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Reusable Nanostructured Clay for Environmental Detoxification: Adsorption of Crystal Violet Dye and Regeneration Performance

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ABSTRACT

Industrial dye pollution, particularly from textile and dyeing sectors, poses a significant threat to aquatic ecosystems and public health due to the toxic, non-biodegradable, and persistent nature of synthetic dyes. In this study, a green, regenerable nanoclay-based adsorbent was developed and evaluated for the effective removal of crystal violet (CV) dye from aqueous solutions. The nanostructured clay material was prepared via a simple physical exfoliation and chemical modification approach to enhance its surface area, active sites, and adsorption efficiency. Comprehensive structural and morphological characterizations were conducted using X-ray diffraction (XRD), field emission scanning electron microscopy (FESEM), transmission electron microscopy (TEM), and thermogravimetric analysis (TGA). The results confirmed the formation of a highly porous, thermally stable nanoclay with an expanded layered structure and a high surface area, making it suitable for dye adsorption. In the initial regeneration trials, three different activating agents—acid, base, and distilled water were evaluated for their effectiveness in washing and desorbing the adsorbed dye from the nanoclay surface. Among these, distilled water demonstrated the highest efficiency in removing the dye residues without causing structural degradation of the material. Due to its superior performance and eco-friendly nature, water was selected as the optimal regenerating agent for all subsequent activation and reusability cycles. Reusability studies were conducted over five consecutive adsorption-desorption cycles using water as a desorbing agent. The nanoclay retained over 91% of its initial adsorption efficiency after five cycles, demonstrating excellent structural integrity, regeneration capability, and long-term applicability.

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INTRODUCTION

Wastewater generated from various industries, such as textile, dyeing, printing, cosmetics, food processing, and paper manufacturing, is among the leading sources of coloured effluents. Among these, the textile industry is particularly recognized for its extensive water usage and discharge of a wide range of synthetic dyes, many

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of which are resistant to biodegradation and highly toxic. The environmental impact of such dyes on aquatic ecosystems and human health is well documented. [1, 2]. Colour is one of the most visually detectable forms of pollution and can be observed even at concentrations as low as 1.0 mg/L, rendering water aesthetically unacceptable and unsafe for consumption. The presence of dyes in wastewater not only hinders light penetration, which negatively affects aquatic photosynthesis, but also contributes to elevated levels of Chemical Oxygen Demand (COD) and Biological Oxygen Demand (BOD). Moreover, many dyes possess the ability to chelate heavy metals, resulting in micro-toxicity and bioaccumulation in aquatic organisms. From a human health perspective, the ingestion or prolonged exposure to dye-contaminated water may lead to mutagenic, carcinogenic, and cytotoxic effects, which can impact vital organs, including the kidneys, liver, reproductive system, and central nervous system. Therefore, effective removal of dyes from industrial effluents is of critical environmental and public health importance [3-6].

Dyes are typically organic compounds characterized by the presence of chromophoric groups responsible for color. Chromophores, such as azo (-N=N-), carbonyl (-C=O), nitro (-NO_2), and sulfur-containing groups (C=S), are conjugated systems with delocalized electrons that absorb specific wavelengths of light. These groups are often part of a chromogen, typically containing aromatic rings such as benzene, naphthalene, or anthracene, and are usually linked to auxochromes that modulate solubility and color intensity. Azo dyes, in particular, represent the largest class of synthetic dyes used in modern industries due to their vivid coloration and chemical stability. Other major dye types include reactive dyes, acid dyes, basic dyes, vat dyes, disperse dyes, and sulfur dyes, many of which are not readily removed through conventional biological treatment processes. Among the various treatment strategies, adsorption has emerged as one of the most effective, economical, and versatile methods for dye removal from aqueous media [7]. Adsorption relies on the physical or chemical interaction between dye molecules and the surface of a solid adsorbent. It is favored due to its simplicity, high efficiency, selectivity, and non-toxic nature, even at low dye concentrations. A wide variety of adsorbents have been investigated, ranging from traditional materials such as activated carbon, clays, and zeolites to more advanced materials, including nanocomposites, biopolymers, and functionalized hydrogels. These materials can be engineered to have high surface area, porosity, and specific functional groups that enhance dye uptake [8,9].

Clays are naturally occurring fine-grained aluminosilicate minerals composed primarily of hydrated silicates of aluminum. They exhibit plasticity when moistened and harden upon drying or firing. Due to their structural, chemical, and surface properties, clays are widely utilized in ceramics, construction, catalysis, nanocomposites, and environmental remediation, particularly as adsorbents for pollutants. Clays can be classified based on mineralogical composition, crystal structure, and layer arrangement. The two major structural groups are :1:1 Type Clays – These have one tetrahedral sheet linked to one octahedral sheet (e.g., kaolinite),2:1 Type Clays – These consist of one octahedral sheet sandwiched between two tetrahedral sheets (e.g., montmorillonite, illite, vermiculite), Another essential category is 2:1:1 clays (e.g., chlorite), which contain an additional octahedral sheet.[10] Major Types of Clays: a. Kaolinite: Belongs to the 1:1 group. Clays are a diverse group of mineral materials with unique structures and properties that make them essential across multiple scientific and industrial fields. Understanding their mineralogy, layer arrangement, and surface chemistry is crucial for tailoring them for advanced applications, particularly in environmental remediation and nanotechnology [11].

Vermiculite is a clay mineral composed of hydrated magnesium-aluminium-iron silicates, characterised by a 2:1 layered structure, with an octahedral sheet sandwiched between two tetrahedral sheets. Upon heating, vermiculite expands significantly due to the evaporation of water between the layers, increasing its surface area and enhancing its adsorption capacity. This clay has a high affinity for cationic dyes due to its negatively charged layers and large interlayer spacing, which facilitates electrostatic interactions and cross-linking processes [12, 13].

MATERIALS AND METHODS

Determination of Maximum Wavelength (λmax) and Calibration Curve for Crystal Violet (CV) Dye

Crystal violet (CV) is a widely used cationic dye, commonly applied in various industrial and

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biological applications. It appears as a violet, odourless crystalline powder and is highly soluble in water. For this study, a stock solution of CV dye at a concentration of 500 mg·L^{-1} was prepared by dissolving 0.5 g of the dye in 1000 mL of distilled water under constant stirring.

To determine the maximum absorption wavelength (λmax) of the CV dye, the UV–Visible absorption spectrum was recorded over the range of 200–800 nm using a UV-Vis spectrophotometer. The dye exhibited its maximum absorbance at 624 nm, as shown in Fig. 1, corresponding to the π→π* electronic transition of the chromophore group in the CV molecule. This wavelength was used for all subsequent absorbance measurements.

A calibration curve was established by preparing a series of standard solutions through serial dilution of the stock solution to achieve concentrations ranging from 2 to 30 mg·L^{-1}. The absorbance of each solution was measured at the predetermined λmax of 624 nm. The resulting calibration plot of absorbance versus concentration demonstrated a strong linear relationship, confirming adherence to Beer-Lambert's Law, as presented in Fig. 2. This calibration curve was used for the quantitative analysis of CV dye concentrations in adsorption

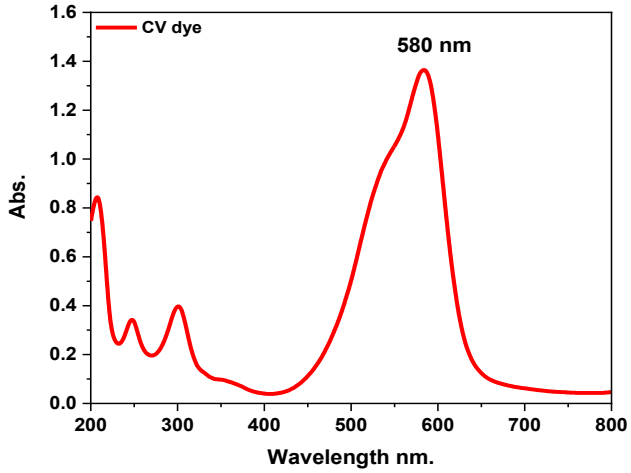

Fig. 1. UV-Visible Absorption Spectra of crystal violet (CV) dye.

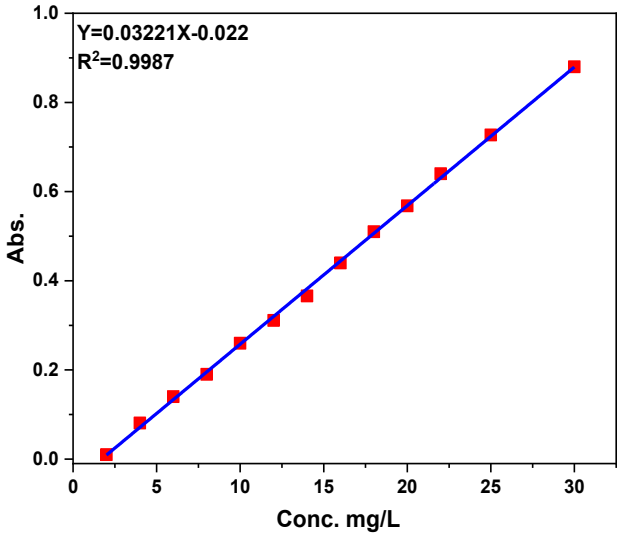

Fig. 2. Calibration curve for crystal violet (CV) dye

experiments.

Reusability and Regeneration Studies of the Clay-Based Adsorbent

To evaluate the reusability of the prepared clay-based adsorbent, a fixed mass of 0.5 g of the adsorbent was introduced into 100 mL of crystal violet (CV) dye solution with an initial concentration of 50 mg·L^{-1}. The adsorption process was conducted at a constant temperature of 25 °C and a controlled pH of 6.0 to ensure saturation of the active adsorption sites.

Following the saturation of the adsorbent, desorption experiments were performed to regenerate the anionic functional sites. Various chemical desorbing agents were investigated, including H_2SO_4, NaOH, H_3PO_4, HCl, HNO_3, ethanol, methanol, and distilled water, at three different concentrations: 0.01 N, 0.05 N, and 0.1 N. The adsorbent was immersed in 100 mL of each desorbing agent and stirred continuously for a specific desorption period to ensure maximum recovery of the adsorbed dye molecules. After the desorption step, the adsorbent was thoroughly washed with excess distilled water to remove residual desorbing agents and residual dye molecules, and then dried at ambient conditions before reuse. The adsorption-desorption process was repeated for six consecutive cycles under identical operational conditions: 100 mL of CV dye solution (50 mg·L^{-1}), 25 °C, and pH 7.0. After each cycle, the adsorption capacity and desorption efficiency were measured to assess the stability, efficiency, and regeneration potential of the clay-based adsorbent. This study demonstrated the clay material's ability to maintain high removal efficiency over multiple cycles, particularly when using distilled water and ethanol as desorbing agents, which were found to be the most effective in regenerating the adsorbent without compromising its structural integrity.

RESULTS AND DISCUSSION

SEM

Field Emission Scanning Electron Microscopy (FESEM) was employed to determine the particle size and shape. FESEM images of Clay and CV-dye adsorbed Clay appear in Fig. 3. The Clay was found to have a homogeneous and smooth surface with no irregularities (Fig. 3a). It is seen in the FESEM image of Clay-CV dye that it has a layered morphology and appears comparatively less uniform and more rough after the attachment of CV with the clay, and the considerably high adsorption efficiency of clay was due to the presence of these interconnected channels of pores in its structure[14] , as shown in Fig. 3b.

TEM

Transmittance Electron Microscopy (TEM) images reveal the morphology of the clay surface. As shown in Fig. 4, the clay appears cloudier, indicating the formation of new geometries. The photo shows that the clay exhibits irregular spherical shapes along with some patchy formations, and it tends to form chain-like structures measuring around 60 , and 100 nm. Additionally, the surface of the clay is covered by a transparent layer. Clay plays a pivotal role in enhancing stability and increasing surface area, making it an essential component in the synthesis of eco-friendly products[15].

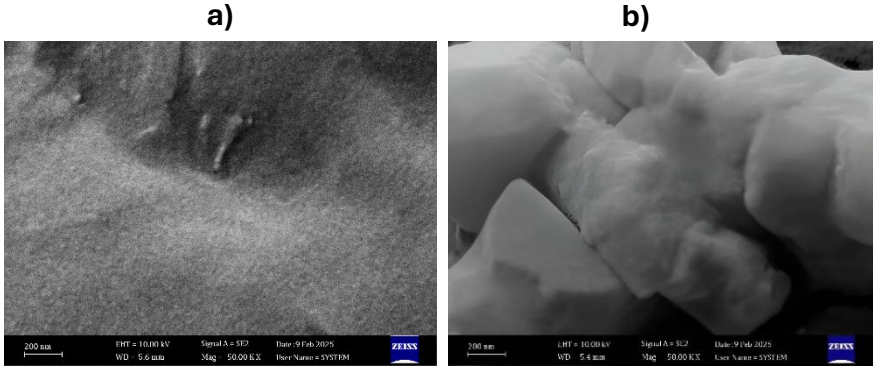

Fig. 3. FESEM image of a) Clay, b) CV dye loading of Clay

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XRD

X-ray diffraction (XRD) is a powerful analytical technique used to investigate the crystalline structure and determine interlayer spacing (d-spacing) in clay minerals. In the present study, XRD patterns of the clay-based adsorbent were recorded over a 2θ range of 10° to 80°, allowing for the identification of characteristic crystalline features and phase compositions. As illustrated in Fig. 5, distinct diffraction peaks were observed at 2θ values of 28.18°, 35.91°, 38.53°, and 45.04°, which are indicative of well-defined crystallographic planes within the clay structure [16, 17].

The shifting and intensity of these peaks suggest the presence of a crystalline phase and potential modification in the interlayer spacing, possibly due to surface interactions or structural rearrangement after adsorption. These peak positions correspond to the reflection from specific Miller indices, which can be further analyzed to deduce the crystal system and phase composition of the adsorbent material. Thus, the XRD analysis provides essential insight into the structural properties, crystallinity, and textural changes of the clay before and after interaction with dye molecules [18-20].

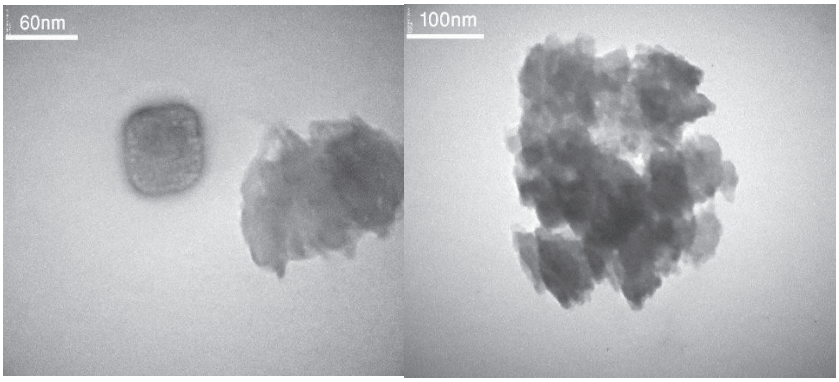

Fig. 4. TEM image of clay surface.

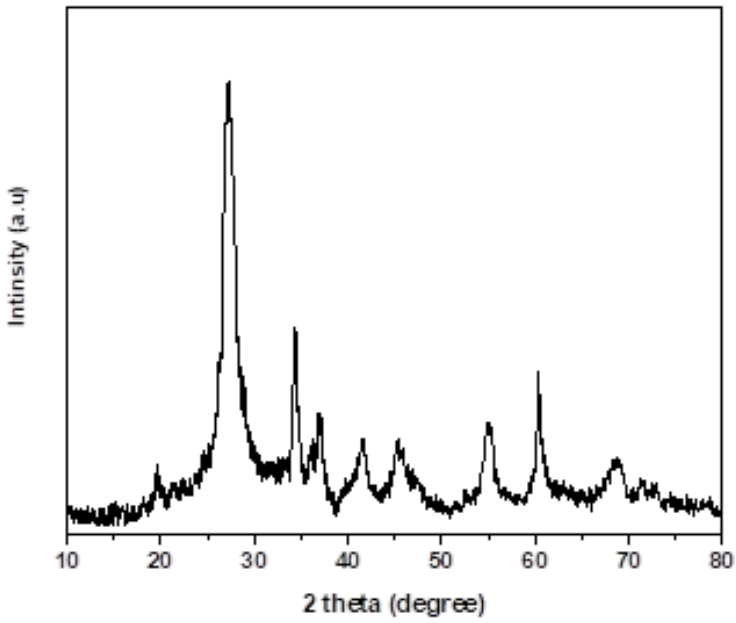

Fig. 5. X-ray diffraction (XRD) of clay surface.

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Thermogravimetric analysis (TGA)

Thermogravimetric analysis (TGA) was employed to evaluate the thermal stability and decomposition behaviour of the clay-based material. The TGA curve, as shown in Fig. 6, displays a single major weight loss event, indicating a one-step thermal degradation profile.

The initial weight loss observed in the temperature range of approximately 10–400 °C is attributed to the evaporation of physically adsorbed water and moisture content present within the clay matrix. This stage reflects the removal of surface and interlayer water molecules, which are commonly retained in the structure of hydrated clay minerals. A further weight loss occurring between 400–600 °C is associated with thermal decomposition of organic functional groups, primarily through decarboxylation reactions. This process results in the evolution of CO_2 gas, indicating the breakdown of carbonate-containing components or chemically bonded surface species. Overall, the TGA profile suggests that the material retains significant thermal stability up to ~400 °C, with decomposition processes becoming more pronounced at elevated temperatures due to the loss of structural and functional groups[21].

Regeneration and reuse

The regeneration of clay following sorption is an essential economic factor in the treatment

Table 1. Comparison of desorption efficiency of several kinds of solutions for the CV dye onto the surface of the Clay.

Reused and regeneration (0.01N)	E%	Reused and regeneration (0.05 N)	E%	Reused and regeneration (0.1 N)	E%
fresh	91.99	Fresh	91.99	Fresh	91.99
water	90.12	water	89	water	87.99
NaOH	86.88	NaOH	83.66	NaOH	80
HNO_3	77.78	HNO_3	71.77	HNO_3	69.00
H_3PO_4	66.66	H_3PO_4	61.88	H_3PO_4	58.99
H_2SO_4	60.99	H_2SO_4	55.55	H_2SO_4	50.87
HCl	58.88	HCl	52.99	HCl	47.99
Ethanol	45.55	Ethanol	40.77	Ethanol	39.77
Methanol	44.55	Methanol	40.11	Methanol	37.99

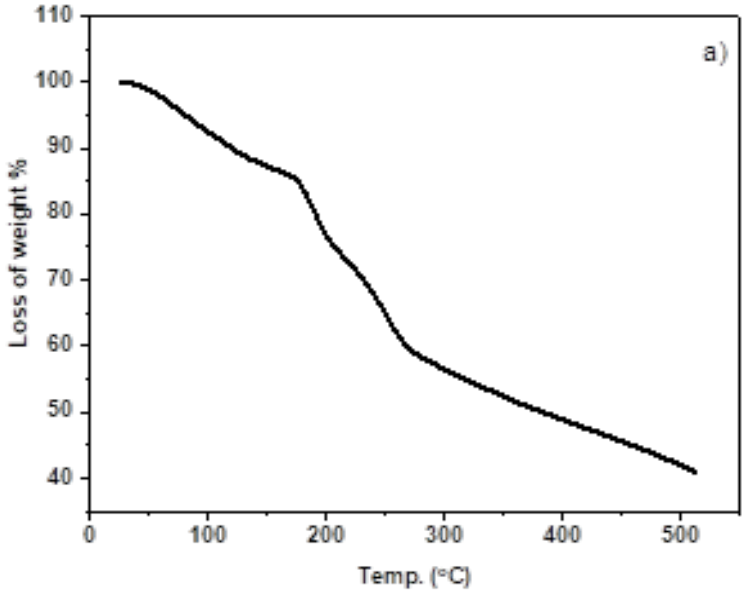

Fig. 6. Thermogravimetric analysis (TGA) of Clay surface.

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process. It helps clarify how the pollutant CV dye is removed from adsorbent materials, the mechanisms involved in regeneration, and the recycling of spent adsorbents. This approach can lower operational costs and help protect the environment from secondary pollution. Desorption studies for the pollutant (CV) were conducted using various desorption agents at different concentrations (0.01, 0.05, and 0.1 N), including H_2SO_4, NaOH, H_3PO_4, HCl, HNO_3, ethanol, methanol, and water. Remarkably, the clay was fully regenerated (100%) using water, as shown in

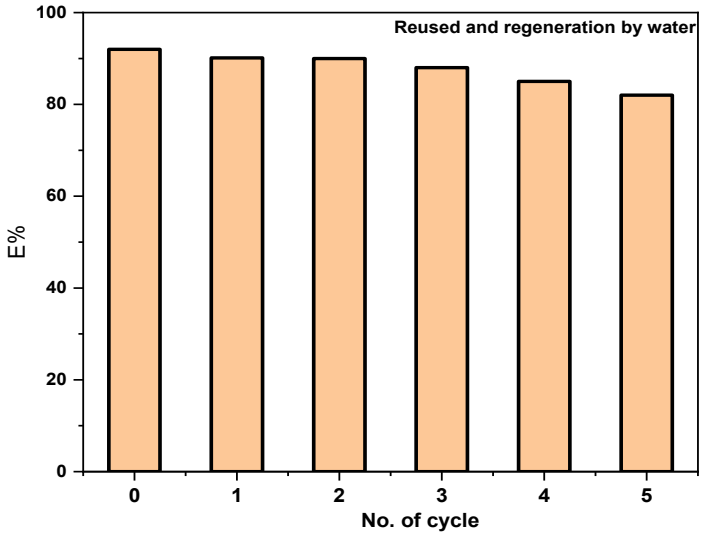

Fig. 7. Multi-cycle use of clay for CV adsorption using water as desorption solution.

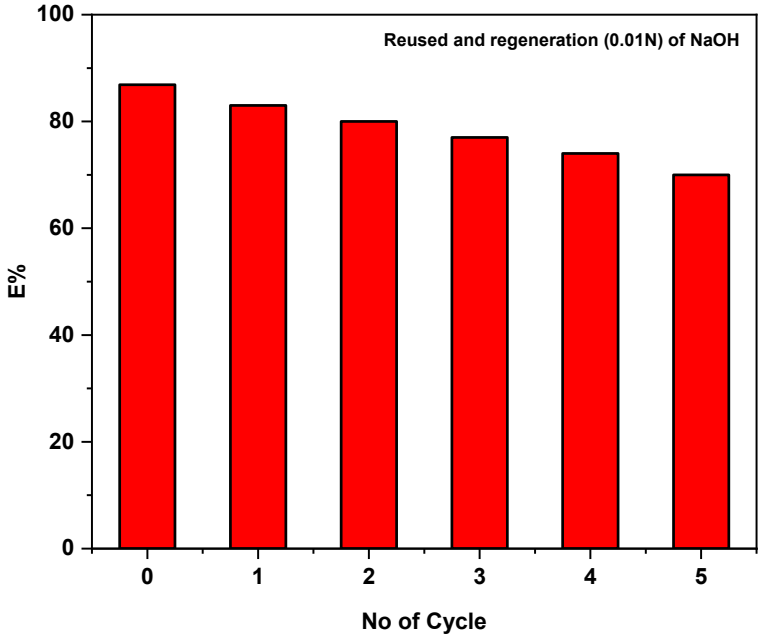

Fig. 8. Multi-cycle use of clay for CV adsorption using 0.01N NaOH as desorption solution.

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Table 1. The performance and reuse of clay for CV adsorption using water were tested over six cycles under optimal conditions (see Fig. 7). Even after four cycles, the efficiency remained high at over 80%, indicating that clay may serve as a renewable adsorbent. However, with an increased number

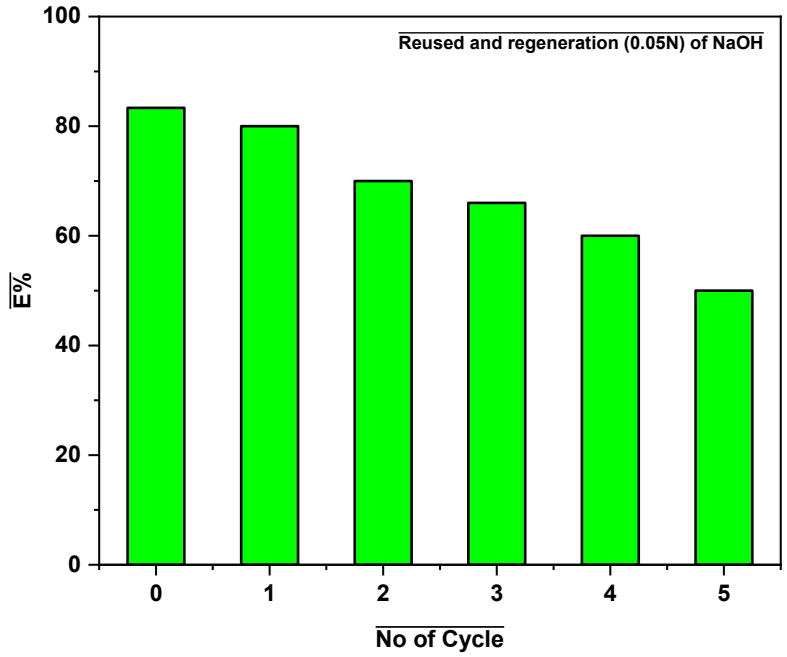

Fig. 9. Multi-cycle use of clay for CV adsorption using 0.05N NaOH as desorption solution.

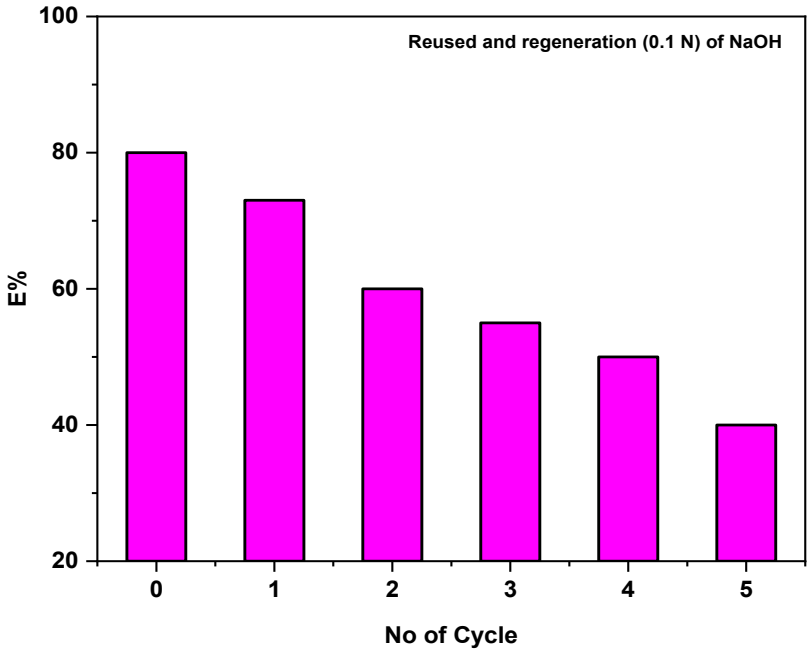

Fig. 10. Multi-cycle use of clay for CV adsorption using 0.1N NaOH as desorption solution.

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of hydrogel adsorber applications, the efficiency slightly declined [22, 23]. This reduction can be attributed to several factors, including damage to active sites on the adsorbent, saturation of active sites, and the breakdown of the clay network during continuous adsorption-desorption cycles, which compromised the structure of the clay particles and reduced removal efficiency.

Sodium hydroxide (NaOH) was investigated as a regenerating agent for the desorption and reactivation of the dye-saturated clay surface. Three different concentrations—0.01 N, 0.05 N, and 0.1 N were used to assess their effect on the efficiency of surface reactivation and subsequent dye removal performance. At 0.01 N NaOH, the low concentration of hydroxide ions facilitates mild desorption of dye molecules from the clay surface. This gentle regeneration condition helps preserve the structural integrity and chemical functionality of the adsorbent, thereby maintaining a relatively high dye removal efficiency in subsequent adsorption cycles [24]., as shown in Fig. 8.

At 0.05 N NaOH, a moderate increase in hydroxide ion concentration enhances the desorption capacity, improving the reactivation of the clay surface. However, partial disruption of surface-active sites may begin to occur at this level, leading to a slight reduction in dye removal efficiency compared to the lowest concentration , as shown in Fig. 9.

At 0.1 N NaOH, the high concentration of hydroxide ions can lead to excessive deprotonation and possible alteration of the clay's surface chemistry. Such aggressive chemical interaction may damage or block the functional groups responsible for adsorption, resulting in a significant decrease in surface reactivation and adsorption efficiency [25, 26]. This phenomenon suggests that overly concentrated NaOH solutions can impair the physical and chemical characteristics of the adsorbent material, as shown in Fig. 10.

In summary, as NaOH concentration increases, the ability of the base to effectively regenerate the clay surface decreases, likely due to chemical alteration or degradation of active adsorption sites. Therefore, milder concentrations (e.g., 0.01 N) are more favorable for maintaining the adsorbent's efficiency and structural stability during repeated regeneration cycles.

CONCLUSION

Various studies have been conducted to enhance our understanding of the equilibrium aspects of adsorption using different adsorbents, particularly clay surfaces. The removal of pollutants, specifically CV dye, from aqueous solutions through adsorption on clay surfaces has been experimentally investigated. The most effective results were observed at a pH of 6.6, a temperature of 25°C, and an adsorbent dosage of 0.1 g of clay, achieving optimal removal percentages. Additionally, the clay was successfully regenerated, with 100% desorption of the CV dye achieved through water in the adsorption process, which was tested for up to five cycles under optimal conditions. Sodium hydroxide (NaOH) was investigated as a regenerating agent for the desorption and reactivation of the dye-saturated clay surface. Three different concentrations 0.01 N, 0.05 N, and 0.1 N were used to assess their effect on the efficiency of surface reactivation and subsequent dye removal performance. In summary, as NaOH concentration increases, the ability of the base to effectively regenerate the clay surface decreases, likely due to chemical alteration or degradation of active adsorption sites. Therefore, milder concentrations (e.g., 0.01 N) are more favorable for maintaining the adsorbent's efficiency and structural stability during repeated regeneration cycles.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this manuscript.

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