

Unlocking Ghana's Kaolin Potential: Enhancing the Adsorption Capacity of Kaolin through Acid Activation for Environmentally Friendly Remediation of Used Automotive Oil

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Unlocking Ghana's Kaolin Potential: Enhancing the Adsorption Capacity of Kaolin through Acid Activation for Environmentally Friendly Remediation of Used Automotive Oil

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Abstract

This study investigated the potential of acid-activated kaolin obtained from one of the commercial markets in Ghana as a low-cost adsorbent for the recycling of used automotive oil. The kaolin was chemically modified using sulphuric acid under varying

conditions to enhance its adsorption properties. The effects of key operational parameters, including contact time, adsorbent dosage, and initial analyte concentration, were systematically evaluated to determine the optimum adsorption performance. The results showed that acid activation significantly improved the adsorption capacity of the raw kaolin, with optimum performance (approximately 80%) observed at a concentration of 3 M, indicating a balance between structural modification and the preservation of active sites. Adsorption efficiency was found to depend on both contact time and adsorbent dosage, whilst increasing initial analyte concentration generally resulted in reduced removal efficiency due to saturation of the available adsorption sites. Isotherm analysis revealed that the adsorption process followed the Langmuir model, suggesting monolayer adsorption. The enhanced performance of the activated kaolin is attributed to physicochemical changes induced by acid treatment, including the removal of metal ions and increased surface reactivity. From an economic perspective, the use of abundant locally available kaolin, combined with a relatively simple activation process, highlights the potential of the material as a cost-effective alternative to conventional adsorbents. Overall, this study provides new insights into the structure-performance relationship of acid-activated kaolin and supports its application as a sustainable adsorbent for environmental remediation.

Keywords: kaolin; activation; adsorption efficiency; recycling; used automotive oil; isotherm; economic feasibility

1. Introduction

In Ghana, used automotive oils (UAO) are neither sustainably recycled nor extensively utilised in industry. Consequently, the volume of UAO accumulates rapidly, creating liquid waste and associated disposal challenges. Mismanagement of UAO adversely affects the environment, public health, the economy, and social well-being[1]. Moving machine parts is considered the major source of metal contamination in used lubricating oil because of wear and tear of metallic components. Additionally, deteriorated additives, dirt, water, and other contaminants contribute to the impurity load in UAO[2]. Metal contaminants, particularly heavy metals, are toxic and pose serious environmental risks. Therefore, the mismanagement of UAO in Ghana may have national, regional, and even global consequences, as contaminants can enter food chains and bioaccumulate in living organisms. Exploring environmentally friendly materials such as clay adsorbents[3] for converting waste oil into a reusable resource can provide significant environmental, economic, and public health benefits.

Kaolin, a type of clay found in many parts of Ghana, has attracted considerable research interest because of its numerous applications[4–6]. It is a hydrated aluminium silicate containing both kaolinite and quartz crystalline phases. The kaolinite group is classified as a 1:1 layer silicate consisting of a tetrahedral silica (SiO_2) sheet linked through oxygen atoms to an octahedral alumina (Al_2O_3) sheet[7]. Kaolinite possesses high chemical stability, low swelling capacity, and low cation-exchange capacity.

Structurally, the kaolinite group is divided into dioctahedral and trioctahedral minerals. The estimated reserves of kaolin in Ghana exceed 100 million tonnes. In terms of economic potential, Ghanaian kaolin is estimated to generate between US\$1 million and US\$5 million annually, based on its dominant domestic use and limited export of processed products [8].

Globally, clay minerals have been applied in a wide range of fields, including industrial processing, personal care products, petroleum exploration and recovery, petrochemical refinery, and medical applications[9, 10]. Their extensive use is attributed to unique characteristics such as pore size, particle morphology, surface chemistry, and other physicochemical properties, which can be modified through activation and functionalisation processes[11]. In Ghana, kaolin has been utilised in diverse ways[6, 12]. Traditionally, it has been used in geophagy, whilst contemporary applications include pottery, ceramics, construction materials, purification processes, and various industrial applications[8]. Kaolin has also been used in the palm oil industry as a bleaching agent[13], demonstrating its potential for oil clarification and purification. Previous studies characterising kaolin samples from different regions of Ghana have demonstrated that certain deposits possess properties suitable for electro-porcelain ceramic production[14]. Consequently, systematic profiling and enhancement of the adsorptive properties of Ghanaian kaolin could generate valuable information to support informed selection of materials for specific industrial applications. Such developments could significantly enhance the market value of kaolin and create employment opportunities through expanded utilisation in petroleum refining [11, 15] and the remediation of heavy metal contamination[2, 16, 17]. Various activation techniques, including treatment with alkaline and acidic solutions, have been employed to improve the adsorptive properties of kaolin[18–21]. Alkaline activation converts kaolin into soluble silicate and aluminate species and promotes the formation of K-F zeolite. However, this process does not significantly increase surface area or porosity[21, 22] . In contrast, acid activation has been shown to improve porosity and surface area through the leaching of metal ions, thereby producing kaolin with enhanced adsorption capacity [19, 21].

The objective of this study was to investigate the potential of acid-activated, locally sourced kaolin as an effective, low-cost, and environmentally friendly adsorbent for the removal of metal contaminants from UAO, thereby establishing its suitability as a sustainable material for environmental remediation. Previous studies demonstrated that activated Ghanaian clay could remove metal contaminants from UAO following acid pretreatment of the oil [4]. In the present study, Ghanaian kaolin was subjected to acid activation using varying acid concentrations to optimise its adsorption properties. UAO, an important environmental pollutant, was employed as a model sample to evaluate the purification potential of the acid-treated kaolin. This work contributes to a deeper understanding of the adsorption behaviour of acid-activated kaolin in UAO recycling through the monitoring of elemental changes in both kaolin and oil samples using Instrumental Neutron Activation Analysis (INAA), together with

confirmation of the crystalline characteristics of the clay samples using X-ray diffraction (XRD).

2. Materials and Methods

2.1 Sources of Materials and Equipment Used

The kaolin samples used in this study were purchased from the kaolin market centre at Busia Junction, Odorkor, Accra, Ghana. Used automotive oil (UAO) was obtained from the Ghana Oil Company (GOIL) automobile service station located at Kwabenya Atomic Down, Ghana. Most of the laboratory work was conducted at the Ecological Laboratory (ECOLAB), University of Ghana (UG). Elemental analyses were performed using the Instrumental Neutron Activation Analysis (INAA) facility at the Ghana Research Reactor-1 (GHARR-1), Ghana Atomic Energy Commission (GAEC). X-ray diffraction (XRD) analyses were carried out using a SmartLab X-ray diffractometer (Rigaku Co., Japan) at the Department of Engineering, University of Ghana. Analytical-grade sulphuric acid (H_2SO_4) was supplied by the GAEC Chemistry Laboratory and used without further purification. Working standards of Pb, Na, and Fe were prepared by diluting a concentrated stock solution (Merck, Darmstadt, Germany) of $1000 \pm 4 \text{ mg L}^{-1}$ with 0.25 mol L^{-1} nitric acid.

2.2 Sample Preparation

2.2.1 Kaolin Sample Pretreatment

All apparatus and glassware used for kaolin preparation were washed with aqua regia, rinsed with deionised water, and dried in an oven. The kaolin samples were crushed, air-dried, ground in a porcelain mortar, and sieved to remove unwanted materials. The powdered kaolin samples were washed with distilled water to remove water-soluble impurities before being dried in an oven overnight at 80°C . The oven-dried kaolin samples were pulverised again and sieved through a $500 \mu\text{m}$ mesh. The sieved samples were transferred into labelled airtight vials and stored in a desiccator until further use.

2.2.2 Kaolin Acid Activation Process

The kaolin acid activation procedure was based on previously reported protocols[4, 22], with a few modifications. Approximately 20 g of pretreated kaolin powder was weighed into each of four reaction beakers labelled K_0 , K_1 , K_3 , and K_5 . The designation K_0 represents raw kaolin, which received no acid treatment. The designations K_1 , K_3 , and K_5 represent kaolin samples treated with H_2SO_4 solutions of concentrations 1, 3, and 5 M respectively. A volume of 100 mL of the appropriate dilute H_2SO_4 solution was added to each reaction beaker containing 20 g of kaolin powder. For the raw kaolin

sample (K_0), 100 mL of distilled water was added instead. All four beakers containing kaolin suspensions were heated on a hot plate at 50 °C for 30 minutes while being continuously stirred using a magnetic stirrer. After heating, the suspensions were allowed to cool and settle before decantation. The treated kaolin samples were subsequently washed with distilled water until the pH of the supernatant reached a range of 4 – 5. The washed samples were then dried in an oven at 95 °C to constant weights, which required approximately three hours of continuous drying. Both the acid-treated and untreated kaolin samples were pulverised, sieved through a 500 µm mesh, transferred into labelled airtight vials, and stored for adsorption studies, waste oil purification experiments, elemental analysis using INAA, and XRD characterisation.

2.3 Physicochemical Characterisation of Kaolin

2.3.1 Elemental Characterisation Using INAA

The GHARR-1 INAA facility used for elemental analyses was equipped with a pneumatic transfer system operating at an air pressure of 600 Pa for transferring samples into the reactor. The reactor operated at 17 kW with an average thermal neutron flux of $4.63 \times 10^{11} \text{ n cm}^{-2} \text{ s}^{-1}$ at the gold comparator position. Irradiation times were selected according to radionuclide half-life characteristics as follows: 1 minute for short-lived elements, 2 hours for medium-lived elements, and 5 hours for long-lived elements. Following irradiation, samples considered for short-lived element analysis were allowed to decay for 5 minutes and were subsequently counted for 600 seconds. Samples considered for medium-lived element analysis were allowed to decay for 48 hours and counted for 15 minutes, whereas samples considered for long-lived element analysis were allowed to decay for 7 days and counted for 2 hours. Approximately 0.15 g of each sample and control material was heat-sealed in labelled polyethylene vials. The vials were packed into irradiation capsules and transferred to the irradiation site. For short-lived radionuclides, samples were irradiated sequentially. Gamma emissions from neutron-activated samples were measured using a PC-based gamma-ray spectrometry system consisting of an ORTEC p-type High Purity Germanium (HPGe) detector (Model GEM-C5970) coupled to a multichannel analyser through appropriate electronic modules and a spectroscopy amplifier. The detector had a relative efficiency of 40% and an energy resolution of 1.8 keV at the 1.33 MeV gamma-ray energy of ^{60}Co . Qualitative identification of gamma-ray-emitting radionuclides was carried out using Gamma Vision software, while quantitative analysis was performed using the k_0 -method (single-comparator method) implemented through the k_0 -IAEA spectrum analysis software. Appropriate cooling times were selected to ensure that detector dead time remained below 10%.

2.3.2 Adsorption Studies

Preliminary adsorption studies were conducted to compare the performance of the various activated kaolin samples as potential adsorbents. Thermodynamic and kinetic investigations, which are well established [23, 24], were not included because of limitations in time and resources. During the adsorption experiments, temperature, pH, and contact time were carefully controlled to minimise their influence on the preliminary adsorption results. As proof-of-concept experiments, aqueous analyte solutions of known concentrations were prepared from standard solutions to provide model systems for adsorption studies. Accordingly, adsorption characteristics such as contact time, adsorbent dosage, adsorption capacity, and initial analyte concentration were considered sufficient for evaluating adsorption performance. The data generated were subsequently used for adsorption isotherm studies.

2.3.3 Effect of Contact Time on Adsorption

To determine the time required for analyte molecules to be effectively adsorbed onto the adsorbent surface, equilibrium times were evaluated using 3 mg L⁻¹ Fe standard solutions and 0.5 g of kaolin adsorbent. Each kaolin sample was weighed into a 250 cm³ conical flask, and 100 cm³ of analyte solution was added. The mixtures were stirred for predetermined time intervals (0, 5, 10 minutes, etc.), after which they were centrifuged and the concentration of Fe in the supernatant solution was determined. The quantity of analyte adsorbed was calculated from the difference between the initial analyte concentration and the concentration remaining in the supernatant.

2.3.4 Effect of Adsorbent Dosage

The effect of adsorbent dosage on analyte removal was investigated by varying the mass of adsorbent used. Initially, 10 mg of adsorbent was added to a 250 cm³ conical flask containing 50 cm³ of a 5 mg L⁻¹ Fe solution. The adsorbent–analyte mixture was stirred for 15 minutes and subsequently centrifuged. The concentration of Fe remaining in the supernatant was then determined. The experiment was repeated by increasing the adsorbent mass in increments of 10 mg up to a maximum of 80 mg, whilst maintaining a constant analyte concentration. The amount of analyte adsorbed was determined from the reduction in analyte concentration relative to the initial concentration.

2.3.5 Effect of Initial Analyte Concentration

To evaluate the influence of initial analyte concentration on adsorption capacity, 50 mg of each adsorbent was weighed into separate 250 cm³ flasks. Each adsorbent sample was treated with 100 cm³ of aqueous analyte solution containing either Na or Pb at an initial concentration of 5 mg L⁻¹. The mixtures were stirred for 15 minutes, centrifuged,

and the concentration of analyte remaining in the supernatant was determined. The procedure was repeated using analyte concentrations up to 40 mg L⁻¹. The amount of analyte adsorbed per unit mass of kaolin was calculated and subsequently used in the determination of equilibrium adsorption characteristics and adsorption isotherms.

2.3.6 Adsorption Isotherms

Adsorption isotherm studies were conducted using data obtained from experiments investigating the effect of initial analyte concentration on adsorption capacity. Both the Langmuir and Freundlich isotherm models were applied to elucidate the adsorption mechanisms governing interactions between the adsorbate and the adsorbent[25–27]. The Langmuir model assumes monolayer adsorption onto a homogeneous surface possessing a finite number of identical adsorption sites. The Freundlich isotherm model describes adsorption on heterogeneous surfaces and multilayer adsorption processes. The goodness-of-fit of each isotherm model was assessed using the coefficient of determination (R^2). Comparison of the Langmuir and Freundlich models provided insight into whether adsorption occurred predominantly through homogeneous monolayer adsorption or heterogeneous multilayer adsorption. This dual-model approach facilitated a more comprehensive understanding of the mechanisms governing adsorbate–adsorbent interactions.

2.4 Used Automotive Oil Recycling Process

To evaluate the contaminant adsorption properties of kaolin, 0.1 g of powdered kaolin sample was added to a beaker containing 100 mL of preheated (50 °C) UAO. The kaolin–UAO suspension was agitated using a magnetic stirrer for 15 minutes and subsequently centrifuged. The treated oil samples were transferred into labelled airtight containers and stored for INAA analysis.

2.4.1 Elemental Characterisation of Fresh, Used, and Kaolin-Treated Oil Samples

To determine heavy metal concentrations in the various oil samples, a previously weighed and labelled filter paper (4 cm diameter) was spiked with 300 µL of oil sample. Both spiked and unspiked (control) filter papers were placed in tarred and labelled beakers and reweighed. The samples were covered and stored overnight in a desiccator. The prepared samples were packed into irradiation capsules, cap-sealed, heat-sealed, and analysed using the INAA procedure described previously. To validate the analytical method, the IAEA Worldwide Open Proficiency Test Sample for Nuclear and Related Analytical Techniques Laboratories (PTNAT-IAEA19) certified clay reference material was analysed alongside the samples[28]. Percentage recoveries were subsequently calculated to assess analytical accuracy.

2.5 X-ray Diffraction Analysis of Kaolin

X-ray diffraction analysis was performed using a Rigaku SmartLab SE multipurpose X-ray diffractometer with a step size of 0.01° . The instrument was equipped with a D/teX Ultra 250 detector and operated using Cu $K\alpha$ radiation ($\lambda = 1.54059 \text{ \AA}$), with a nickel filter for $K\beta$ removal, a tube voltage of 40 kV, and a current of 50 mA. Prior to analysis, the kaolin samples were ground into fine powders and packed into steel sample holders for XRD measurement.

3. Results and Discussion

3.1 Characterisation of Kaolin

3.1.1 INAA Elemental Characterisation of Kaolin

This study presents a comprehensive investigation into the effect of H_2SO_4 treatment on the optimisation of Ghanaian kaolin for environmental remediation applications. The research examined how varying H_2SO_4 concentrations (0, 1, 3, and 5 M) modified the structural properties of kaolin and enhanced its adsorption capabilities for the recycling of UAO[17, 29]. The acid-treated kaolin samples (K_1 , K_3 , and K_5) and the untreated sample (K_0) were analysed to determine their elemental compositions before and after acid treatment using INAA. The various oil samples were also

Table 1. Elemental composition (%) of raw and acid-treated kaolin samples (values represent means of $n = 3$ repetitions and all repeated measurements varied by less than 5%)

Element	K_0 (Raw Kaolin)	K_1 (1 M)	K_3 (3 M)	K_5 (5 M)
Al	8.7800	8.4200	8.2000	7.5600
Fe	3.2100	1.9200	1.2000	1.0000
K	2.9400	2.9100	2.9000	2.9000
Ti	0.6700	0.5400	0.2400	0.0300
Ca	0.0400	0.0100	0.0030	0.0030
Na	0.0582	0.0565	0.0563	0.0438
Mn	0.0028	0.0027	0.0026	0.0026
Ce	0.0082	0.0076	0.0071	0.0062

analysed to determine their elemental contaminant profiles before and after the recycling process. Table 1 presents the concentrations of the metals detected in the various kaolin samples. The concentrations of metals in the raw kaolin samples were consistently higher than those measured in the acid-treated samples, indicating that treatment with H_2SO_4 promoted the leaching of metal ions from the kaolin matrix. In

general, the loss of metals increased with increasing acid concentration (from 1 to 5 M) for all elements analysed.

These observations are consistent with previous studies demonstrating that acid treatment of kaolin reduces the concentration of metal constituents and decreases the density of the clay structure[18, 30–32]. It was also observed that different metals were leached at different rates. Following treatment with increasing concentrations of H_2SO_4 , titanium exhibited the greatest degree of decrease, with approximately 96% of the original Ti content being lost after treatment. In contrast, potassium showed the least susceptibility to leaching, with only about 1.3% of its original concentration removed after treatment with 5 M H_2SO_4 . Based on the degree of removal observed, the relative rates of metal loss from the kaolin during acid treatment can be ranked as follows: Ti > Ca > Fe > Na > Ce > Al > Mn > K. This trend demonstrates the selective dissolution behaviour of different metal species during acid activation and highlights the significant physicochemical modifications induced by sulphuric acid treatment.

3.1.2 Adsorption Studies

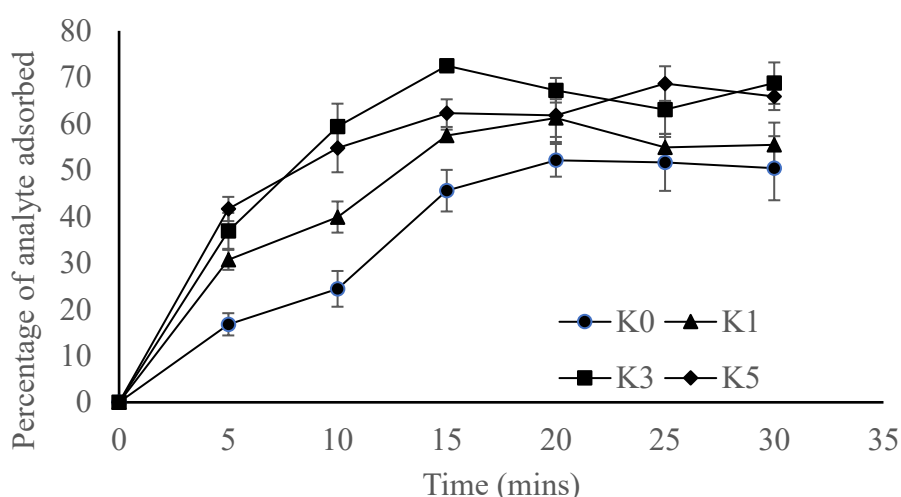


Fig. 1.

Effect of contact time on the percentage of analyte adsorbed by the four kaolin samples. K_0 represents raw kaolin, whilst K_1 , K_3 , and K_5 represent kaolin samples treated with 1 M, 3 M, and 5 M H_2SO_4 , respectively, (values represent means of $n = 4$ repetitions)

Preliminary adsorption studies were conducted to compare the adsorption capacities of the treated kaolin samples and to determine the equilibrium contact time between the adsorbent and analyte. The results showed that the adsorption capacity increased with increasing contact time, indicating progressive interaction between analyte molecules and the adsorption sites on the kaolin surface. Adsorption increased up to approximately 15 minutes, after which equilibrium appeared to be reached between

15 and 20 minutes (Fig. 1). Interestingly, all three acid-treated kaolin samples exhibited greater adsorption capacities than the untreated kaolin. At equilibrium, the kaolin sample activated with 3 M H_2SO_4 demonstrated the highest adsorption capacity. This observation is consistent with previous studies reporting improved adsorption performance following acid activation of kaolin[20].

To determine the optimum quantity of adsorbent required for efficient analyte removal, the adsorbent dosage was systematically increased. As shown in Fig. 2, the percentage of analyte removed from the 5 mg L^{-1} solution increased with increasing adsorbent mass by 10 mg increment up to approximately 50 mg. Beyond 50 mg, further increases in adsorbent dosage did not result in corresponding increases in analyte removal. The observed increase in adsorption with increasing adsorbent dosage can be attributed to the greater number of available adsorption sites. However, at higher dosages, adsorption efficiency tends to plateau because of factors such as underutilisation of adsorption sites, particle aggregation, and reduction in the concentration gradient driving adsorption. This behaviour is characteristic of adsorption systems and is consistent with observations reported in previous studies[16, 33].

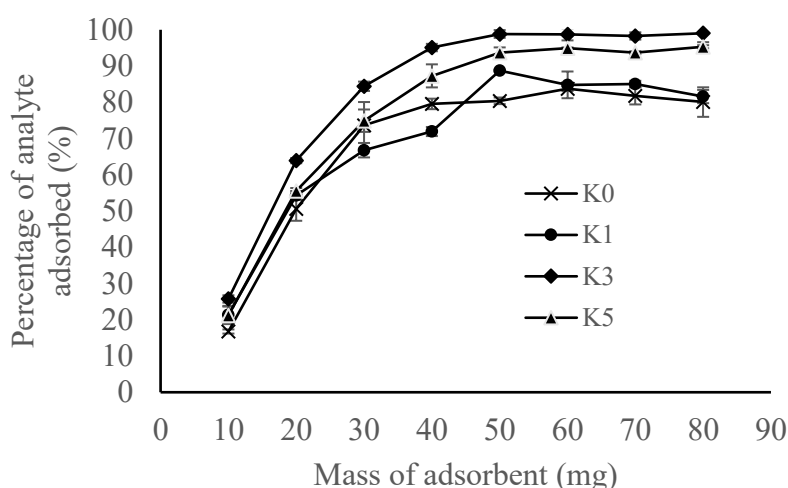


Fig.2.

Effect of adsorbent dosage on the adsorption performance of the four kaolin samples. K_0 represents raw kaolin, whilst K_1 , K_3 , and K_5 represent kaolin samples treated with 1 M, 3 M, and 5 M H_2SO_4 , respectively (values represent means of $n = 4$ repetitions)

The influence of initial analyte concentration on adsorption behaviour was investigated by systematically increasing analyte concentration while maintaining constant adsorbent mass and contact time. Adsorption is fundamentally an equilibrium process. Higher initial analyte concentrations provide a larger number of analyte species competing for available adsorption sites on the adsorbent surface, thereby influencing equilibrium conditions. As shown in Table 2, the percentage of analyte removed decreased as the initial analyte concentration increased for a fixed quantity of

adsorbent. This observation confirms that the adsorption capacity of a given mass of adsorbent is limited by the number of available active sites. The results are consistent with trends observed in the present study and reported elsewhere[33, 34], demonstrating that enhancement of kaolin active sites through acid activation improves adsorption performance.

Table 2. Effect of initial analyte concentration on adsorption efficiency of raw and acid-treated kaolin (values are in percentages of analyte adsorbed and represent means of $n = 4$ repetitions)

Concentration (mg/L)	K_0 (Na)	K_3 (Na)	K_0 (Pb)	K_3 (Pb)
5	73 ± 2	85.4 ± 0.4	72 ± 3	98 ± 1
10	70 ± 1	86.5 ± 0.6	69 ± 1	90 ± 1
15	64.1 ± 0.5	87.0 ± 0.4	64.2 ± 0.9	91.2 ± 1
20	66.7 ± 0.7	79.3 ± 0.6	64.4 ± 0.6	88.5 ± 0.6
25	60 ± 1	75.6 ± 0.3	60 ± 2	78.1 ± 0.1
30	58.9 ± 0.5	70.9 ± 0.5	56 ± 6	75.9 ± 0.9
35	54.1 ± 0.3	65.7 ± 0.3	55.7 ± 0.4	70 ± 5
40	50 ± 1	63.8 ± 0.3	50.2 ± 0.7	64 ± 7

3.1.3 Adsorption Isotherm Studies

Adsorption isotherm studies were conducted using data obtained from experiments involving variation of initial analyte concentrations. Both the Freundlich and Langmuir adsorption models were applied to evaluate adsorption behaviour. The Freundlich isotherm model was used to assess adsorption on heterogeneous surfaces and to evaluate adsorption intensity for Na and Pb under K_0 and K_3 conditions. The linear form is stated in Equation 1 as follows:

$$\log q_e = \log K_f + \frac{1}{n} \log C_e \quad \text{Equation 1}$$

where q_e is the amount of adsorbate at equilibrium (mg/g), C_e is the equilibrium concentration of the adsorbate on the adsorbent, K_f indicates adsorption capacity (L/mg), and $1/n$ is the heterogeneity factor reflecting adsorption intensity. Freundlich constants K_f and n were obtained from plots of $\log q_e$ against $\log C_e$. The calculated n values (1.53–2.14) indicate favourable adsorption behaviour across all systems investigated[35]. Furthermore, K_f values increased from K_0 to K_3 (i.e., Na: 6.68–13.67 mg g⁻¹; Pb: 6.37–17.65 mg g⁻¹) indicating enhanced adsorption capacity following acid-induced surface modification (Supplementary Material SMF1 and SMT1). The higher K_f values obtained for Pb indicate a stronger affinity of Pb ions for the adsorbent surface, most likely due to stronger surface interactions. The Freundlich model

provided a reasonable description of the adsorption process, with R^2 values ranging from 0.8396 to 0.9828, particularly for the K_0 systems. However, the lower correlation coefficients obtained for K_3 systems suggest deviations from ideal heterogeneous adsorption behaviour, implying that acid treatment modified the surface characteristics of the adsorbent[36]. The Langmuir adsorption model was subsequently applied under the assumption that adsorption proceeds through monolayer coverage of the adsorbent surface. The logarithmic form of Langmuir model is shown in Equation 2 as follows:

$$\frac{1}{q_e} = \frac{1}{q_{max}} + \frac{1}{K_L q_{max} C_e} \quad \text{equation 2}$$

where q_{max} represents the maximum monolayer adsorption capacity (mg/g), and K_L is the Langmuir constant related to the affinity of binding systems (L/mg).

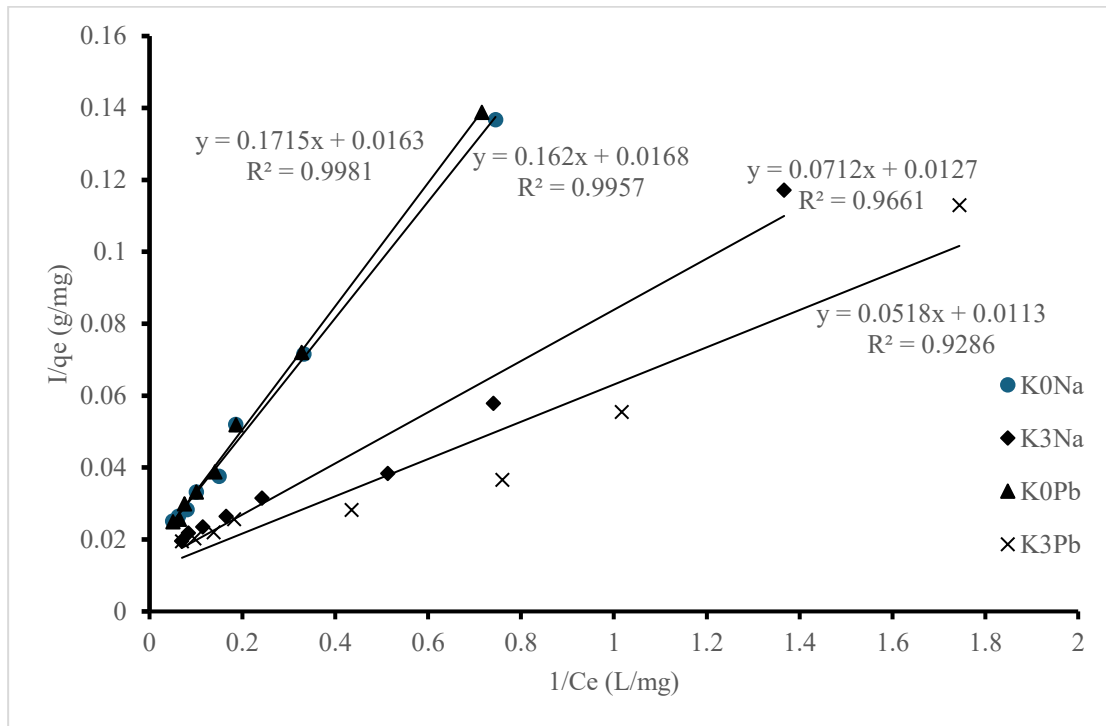


Fig. 3.

Langmuir isotherm plots for Na and Pb adsorption using raw and acid-treated kaolin adsorbents. K_0 Na and K_0 Pb represent adsorption of Na and Pb on raw kaolin, whilst K_3 Na and K_3 Pb represent adsorption on kaolin activated with 3 M H_2SO_4 , (values represent means of $n = 4$ repetitions)

The Langmuir model provided an excellent fit to the experimental data, with R^2 values ranging from 0.9287 to 0.9980, outperforming the Freundlich model and indicating that adsorption was predominantly governed by monolayer surface coverage. The

Langmuir parameters q_{max} and K_L were determined from the linearised Langmuir plots.

Table 3. Langmuir adsorption parameters for raw and acid-treated kaolin

Metal	Acid-Treated Kaolin q_{max} (mg/g)	K_L (L/mg)	R^2	Raw Kaolin q_{max} (mg/g)	K_L (L/mg)	R^2
Pb	87.7193	0.2213	0.9287	60.9756	0.2213	0.9980
Na	78.7402	0.1786	0.9660	59.5238	0.1039	0.9958

The Langmuir model assumes that adsorption occurs through formation of a monolayer of analyte molecules on the adsorbent surface. Adsorbate species are transferred from the liquid phase to discrete adsorption sites on the solid surface until equilibrium is reached and a monolayer is formed. The correlation coefficients obtained indicated that adsorption of metal ions by kaolin followed the Langmuir model. The maximum adsorption capacities obtained for both analytes demonstrated that acid-treated kaolin possessed greater adsorption capacity than raw kaolin. Previous studies have similarly reported that acid activation increases the number of adsorption sites through the removal of metal ions from the clay structure[20, 30]. The values obtained for maximum adsorption capacity are comparable to those reported in previous investigations[37, 38]. Similarly, the increase in K_L values under K_3 conditions indicated stronger adsorbate–adsorbent interactions following acid activation. Lead consistently exhibited higher q_{max} values than sodium, indicating stronger binding and greater affinity for the modified adsorbent surface. The favourability of adsorption was further assessed using the dimensionless separation factor (R_L) [39]. The R_L values obtained (0.102–0.676) (SMT2) fall within the range: $0 < R_L < 1$ which confirms favourable adsorption over the concentration range investigated (5–40 mg L⁻¹). A decreasing R_L trend with increasing analyte concentration indicates improved adsorption favourability at higher concentrations. The lower R_L values observed for K_3 -treated systems, particularly for Pb adsorption, further confirmed the enhanced adsorption efficiency resulting from acid activation. Overall, the comparative analysis demonstrated that the Langmuir model provided a better description of the adsorption process than the Freundlich model, indicating that adsorption occurred predominantly through monolayer coverage on relatively homogeneous active sites. The enhanced adsorption capacity (q_{max}), increased adsorption affinity (K_L), and lower separation factor (R_L) values observed for K_3 collectively demonstrated superior adsorption performance. The consistently higher adsorption of Pb relative to Na further suggested that the modified kaolin is particularly effective for the removal of heavy metal contaminants. From an environmental perspective, these findings highlight the potential application of acid-activated Ghanaian kaolin as an efficient and sustainable adsorbent for the removal of toxic metal ions from contaminated systems.

3.2 Recycling of Used Automotive Oil Using Kaolin

3.2.1 Elemental Contaminants in Fresh, Used, and Kaolin-Treated Oil

Table 4 presents the elemental concentrations determined in the various oil samples. Fresh, unused lubricating oil served as the baseline for evaluating elemental changes resulting from engine use and subsequent treatment. Seven elements were detected in the fresh oil sample. The dominant elements were Ca, Zn, and Na, whilst Br, Co, Mo, and La were present at comparatively lower concentrations. In the UAO, both the number of detectable elements and their concentrations increased substantially. Five additional elements, namely Cs, Cr, Sc, Th, and U, were detected, bringing the total number of identified elements to twelve. The increase in both elemental diversity and concentration confirms contamination of the lubricating oil during use in automotive engines. The percentage increase in elemental concentrations ranged

Table 4. Elemental composition of fresh oil, used automotive oil (UAO), and kaolin-treated oil samples (*values represent means of $n = 3$ repetitions and all repeated measurements varied by less than 5%*)

Element (mg/L)	Fresh Oil (V_0)	UAO	K_0	K_1	K_3	K_5
Na	68.28	224.50	67.10	57.20	43.88	75.93
Ca	4301	26300	6820	6212	4894	6988
Sc	–	0.05	0.040	0.03	0.01	0.03
Cr	–	7.17	3.00	1.94	1.01	3.03
Co	2.19	6.92	1.79	1.53	1.31	2.54
Zn	332.60	773.50	68.78	62.80	44.20	82.04
Br	1.53	3.85	1.33	1.09	0.99	1.66
Mo	8.97	23.96	2.35	2.25	1.36	3.63
Cs	–	0.37	0.005	BDL	BDL	0.30
La	0.08	0.30	0.14	0.11	0.10	0.15
Th	–	0.19	0.11	0.06	0.03	0.17
U	–	0.16	0.001	BDL	BDL	0.005

BDL = Below Detection Limit; – = Not detected

from 132% to 511%, with Ca exhibiting the greatest increase and Zn showing the smallest increase. Treatment of the UAO with raw and acid-activated kaolin resulted in varying degrees of reduction in elemental concentrations (Table 4). For example, the concentration of calcium, which increased to 26,300 mg/L in the UAO, decreased to values ranging from 4,894 to 6,988 mg/L following treatment with the different kaolin adsorbents. Compared to untreated UAO, approximately 81% of the calcium was removed when the oil was treated with the 3 M acid-activated kaolin sample (K_3). Overall, the results indicate that acid-activated kaolin adsorbents were more effective in removing metal contaminants from UAO than raw kaolin, with the K_3 adsorbent exhibiting the highest removal efficiency among the samples investigated.

Although kaolin is naturally adsorptive, it was anticipated that treatment with suitable acid concentrations would enhance its adsorption performance. Acid activation promotes the leaching of metal ions from the clay structure, thereby increasing surface area and creating additional active adsorption sites[31, 39, 40]. As demonstrated in Table 1, the kaolin sample treated with 5 M H_2SO_4 exhibited the greatest degree of metal-ion removal, indicating the formation of a larger number of potential adsorption sites. However, despite this extensive leaching, the K_3 sample exhibited the highest contaminant-removal efficiency. This observation suggests that treatment with 1 M H_2SO_4 may not have been sufficiently aggressive to generate an optimum number of active adsorption sites under the conditions employed. Conversely, treatment with 5 M H_2SO_4 appears to have caused excessive leaching, leading to partial collapse of the crystalline structure of the kaolin [40].

3.2.2 X-ray Diffraction Analysis of Kaolin

To further investigate the effect of acid treatment on structural integrity, XRD analyses were performed on all kaolin samples. Previous studies have established that kaolin typically contains two major crystalline phases: kaolinite ($Al_2O_3 \cdot 2SiO_2 \cdot 2H_2O$) and quartz (SiO_2). The kaolinite phase is characterised by diffraction peaks at approximately 12.4° (strong), 20.8° (weak), 24.9° (strong), and several weaker peaks between 35° and 65° 2θ . Quartz is characterised by a moderate peak near 21° and a strong peak at approximately 26.6° 2θ [20, 38, 41].

In the present study, the XRD spectra (Fig. 4) revealed extremely weak peaks at approximately 12° 2θ and moderate peaks at 20.8° 2θ , whilst the peak expected near 25° 2θ was absent. Moderate peaks observed near 35° , 40° , and 55° 2θ indicate the presence of kaolinite. The quartz phase is represented by a strong peak near 27° 2θ and a moderate peak near 21° 2θ . Although the characteristic kaolinite peaks at 12° and 25° 2θ were not prominent, this observation is consistent with previous reports showing that kaolin from certain regions of Ghana exhibits similar diffraction behaviour[12, 14]. Furthermore, the elemental analysis results (Table 1) confirmed the presence of aluminium, providing additional evidence for the existence of a kaolinite phase.

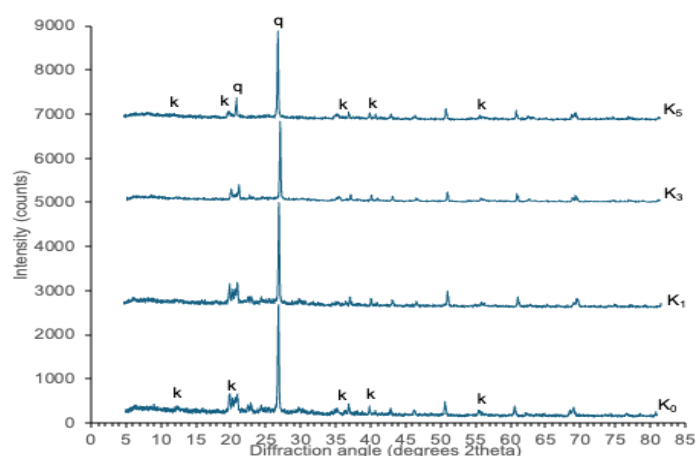


Fig. 4.

XRD spectra of kaolin samples treated with different concentrations of H_2SO_4 . K_0 represents raw kaolin, whilst K_1 , K_3 , and K_5 represent kaolin treated with 1 M, 3 M, and 5 M acid, respectively. Peaks assigned “k” correspond to kaolinite and those assigned “q” correspond to quartz. The vertical axis has been offset for clarity.

Table 5. Compositional and structural parameters of kaolinite–quartz composites as a function of H_2SO_4 concentration

Parameter	Raw Kaolin	1 M	3 M	5 M
Kaolinite1A (wt.%)	38.92	37.04	34.29	33.67
Quartz (wt.%)	61.08	62.96	65.71	66.33
Rwp	15.40	16.75	17.98	17.89
Rexp	14.52	14.25	14.57	14.41
χ^2	1.1249	1.3817	1.5229	1.5413
GoF	1.0606	1.1754	1.2340	1.2415

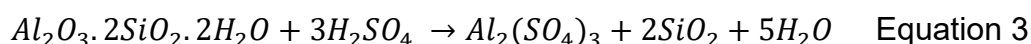
Note: All compositional values are expressed as weight percentages (wt.%). Rwp and Rexp represent refinement reliability factors, whilst χ^2 and GoF represent statistical indicators of refinement quality.

Following acid treatment, noticeable changes were observed in the diffraction patterns, particularly around 12° and 20.8° 2θ . The peak at approximately 12° 2θ became progressively weaker, whilst a pronounced shoulder developed near 20.8° 2θ (Fig. 4). Similar spectral modifications have been reported previously and are

indicative of structural alteration of the kaolinite phase[30]. These structural changes are consistent with the elemental analysis results, which showed that aluminium content decreased from 8.78% in raw kaolin to 7.56% following treatment with 5 M H₂SO₄. In contrast, the quartz peak at approximately 27° 2θ remained relatively unchanged throughout the activation process, indicating that quartz was largely unaffected by acid treatment.

Quantitative XRD analysis indicated that the raw kaolin consisted of approximately 38.92 wt.% kaolinite and 61.08 wt.% quartz (Table 5), indicating that quartz is the dominant mineral phase in the sample. The relatively low intensity of kaolinite peaks suggests that a substantial proportion of the kaolinite may exist in a poorly crystalline or partially amorphous form. Systematic changes were observed in the refinement parameters with increasing acid concentration. For example, the Goodness of Fit (GoF) increased from 1.0606 for the untreated sample to 1.2415 for the 5 M acid-treated sample. Similarly, phase evolution analysis demonstrated a gradual reduction in kaolinite content from 38.92 wt.% in the raw sample to 33.67 wt.% following treatment with 5 M H₂SO₄. This decrease was accompanied by a corresponding increase in quartz content from 61.08 wt.% to 66.33 wt.%.

The acidification of kaolinite can be represented by Equation 2.



Previous studies have shown that acid activation promotes the dissolution of aluminium and other metal ions from the kaolinite structure, resulting in a reduction in kaolinite content and a relative increase in quartz proportion [30, 40, 42][33].

At moderate acid concentrations (approximately 3 M), the Al–O–Si framework remains sufficiently intact to preserve the octahedral structure of kaolinite. However, at higher acid concentrations (5 M or greater), rapid dissolution of aluminium compromises the octahedral layer and weakens structural integrity. In the present study, XRD analysis revealed peak broadening near 20.8° 2θ and peak attenuation near 12° 2θ in acid-treated samples relative to raw kaolin. These observations support the conclusion that excessive acid treatment resulted in partial collapse of the kaolinite structure, thereby reducing adsorption efficiency despite the creation of additional surface sites. Consequently, treatment with 3 M H₂SO₄ provided the optimum balance between metal-ion leaching and preservation of structural integrity. This explains why the K₃ adsorbent demonstrated superior contaminant-removal performance compared with the K₅ adsorbent. The results therefore indicate that 3 M acid activation provided optimum conditions for enhancing the adsorption properties of Ghanaian kaolin without causing significant deterioration of the kaolinite framework [40, 43, 44].

3.3 Economic Feasibility

Economic viability is a critical consideration in assessing the large-scale applicability of adsorbent materials. The principal precursor used in this study, kaolin clay, is naturally abundant and readily available in Ghana, providing a significant economic advantage compared to commercially activated adsorbents such as activated carbon. The major costs associated with the preparation of acid-activated kaolin include: 1. Sulphuric acid consumption, 2. Thermal energy requirements, 3. Water usage during washing, and 4. Drying operations.

Among these factors, acid consumption represents the most significant expenditure. The identification of 3 M H_2SO_4 as the optimum activation concentration is particularly important because it balances adsorption performance with chemical consumption, thereby improving overall cost efficiency. Compared to conventional adsorbents, the acid-activated kaolin developed in this study offers several economic advantages: 1. Minimal raw-material cost, 2. A relatively simple preparation procedure, and 3. Elimination of energy-intensive activation processes such as high-temperature carbonisation. Furthermore, the enhanced adsorption performance achieved through acid activation suggests that smaller quantities of adsorbent may be required to achieve equivalent contaminant-removal efficiencies, thereby reducing operational costs.

Although a detailed techno-economic assessment was beyond the scope of this study, the findings strongly suggest that acid-activated Ghanaian kaolin represents a promising low-cost and sustainable adsorbent for the treatment of contaminated waste streams such as used automotive oil. Future studies should incorporate comprehensive cost modelling and life-cycle assessment to further evaluate the economic and environmental sustainability of the technology.

4. Conclusion

This study demonstrates that acid activation significantly enhances the adsorption behaviour of kaolin obtained from a commercial market in Ghana, thereby reinforcing its potential as a low-cost and sustainable material for the recycling of UAO. The results indicate that activation with H_2SO_4 effectively modifies the physicochemical properties of kaolin, leading to improved adsorption efficiency. Among the activation conditions investigated, treatment with 3 M H_2SO_4 provided the optimum balance between structural modification and preservation of active adsorption sites. The adsorption process was strongly influenced by operational parameters such as contact time, adsorbent dosage, and initial analyte concentration, with the observed trends being consistent with adsorption-site availability and saturation effects. Adsorption isotherm modelling indicated that the adsorption process follows the Langmuir model, suggesting predominantly monolayer surface interactions. These findings provide valuable insights into the structure-performance relationship governing the adsorption process. This study further highlights the potential of locally sourced kaolin as an effective alternative to conventional adsorbents, offering both economic and

environmental advantages because of its abundance, low cost, and relatively simple activation process. The results also demonstrate the applicability of the developed material for the treatment of UAO, supporting its potential for real-world implementation. Overall, this work contributes to the growing body of knowledge on clay-based adsorbents by linking activation conditions to adsorption performance and demonstrating the optimisation of low-cost materials for environmental remediation. Future studies should focus on adsorbent reusability, advanced surface characterisation, and large-scale application in order to further validate the long-term performance and economic viability of the material.

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Conflict of Interest

The authors declare that they have no conflict of interest.

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The raw and processed data required to reproduce the findings of this study are available from the corresponding author and will be provided upon reasonable request.

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5. References

1. El-Fadel M, Khoury R (2001) Strategies for vehicle waste-oil management: a case study. *Resour Conserv Recycl* 33:75–91
2. Abdel-Jabbar NM, Al Zubaidy EAH, Mehrvar M (2010) Waste lubricating oil treatment by adsorption process using different adsorbents. *International Journal of Chemical and Molecular Engineering* 4:141–144
3. Misaelides P (2011) Application of natural zeolites in environmental remediation: A short review. *Microporous and Mesoporous Materials* 144:15–18
4. Kwao-Boateng E, Anokye-Poku T, Agyemang ANP, Fokuo MK (2022) Re-refining Used Engine Oil in Ghana Using Solvent Extraction and Acid-Clay

- Treatment. *International Journal of Chemical Engineering* 2022:6344409.
<https://doi.org/10.1155/2022/6344409>
5. Nsiah JK (2007) The study of Mfensi clay in the Ashanti Region of Ghana. *Ghana Journal of Science* 47:123–129
 6. Bediako M, Purohit SS, Kevern JT (2017) Investigation into Ghanaian calcined clay as supplementary cementitious material. *ACI Mater J* 114:889–896
 7. Brigatti MF, Galan E, Theng BKG (2006) Chapter 2 Structures and Mineralogy of Clay Minerals. In: Bergaya F, Theng BKG, Lagaly G (eds) *Developments in Clay Science*. Elsevier, pp 19–86
 8. Asamoah RB, Nyankson E, Annan E, et al (2018) Industrial applications of clay materials from Ghana-a review. *Oriental Journal of Chemistry* 34:1719
 9. Murray HH (1991) Overview—clay mineral applications. *Appl Clay Sci* 5:379–395
 10. Ekosse G-IE (2010) Kaolin deposits and occurrences in Africa: Geology, mineralogy and utilization. *Appl Clay Sci* 50:212–236.
<https://doi.org/https://doi.org/10.1016/j.clay.2010.08.003>
 11. Emam EA (2018) Clay adsorption perspective on petroleum refining industry. *Industrial Engineering* 2:19–25
 12. Onwona-Agyeman B, Lyczko N, Minh DP, et al (2020) Characterization of some selected Ghanaian clay minerals for potential industrial applications. *Journal of Ceramic Processing Research* 21:35–41
 13. Foletto EL, Colazzo GC, Volzone C, Porto LM (2011) Sunflower oil bleaching by adsorption onto acid-activated bentonite. *Brazilian Journal of Chemical Engineering* 28:169–174
 14. Yaya A, Tiburu EK, Vickers ME, et al (2017) Characterisation and identification of local kaolin clay from Ghana: A potential material for electroporcelain insulator fabrication. *Appl Clay Sci* 150:125–130
 15. Emam EA (2013) Clays as catalysts in petroleum refining industry. *ARPN Journal of Science and Technology* 3:356–375
 16. Halilu AA, Abubakar S, Muhammad YB (2019) Oil Adsorption Using Kaolinite Clay and Its Characterization. *Asian J Appl Sci Technol* 3:242–256
 17. Bhattacharyya KG, Gupta S, Sen (2008) Adsorption of a few heavy metals on natural and modified kaolinite and montmorillonite: a review. *Adv Colloid Interface Sci* 140:114–131

18. Zhou Y, Cheng H, Wei C, Zhang Y (2021) Effect of acid activation on structural evolution and surface charge of different derived kaolinites. *Appl Clay Sci* 203:105997. <https://doi.org/https://doi.org/10.1016/j.clay.2021.105997>
19. Gao W, Zhao S, Wu H, et al (2016) Direct acid activation of kaolinite and its effects on the adsorption of methylene blue. *Appl Clay Sci* 126:98–106
20. Biswas B, Islam MdR, Deb AK, et al (2023) Understanding Iron Impurities in Australian Kaolin and Their Effect on Acid and Heat Activation Processes of Clay. *ACS Omega* 8:5533–5544. <https://doi.org/10.1021/acsomega.2c06795>
21. Belver C, Bañares Muñoz MA, Vicente MA (2002) Chemical activation of a kaolinite under acid and alkaline conditions. *Chemistry of materials* 14:2033–2043
22. Wang H, Feng Q, Liu K (2016) The dissolution behavior and mechanism of kaolinite in alkali-acid leaching process. *Appl Clay Sci* 132–133:273–280. <https://doi.org/https://doi.org/10.1016/j.clay.2016.06.013>
23. Fizir M, Richa A, Touil S, et al (2024) Comparing Adsorption Properties of Algerian Kaolinite Towards Phosphate with Those of Activated Carbon: Adsorption Experiments, Molecular Modeling and an Initiative Toward Hydroponic Wastewaters Remediation. *Water Air Soil Pollut* 235:422
24. Rouibah K, Ferkous H, Abdessalam-Hassan M, et al (2024) Exploring the efficiency of Algerian Kaolinite Clay in the adsorption of Cr (III) from Aqueous solutions: Experimental and computational insights. *Molecules* 29:2135
25. Chen X (2015) Modeling of experimental adsorption isotherm data. *information* 6:14–22
26. Tran HN, You S-J, Hosseini-Bandegharaei A, Chao H-P (2017) Mistakes and inconsistencies regarding adsorption of contaminants from aqueous solutions: a critical review. *Water Res* 120:88–116
27. Foo KY, Hameed BH (2010) Insights into the modeling of adsorption isotherm systems. *Chemical engineering journal* 156:2–10
28. World Wide Proficiency Test for Nuclear and Related Analytical Techniques Laboratories PTNAT-IAEA19 (2022) Determination of major, minor and trace elements in clay a sample and in a plant sample PTATIAEA19 - 30 March 2022
29. Fialips C-I, Petit S, Decarreau A, Beaufort D (2000) Influence of synthesis pH on kaolinite “crystallinity” and surface properties. *Clays Clay Miner* 48:173–184
30. Panda AK, Mishra BG, Mishra DK, Singh RK (2010) Effect of sulphuric acid treatment on the physico-chemical characteristics of kaolin clay. *Colloids Surf A Physicochem Eng Asp* 363:98–104. <https://doi.org/https://doi.org/10.1016/j.colsurfa.2010.04.022>

31. Wahyuni N, Zaharah TA, Ria R (2021) Characterization of Hydrochloric Acid Activated Natural Kaolin and its application as adsorbent for Mg^{2+} . *J Phys Conf Ser* 1882:12099. <https://doi.org/10.1088/1742-6596/1882/1/012099>
32. Eze KA, Nwadiogbu JO, Nwankwere ET, et al (2012) Effect of acid treatments on the physicochemical properties of kaolin clay. *Arch Appl Sci Res* 4:792–794
33. Aragaw TA, Alene AN (2022) A comparative study of acidic, basic, and reactive dyes adsorption from aqueous solution onto kaolin adsorbent: Effect of operating parameters, isotherms, kinetics, and thermodynamics. *Emerg Contam* 8:59–74. <https://doi.org/https://doi.org/10.1016/j.emcon.2022.01.002>
34. Hamri N, Imessaoudene A, Hadadi A, et al (2024) Enhanced adsorption capacity of methylene blue dye onto kaolin through acid treatment: batch adsorption and machine learning studies. *Water (Basel)* 16:243
35. Ayawei N, Ebelegi AN, Wankasi D (2017) Modelling and interpretation of adsorption isotherms. *J Chem* 2017:3039817
36. Hall KR, Eagleton LC, Acrivos A, Vermeulen T (1966) Pore-and solid-diffusion kinetics in fixed-bed adsorption under constant-pattern conditions. *Industrial & engineering chemistry fundamentals* 5:212–223
37. Mouni L, Merabet D, Bouzaza A, Belkhiri L (2010) Removal of Pb^{2+} and Zn^{2+} from the aqueous solutions by activated carbon prepared from Dates stone. *Desalination Water Treat* 16:66–73
38. Tian L, Fu K, Chen S, et al (2022) Comparison of microscopic adsorption characteristics of Zn (II), Pb (II), and Cu (II) on kaolinite. *Sci Rep* 12:15936
39. Edama NA, Sulaiman A, Ku Hamid KH, et al (2014) The Effect of Hydrochloric Acid on the Surface Area, Morphology and Physico-Chemical Properties of Sayong Kaolinite Clay. *Key Eng Mater* 594–595:49–56. <https://doi.org/10.4028/www.scientific.net/KEM.594-595.49>
40. Zhang A-B, Pan L, Zhang H-Y, et al (2012) Effects of acid treatment on the physico-chemical and pore characteristics of halloysite. *Colloids Surf A Physicochem Eng Asp* 396:182–188. <https://doi.org/https://doi.org/10.1016/j.colsurfa.2011.12.067>
41. Zhao G, Zhou H, Wang Z (2010) Concentrations of selected heavy metals in food from four e-waste disassembly localities and daily intake by local residents. *Journal of Environmental Science and Health, Part A* 45:824–835. <https://doi.org/10.1080/10934521003709016>
42. Xue C, Qin Y, Fu H, Fan J (2022) Thermal stability, mechanical properties and ceramization mechanism of epoxy resin/kaolin/quartz fiber ceramifiable composites. *Polymers (Basel)* 14:3372

43. Angaji MTZAZQNT (2013) Study of physical, chemical and morphological alterations of smectite clay upon activation and functionalization via the acid treatment. *World Journal of Nano Science and Engineering*
44. Yahaya S, Jikan SS, Badarulzaman NA, Adamu AD (2017) Effects of acid treatment on the SEM-EDX characteristics of kaolin clay. *Path Sci* 3:4001–4005

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