganic chemical composition of the sample was measured with an energy disperse X-ray analyzer (EDX) by using fundamental parameter methods. EDX results indicated the sample's significant presence of silicon, oxygen, and aluminum.

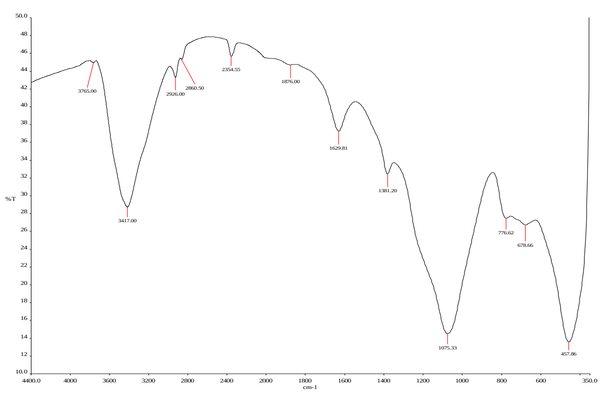

Fig. 1: FTIR spectra of ADF**OSPOLYI** clay sample

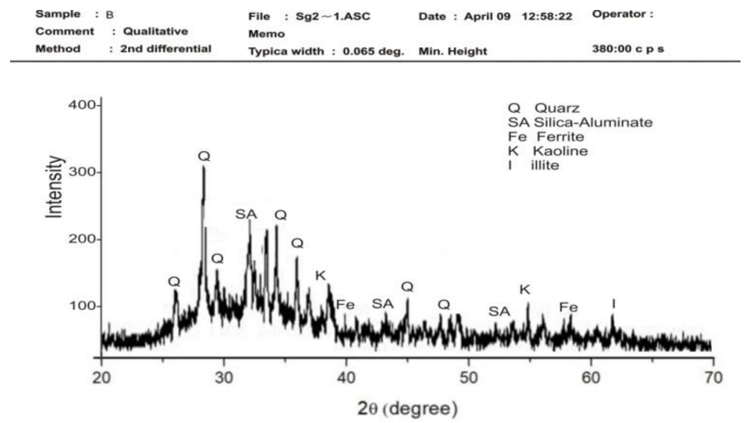

Fig. 2: XRD pattern of ADF**OSPOLYI** clay

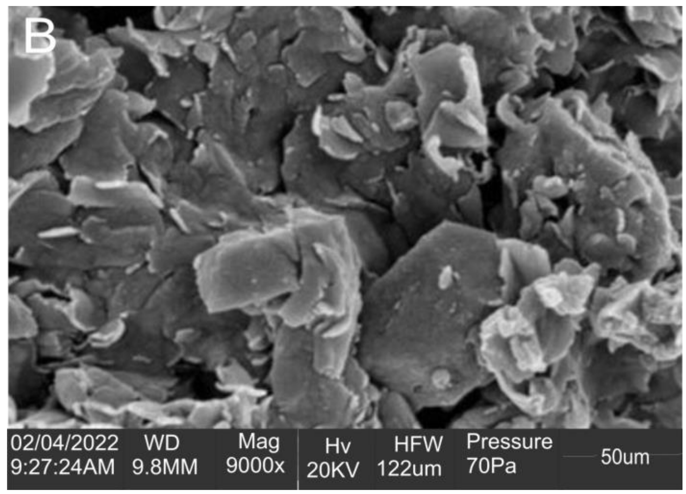

Fig. 3: SEM micrographic of **ADFOSPOLYI clay**

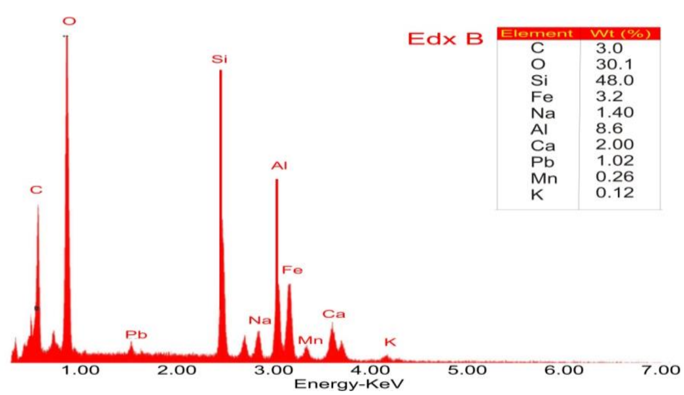

Fig. 4: EDX of ADF**OSPOLYI** clay

The surface area measurements of ADFOSPOLYI clay are shown in Table 1 and were assessed using the Langmuir Surface Area and the Brunauer-Emmett-Teller Surface Area techniques. The total surface area of porous materials, such as clays, is frequently measured using the BET method. This approach is ideal for figuring out the total surface area accessible for adsorption since it takes into account multilayer adsorption on the surface. The comparatively large surface area of the ADF$_{OSPOLYI}$ clay, as indicated by its surface area of 68.22 m²/g, is advantageous for adsorptive processes such as dye removal.

Table 1: Surface area of ADF$_{OSPOLYI}$ clay

Adsorbent	Bet Surface area (m^2g^{-1}) Langmuir SA (m^2g^{-1})
ADF$_{OSPOLYI}$	68.22 52.5

The methylene blue dye molecules have access to more adsorption sites when the surface area is greater. Although some adsorption sites may not be fully utilized as suggested by the Langmuir data. Monolayer adsorption on a surface with a finite number of identical sites is assumed by the Langmuir model. When a single layer of dye molecules covers the surface, the surface area obtained using this method represents the maximal adsorption capacity. Only a percentage of the total accessible surface area (from the BET) appears to contribute to efficient adsorption, as indicated by the lower value of 52.5 m²/g as compared to the BET measurement. This is because the BET technique more effectively accounts for multilayer adsorption and the porous structure of the clay than the Langmuir model does. Nonetheless, the very large surface area values obtained from both techniques suggest that ADF$_{OSPOLYI}$ clay may be a useful adsorbent for eliminating methylene blue dye from wastewater, although some adsorption sites may not be fully utilized as suggested by the Langmuir data.
Table 2 represents the results of an experiment focused on optimizing the adsorption of methylene blue dye from wastewater using ADF$_{OSPOLYI}$ clay activated with 2M KOH. The adsorption process is evaluated by varying the mass of the adsorbent (ADF$_{OSPOLYI}$ clay) and measuring the change in dye concentration. The data points include the initial concentration (Ci), the final concentration after treatment (Ct), the difference between these concentrations (Ci - Ct), and the adsorption efficiency or removal percentage (Cq).

Table 2: The results of activated 2M KOH of ADF$_{OSPOLYI}$ clay sample

Mass (Wg)	C_i	C_t	C_i-C_t(ppm)	Cq(%)
0.25	50	o.343	49.65	99.260
0.5	50	0.936	48.065	98.120
1.0	50	1.357	48.643	97.286
2.0	50	2.410	47.590	95.100
5.0	50	3.000	47.00	94.000

For methylene blue adsorption, the ideal mass of ADF$_{OSPOLYI}$ clay seems to be between 0.25 and 0.5 g, where adsorption effectiveness is at its highest (around 98%). This result was in tandem with a removal efficiency of 82.2 to 99.9% achieved by Fito et al., (2023). Also in tandem with 96.96 to 99.48% achieved by Shah et al., (2022) While more dye is removed overall (as indicated by Ci-Ct values), the adsorption effectiveness decreases with increasing adsorbent masses. This could be the result of ineffective dye diffusion on the clay surface or active site saturation. The experiment therefore shows that ADF$_{OSPOLYI}$ clay activated with 2M KOH is a powerful adsorbent for the removal of methylene blue dye from wastewater. The findings demonstrate that clay has a high adsorption efficiency at lower concentrations, making it a cost-effective wastewater treatment option. The removal percentage is not considerably improved by raising the mass above a particular threshold (about 0.5 g), suggesting that the adsorption process can be maximized by carefully regulating the adsorbent mass employed in the treatment procedure. Table 3 presents the adsorption results for methylene blue dye removal using ADF$_{OSPOLYI}$ clay activated with 2M HCl at varying adsorbent masses. The data reflects changes in dye concentration and adsorption efficiency (Cq) as the mass of the clay is adjusted. At 0.5 g and 2.0 g, the adsorption efficiencies are high (96.22% and 97.43%, respectively), indicating that these masses of ADF$_{OSPOLYI}$ clay are effective in removing methylene blue dye. The 1.0 g sample, however, shows a slight dip in efficiency to 93.38%, which may suggest that the mass of clay at this point is less optimal, potentially due to site saturation or uneven distribution of dye molecules on the clay surface. Comparative adsorption performance indicated that 0.5 g of clay is highly effective, removing over 96% of the dye, but increasing the mass to 1.0 g doesn't improve the efficiency, indicating diminishing returns. At 2.0 g, the efficiency increases again to over 97%, implying that there may be a threshold at which additional adsorbent mass becomes beneficial again, possibly due to better utilization of surface area or increased exposure to adsorption sites

Table 3: The results of activated 2MHCl of ADF$_{OSPOLYI}$ clay sample

Mass (Wg)	Ci	Ct	Ci- Ct	Cq (%)
0.5	50	1.889	48.11	96.22
1	50	3.311	46.60	93.38
2	50	1.290	48.71	97.43

With adsorption efficiencies continuously above 90%, the results indicate that ADF$_{OSPOLYI}$ clay activated with 2M HCl is very effective for removing methylene blue dye. The data also show that careful adsorbent mass optimization is essential since efficiency can change with varying clay contents, potentially as a result of adsorption site distribution and availability.

Table 4 presents the results of the adsorption experiment for ADF$_{OSPOLYI}$ clay activated with 3M HCl. The initial concentration of the solute (Ci) was consistently 50 ppm for all three trials, while the final concentration after treatment

(Ct) varied depending on the mass of the clay used. At 0.5 g of clay, the final concentration of the solute was reduced to 1.36 ppm, leading to a removal of 48.64 ppm, corresponding to a 97.28% adsorption efficiency (Cq). For a mass of 1 g of clay, the final concentration dropped to 0.42 ppm, resulting in a removal of 49.58 ppm and an adsorption efficiency of 99.16%.

When the mass of clay was increased to 2 g, the final concentration of the solute was 1.832 ppm, with a removal of 48.168 ppm, yielding a slightly lower adsorption efficiency of 96.33%. These results indicate that increasing the clay mass generally enhances the adsorption efficiency, with the 1 g sample achieving the highest removal of the solute. However, the adsorption efficiency decreases slightly at 2 g, possibly due to saturation or other limiting factors affecting adsorption capacity at higher masses.

Table 4: The results of activated 3MHCl of ADF$_{OSPOLYI}$ clay sample

Mass (Wg)	Ci	Ct	Ci- Ct	Cq (%)
0.5	50	1.36	48.64	97.28
1	50	0.42	49.58	99.16
2	50	1.832	48.168	96.33

Table 5: The results of activated 4MHCl of ADF$_{OSPOLYI}$ clay sample

Mass (Wg)	Ci	Ct	Ci- Ct	Cq (%)
0.5	50	1.198	48.80	97.6
1	50	1.95	48.05	96.1
2	50	2.007	47.99	95.98

Table 5 presents the results of the adsorption experiment using ADF$_{OSPOLYI}$ clay activated with 4M HCl. The initial solute concentration (Ci) was maintained at 50 ppm for all trials, while the final concentration (Ct) varied depending on the mass of the clay. With a 0.5 g sample of clay, the final concentration was reduced to 1.198 ppm, resulting in a removal of 48.80 ppm and an adsorption efficiency (Cq) of 97.6%. At 1 g of clay, the final concentration was slightly higher at 1.95 ppm, leading to a removal of 48.05 ppm and an adsorption efficiency of 96.1%. When the clay mass was increased to 2 g, the final concentration of the solute remained fairly constant at 2.007 ppm, resulting in the removal of 47.99 ppm and an adsorption efficiency of 95.98%. The results show a trend where increasing the clay mass slightly reduces the adsorption efficiency, possibly due to saturation or diminishing returns in adsorption capacity at higher masses. Despite this, the clay consistently demonstrated high adsorption performance, with efficiencies above 95% across all masses tested. The 0.5 g sample achieved the highest efficiency (97.6%), indicating that lower masses of clay may provide more favorable conditions for adsorption in this case.

Conclusion

The findings across various experiments demonstrate that ADF$_{OSPOLYI}$ clay is a highly effective adsorbent for removing methylene blue dye and other solutes from wastewater. The results consistently show adsorption efficiencies above 90%, with activation using different reagents (KOH and HCl) yielding promising outcomes. A key factor highlighted in the studies is the surface area of the clay. While the high BET surface area indicates substantial interaction potential with methylene blue molecules, the lower Langmuir surface area suggests that certain adsorption sites may not be fully utilized, likely due to surface characteristics or pore size distribution. This suggests that while ADF$_{OSPOLYI}$ clay has strong adsorption potential, there is room for optimization to make better use of all available adsorption sites, potentially enhancing its overall capacity. Additionally, the results reveal that the adsorbent mass plays a significant role in determining the efficiency of the adsorption process. Lower amounts of clay (particularly around 0.5 g) consistently deliver the highest efficiencies, sometimes exceeding 97%, making the process more economical and efficient. Increasing the clay mass beyond 1 g does not significantly improve the removal percentage and can even slightly reduce the efficiency, likely due to saturation of adsorption sites. This trend was observed across experiments using both 3M and 4M HCl activation, where the optimal performance was achieved at lower clay masses. These findings suggest that careful control of the adsorbent mass is crucial to optimizing the process, ensuring that high adsorption efficiency is maintained without wasting excess clay. Overall, ADF$_{OSPOLYI}$ clay demonstrates excellent potential for wastewater treatment applications, with its effectiveness particularly notable at lower masses, but further surface optimization could unlock even greater adsorption capacity.

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