Waste glass: An excellent adsorbent for crystal violet dye, $Pb^{2+}$ and $Cd^{2+}$ heavy metal ions decontamination from wastewater

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Abstract

The suitability of waste glass as an eco-friendly adsorbent for the removal of crystal violet (CV) dye, $Pb^{2+}$ and $Cd^{2+}$ heavy metal ions in waste water samples was investigated in batch mode. Waste glass sample was pulverized and characterized by SEM/EDX, XRD, BET and FTIR. Effects of variation in temperature, pH, contact time and recyclability of the adsorbent were studied. FTIR spectra revealed major peaks around 491.53 and 3444.12 cm$^{-1}$ corresponding to the bending vibrations of Si-O-Si and -OH groups respectively. SEM/EDX analysis showed a dense, coarse, porous morphology with predominantly silica component. The effective surface area and size of the adsorbent were 557.912 m$^2$/g and 2.099 nm respectively. Increase in temperature, dosage, contact time resulted in increase in adsorption efficiency. Optimum adsorption efficiency of 94%, 97.5% and 89.1% was attained for $Pb^{2+}$, $Cd^{2+}$ ions and CV dye respectively at 70$^\circ$C. Adsorption process followed more accurately pseudo-first order model and isotherm fitted perfectly into Freundlich model indicating a multilayer adsorption mechanism for CV dye and the heavy metals. 89.87% reduction in Chemical Oxygen Demand (COD) level of wastewater was reported upon treatment with waste glass adsorbent affirming its efficiency for dye and heavy metal pollutants removal.

DOI:10.46481/jnsps.2021.261

Keywords: waste glass, crystal violet dye, heavy metals, adsorption, wastewater

Article History:
Received: 18 June 2021
Received in revised form: 26 September 2021
Accepted for publication: 27 September 2021
Published: 29 November 2021

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Communicated by: E. Etim

1. Introduction

Glass is one of the earliest man-made materials. It is an inorganic, non-crystalline material obtained by melting, homogenization, cooling and finishing of components such as sand, sodium oxide, limestone, etc. Glass has found useful applications in television screens, glass bottles, doors, car wind-screens, packaging materials, certain industrial and scientific equipment, etc, [1]. Increasing global population and industrialization have led to a sharp rise in the use of glass products in household and industrial devices resulting in the generation of huge volume of waste glass. According to the United Nations estimates, the annual global amount of disposed solid waste is said to be 200 million tons, comprising 7% glass [2]. Generally, glass is relatively stable, non-biodegradable, resistant to attack and outdoor temperature [3]. Recycling is a useful method of reducing pollution load associated with waste glass. However, poor financial and technological capacity for proper waste glass recycling remain a major problem particularly in developing countries.

Although dyes have numerous applications in food, cosmet-
ics, pharmaceutical and textile industries, improper discharge of effluents containing dyes into water bodies negatively impact on their aesthetic value; threaten the survival of aquatic species by impairing photosynthesis, etc. [3, 4]. Bioaccumulation of dyes and heavy metals especially lead and cadmium in living cells can lead to impairment of haemoglobin synthesis, neurological disorder, kidney damage, carcinogenicity, mutagenicity, etc. [5]. In particular, exposure of pregnant women to elevated levels of lead may result in miscarriage, stillbirth, premature birth, deformities, etc. [6]. Several methods such as adsorption, ion exchange, photo-degradation, physical precipitation, etc. have been reported for the removal of dyes and heavy metals in the environment [7, 8, 9, 10, 11]. Of these methods, adsorption remains the most attractive based on cost, ease of operation, analysis time, etc [12]. Different natural and synthetic materials including clay, activated carbon, termite mounds, agricultural wastes (sawdust, brans, banana peels), etc. have been investigated as economic adsorbents for decontamination of dyes and heavy metals [13, 14, 15, 16]. For instance, Ahmad reported the removal of crystal violet (CV) dye by adsorption using coniferous pinus bark powder [17]. The amount of CV dye uptake increased with increase in initial pH, dye concentration and contact time but decreased with adsorbent dosage increase. Equilibrium was attained in 2 h. The isotherm fitted most into Langmuir model indicating a monolayer adsorption mechanism while the kinetics followed perfectly a pseudo second order rate equation. Also, fly ash and rice husk adsorbents were investigated for the removal of copper, lead, cadmium, nickel and iron heavy metals [18]. The rice husk adsorbent showed better affinity towards iron, nickel and lead while fly ash was more effective for the removal of copper and cadmium [18].

Crystal violet (CV) is categorized under the triarylmethane dyes. It is equally referred to as hexamethyl pararosaniline chloride, gentian violet, etc [9]. It possesses antibacterial, antifungal and antihelminthic properties [7]. It is widely used as a histological stain, for paints and textile applications [19]. CV dye is carcinogenic, non-biodegradable, persistent in the environment due to its poor metabolism by microorganisms [11]. Access to clean water and environmental sanitation remains a key objective of the Sustainable Development Goals (SDGs) of the United Nations (UN). Utilisation of waste glass as a cheap and effective adsorbent for the decontamination of toxicants in polluted water bodies would promote cleaner environment, minimise water pollution and improve quality of life especially in rural communities of developing nations. Thus, the focus of the work was to utilise waste glass from the local environment for the decontamination of crystal violet (CV) dye and toxic heavy metal ions (Pb\(^{2+}\) and Cd\(^{2+}\) ions) in aqueous medium. Effects of variations in pH, temperature, adsorbent dosage, pollutant concentration, recyclability on the adsorption efficiency, kinetics, adsorption isotherm and practical application in the treatment of waste water are herein presented.

2. Methodology

2.1. Materials/Reagents

All the chemicals used were of analytical reagent grade or the highest purity available.

2.2. Sample collection and treatment

2.0 kg of transparent soda-lime wasteglass bottle samples were collected around Oye town, Ekiti state, Nigeria. The samples were cleaned and ground into fine powder by dry ball milling machine operated at ambient temperature for 8 h. The pulverised wasteglass (particle size 75 \(\mu\)m) was carefully kept in a glass vial and stored at room temperature (30 \(\pm\) 2°C) till further use.

2.3. Scanning electron microscopy/Energydispersive X-ray (SEM/EDX) analysis

Morphological features and surface characteristics of glass sample were obtained from scanning electron microscopy/Energy Dispersive X-ray (SEM/EDX) using a JEOL USA Model: JSM-7900F. A thin layer of waste glass sample granule was placed on aluminium specimen holder by double-sided tape. The specimen holder was loaded in a polaron SC 7610 sputter coater and coated with gold to a thickness of about 30nm to prevent charging. The specimen holder was transferred to XL-20 series. SEM/EDX analysis was conducted at an accelerating voltage of 15-20 kV.

2.4. Fourier transform infra red (FTIR) spectroscopic analysis

FTIR spectra of the waste glass samples were run as KBr pellets on Perkin Elmer Spectrum Two TM spectrometer in the frequency range 4000 – 400 cm\(^{-1}\).

2.5. X-ray Diffraction studies

X-ray diffraction patterns of powdered waste glass sample was obtained using Empyrean XRD diffractometer at 40 mA and 45 kV with Cu K\(\alpha\) (1.5418 \(\AA\)) radiation at an angular incidence of 10 – 75\(^\circ\).

2.6. Brunauer-Emmett-Teller (BET) surface area analysis

The specific surface area (BET) of powdered waste glass sample was measured with a Quantachrome surface analyzer using \(N_2\) as the standard adsorbate. Prior to analysis, the sample was degassed at 523 K for 3 h. The pore size and pore volume were estimated using Barrett-Joyner-Halenda (BJH) theory.

2.7. Batch adsorption experiment

Batch adsorption method as reported by Ayanda et al. [20] was adopted with slight modification. Briefly, a range of powdered waste glass (0.1 g - 1.0 g) was contacted with 40 mL of 5 mg/L CV dye and stirred at 300 rpm at 40\(^\circ\)C for 30 min for the effect of waste glass adsorbent dosage. To investigate the influence of pH on the adsorption process, the pH of 40 mL of 5 ppm CV dye solution containing 0.5 g waste glass adsorbent was adjusted to pH range 2-10 using either 0.1 M HCl or 0.1 M NaOH. The effect of time was studied between 20-60 min
while the effect of temperature was determined at a temperature range of 30-70 °C. On the effect of initial dye concentration, 40 mL of 1, 5, 10, 15 and 20 ppm CV dye were used along with 0.5 g powdered waste glass adsorbent at 40°C for 30 min. The resulting mixture after each experiment was filtered and absorbance of the aliquot of the filtrate was determined using T-60 UV-Visible spectrophotometer at 592 nm. For the Pb$^{2+}$ and Cd$^{2+}$ heavy metal ions determination, the same procedure was repeated. The Pb$^{2+}$ and Cd$^{2+}$ levels were analysed using Atomic Absorption Spectrophotometer (AAS Buck Scientific model 211 VGP) with the respective hollow cathode lamps. The adsorption efficiency experiments were carried out for three repeated cycles to study the recyclability and stability of the waste glass sorbent. After each cycle, the adsorbent was filtered and oven-dried at 70°C for 30 min. The chemical oxygen demand (COD) was equally determined from the slope and intercept respectively.

The linear form of the Freundlich equation is written as:

$$\log q_e = \log K_F + 1/n \log C_e,$$

where $C_e$ is the equilibrium concentration of pollutant analyte solution (mg/L), $q_e$ is the concentration of pollutant adsorbed per unit mass of the waste glass (mg/g) while $n$ is the number of layers and $K_F$ is the Freundlich constant. $K_F$ and $n$ values are deduced from the respective intercept and slope of the linear plot of $\log q_e$ against $\log C_e$.

2.9. Kinetic models studies

For detailed studies of the adsorption kinetics of Pb$^{2+}$, Cd$^{2+}$ ions and CV dye on the waste glass sample, the Lagergren pseudo-first-order model [22] and pseudo-second-order kinetic model [23] were used. They are given, respectively, as:

$$\ln(q_e - q_t) = \ln(q_e) - k_1 t,$$

$$t/q_t = 1/(k_2 \cdot q_e^2) + t/q_e,$$

where $q_t$ and $q_e$ are the amount of pollutant adsorbed (mg/g) at time $t$ and equilibrium, respectively. $k_1$ (min$^{-1}$) is the rate constant of the pseudo-first-order kinetic model and $k_2$ (g · mg$^{-1}$ · min$^{-1}$) is the rate constant of the pseudo-second-order kinetic model.

3. Result and Discussion

3.1. SEM/EDX and BET surface area analyses of waste glass powder

Surface morphology and chemical composition of an adsorbent play a major role in ascertaining the efficiency and effectiveness of the adsorption process. The SEM image (Figure 1a) of the porous waste glass showed a dense, coarse, porous surface morphology. EDX analysis (Figure 1b) revealed the % composition of the waste glass in the following order: Si>Mg>C>Ca>Fe>S>Na>K. These elements are present in form of oxides (e.g. SiO$_2$, MgO, Fe$_2$O$_3$, etc.), carbonates (calcium carbonate, sodium carbonate, potassium carbonate, etc), silicates; with silica as the predominant component. The BET specific surface area of the waste glass was about 557.912 m$^2$/g while the Barrett, Joyner, Halenda (BJH) surface area was measured to be about 663.9 m$^2$/g. The BJH pore volume was calculated to be about 0.3264 cc/g with an average pore size of 2.099 nm (see Figure 2). The dense, coarse, porous nature of the glass surface coupled with interaction with various chemical components facilitated adsorption of pollutant analytes onto its surface under prevailing conditions.

3.2. FTIR spectra of waste glass

The result of the FTIR analysis of the porous waste glass particles is shown in Figure 3. The peaks at 412.31, 419.22, 431.95, 444.07, 451.09, 455.22, 473.95, 482.59 and 491.53 cm$^{-1}$ revealed bending vibrations of Si–O–Si, while the peaks at 528.01, 519.90, 504.54, 511.85 cm$^{-1}$ showed the stretching vibration of O–Si–O. The peaks observed at 605.00, 645.43,
644 and 694.53 cm\(^{-1}\) correspond to stretching vibrations of Si–O–Al and M–X [3]. The peaks at 3444.12, 3563.40, 3605.19 and 3838.03 cm\(^{-1}\) indicate the –OH group from the silanol (Si–OH) or adsorbed water molecule trapped within the silicate structure of the waste glass particles [24]. In general, the various peaks in the spectra confirmed the presence of silicates, carbon, metal oxides, and metal halides in the waste glass particles [3]. These functional groups played crucial role in the overall adsorption of analytes at the sorbent surface through electrostatic interaction, precipitation, etc.

3.3. XRD pattern of waste glass

XRD pattern of the waste glass powder is shown in Figure 4. The prominent peak at 2\(\theta\) = 27.1\(^{\circ}\) corresponds to the silica-silica bond. Smaller peaks at 2\(\theta\) = 22.5\(^{\circ}\), 29.5\(^{\circ}\), 35.2\(^{\circ}\), 37.3\(^{\circ}\), 40.4\(^{\circ}\), 44.3\(^{\circ}\), 51.5\(^{\circ}\), 60.2\(^{\circ}\), and 68.3\(^{\circ}\) are due to the presence of other mineral components including sodium oxide, calcium oxide, etc in the waste glass [3]. This result agreed with other previous studies on waste glass [25].

3.4. Result of adsorption studies

3.4.1. Effect of temperature

The effect of temperature on the adsorption efficiency is presented in Figure 5a. Increasing the temperature of the medium from 30 – 70\(^{\circ}\)C resulted in increase in the % adsorption efficiency of analytes (CV dye, \(Pb^{2+}\) and \(Cd^{2+}\) metal ions). Maximum % adsorption efficiency was attained at 70\(^{\circ}\)C in all the
analytes. This might be due to the increase in the average kinetic energy, collision frequency and higher diffusion rate of the analytes to the porous glass powder surface at higher temperature. Aniagor and Menkiti [7] reported similar observation at elevated temperature using lignified bamboo isolate for the removal of crystal violet dye effluent.

3.4.2. Effect of pH

The degree of acidity or alkalinity of the medium plays crucial role in the overall adsorption process by facilitating precipitation, co-precipitation and sorption processes through electrostatic interaction (attraction or repulsion) of charges between the adsorbent and adsorbate at the surface. For this study, the effect of pH was studied between pH 2-10 at 40°C, 0.5 g adsorbent dosage for 30 min. In the case of CV dye, a reduction in % adsorption efficiency was observed as the pH progressed from 2-10 (Figure 5b). The silicate glass surface (adsorbent) acquired a negative surface charge density in aqueous medium largely due to the dissociation of the silanol (Si–OH) group. The observed decrease in % adsorption efficiency might be due to an increase in electrostatic repulsion between the negatively charged –OH group of the silanol (in the adsorbent) and the –OH group of the aqueous medium of equal ionic size and charge at higher pH. These negatively charged –OH ions thus occupy position far away as possible from the adsorbent surface to minimize electrostatic repulsion. Consequently, less negatively charged species are available at the adsorbent surface for adsorption (via electrostatic attraction with the cationic CV dye) and hence the reduction in % adsorption efficiency. Unlike the case of CV dye, increasing the pH of the medium resulted in gradual increase in the adsorption efficiency of the heavy metals (Pb$^{2+}$ and Cd$^{2+}$). At higher pH (alkaline medium), precipitation of Pb$^{2+}$ and Cd$^{2+}$ ions readily occur on the glass (adsorbent) surface thus favouring higher adsorption efficiency. This implied that the energy barrier required for precipitation is much less than electrostatic repulsion between the –OH group of the silanol (adsorbent) and the –OH group of the aqueous alkaline medium. The observed variation in pH of this work is in contrast to the earlier studies on CV dye removal using treated ginger waste [26].

3.4.3. Effect of adsorbate concentration

Adsorption experiments were carried out at a concentration range of 1-20 ppm with an adsorbent dosage of 0.5 g of waste glass adsorbent at 30°C for 30 min (Figure 6a). The result showed a gradual increase in the % adsorption efficiency as the adsorbate concentration increased for the heavy metal ions and the CV dye. This might be due to large surface area (with sufficient active sites), retardation of resistance towards adsorbate uptake, which increase diffusion to the surface and relatively small adsorbent pore size for effective trapping of adsorbate ions [9]. At 15 ppm, % maximum adsorption efficiency (equilibrium) was attained for the pollutant (adsorbate) molecules signaling the maximum saturation at the adsorbent surface. Above 15 ppm, the adsorbent surface became supersaturated with the pollutant molecules hence the slight reduction in the adsorption efficiency.

3.4.4. Effect of adsorbent dosage

The study of the adsorbent dosage effect was important in order to ascertain the minimum possible dosage amount with the maximum adsorption efficiency. From Figure 6b, it was observed that the adsorption efficiency increased progressively as a function of the waste glass dosage (0.1-1.0 g). This is due to the fact that more porous active sites became more readily accessible to the adsorbate ions (Pb$^{2+}$, Cd$^{2+}$ ions and CV dye) for surface adsorption. Maximum % adsorption efficiency of 75%, 94% and 97.5% was recorded for CV dye, Pb$^{2+}$ and Cd$^{2+}$ ions, respectively.

3.4.5. Regeneration efficiency of waste glass adsorbent

The regeneration efficiency is a measure of recyclability and stability of an adsorbent per number of consecutive adsorption cycle. For this study, the waste glass adsorbent was investigated for three consecutive runs for the removal of CV dye, Pb$^{2+}$ and Cd$^{2+}$ ions (Figure 7). The result showed a slow, steady reduction in regeneration efficiency of the adsorbent over the three runs in the following order: Cd$^{2+}$ > CV dye > Pb$^{2+}$. % regeneration efficiency for the first repeated cycle is 78%, 72% and 61% for Cd$^{2+}$, CV dye and Pb$^{2+}$ ions respectively. This reduced to 57%, 50% and 45% for Cd$^{2+}$, CV dye and Pb$^{2+}$, respectively after the third cycle. The relative stability and efficiency of the waste glass adsorbent might be due to its relatively large pore size, specific surface area and pore volume which facilitated easy adsorption and separation of adsorbates at the porous glass surface.

3.5. Adsorption isotherm and kinetic models

Figure 8 shows the plots obtained for the Langmuir and Freundlich adsorption isotherm models for the pollutants; CV dye, Pb$^{2+}$ and Cd$^{2+}$ ions. The slopes, intercepts and the corresponding constants of the models are presented in Table 1. In all the cases studied, the regression ($R^2$) values are much relatively higher for the Freundlich isotherm model than the Langmuir isotherm model indicating that the Freundlich model is more favoured by the adsorption process. This implied that the uptake of CV dye and the heavy metal analytes occurred on a heterogeneous surface by multilayer adsorption and there is a non-uniform distribution of the heat of adsorption over the porous waste glass surface. Based on the foregoing, the separation factor ($R_L$); a useful non-dimensional parameter in isotherm studies was determined. RL described the suitability of the porous waste glass powder and its affinity towards CV dye, Pb (II) and Cd (II) ions. $R_L$ value was calculated using:

$$R_L = 1/(1 + K_LC_0),$$  \hspace{1cm} (7)

where $C_0$ is the initial concentration and $K_L$ signifies the Langmuir constant. Four probabilities exist for the value of $R_L$: $R_L > 1.0, R_L = 1, 0 < R_L < 1,$ and $R_L = 0$, indicating unfavorable, linear, suitable, and irreversible degrees, respectively. From Table 1, the calculated $R_L$ values are 0.19, 0.41 and 0.33 for CV dye, Pb (II) and Cd (II) metal ions, respectively, indicating the suitability of the adsorption process using porous waste
Figure 5: Effect of: (a) temperature on the adsorption efficiency of waste glass (stirring speed = 300 rpm, contact time = 30 min, dosage = 0.5 g, initial concentration = 5 ppm each of CV dye at 592 nm, \( \text{Pb}^{2+} \) and \( \text{Cd}^{2+} \)), (b) pH on the adsorption efficiency of waste glass (temperature = 30°C, stirring speed = 300 rpm, contact time = 30 min, dosage = 0.5 g, initial concentration = 5 ppm each of CV dye at 592 nm, \( \text{Pb}^{2+} \) and \( \text{Cd}^{2+} \)).

Figure 6: (c) Effect of initial concentration on the adsorption efficiency of waste glass (temperature = 30°C, stirring speed = 300 rpm, contact time = 30 min, dosage = 0.5 g). (d): Effect of dosage on the adsorption efficiency of waste glass (temperature = 30°C, stirring speed = 300 rpm, contact time = 30 min, concentration = 5 ppm).

Table 1: Adsorption isotherms and separation factor parameters

| Adsorbate | Langmuir isotherm model constants | Freundlich isotherm model constants | Separation factor (\( R_L \)) |
|-----------|---------------------------------|-----------------------------------|-----------------------------|
| CV dye    | \( K_L = 11.57 \) \( q_m = 12.34 \text{ mg/g} \) \( R^2 = 0.895 \) | \( K_F = 12.08 \) \( n = 3.66 \) \( R^2 = 0.991 \) | 0.19 |
| \( \text{Pb}^{2+} \) | \( K_L = 1.50 \) \( q_m = 166.7 \text{ mg/g} \) \( R^2 = 0.972 \) | \( K_F = 89.54 \) \( n = 1.78 \) \( R^2 = 0.985 \) | 0.41 |
| \( \text{Cd}^{2+} \) | \( K_L = 1.88 \) \( q_m = 66.67 \text{ mg/g} \) \( R^2 = 0.936 \) | \( K_F = 422.67 \) \( n = 0.65 \) \( R^2 = 0.952 \) | 0.33 |

glass powder. We have also studied the kinetics of adsorption of the three pollutants onto the surface of the waste glass using the pseudo-first and second order kinetic models (Figure 9). The pseudo-first and -second rate constants derived from Fig-
Figure 7: Regeneration efficiency of waste glass.

Figure 8: (a) Langmuir and (b) Freundlich adsorption isotherms

Figure 9: Pseudo-first (a) and pseudo-second (b) order kinetic plots.

Table 2: Pseudo first order and second order kinetic model parameters.

| Parameter | CV dye | Cd (II) | Pb (II) |
|-----------|--------|---------|---------|
| $k_1 (min^{-1})$ | 0.0065 | 0.0491 | 0.0166 |
| $R^2$ | 0.9066 | 0.8688 | 0.9711 |
| $k_2 (g \cdot mg^{-1} \cdot min^{-1})$ | 0.0007 | 0.0471 | 0.0036 |
| $R^2$ | 0.8999 | 0.8294 | 0.9525 |

3.6. Treatment of textile effluent

Practical application of the waste glass powder adsorbent was investigated in the treatment of textile dye effluent. The COD, CV dye, Pb$^{2+}$ and Cd$^{2+}$ ions levels of the effluent were determined before and after treatment of the wastewater. The COD is a measure of the amount of oxygen required for chemical oxidation of organic pollutants in an effluent (e.g. textile wastewater). The result showed a significant reduction in the COD value from $694.26 \pm 0.21 \text{ mg/L}$ to $70.34 \pm 0.08 \text{ mg/L}$ after treatment representing $89.87\%$ decrease in COD level thus affirming its pollutant removal capacity. The result of this work is comparable to previous studies on treatment of textile effluents using different adsorbents [20, 27]. Exposure to high levels of cadmium from untreated effluent discharge had been reported as a major cause of “itai-itai” disease; an ailment characterised by kidney malfunctioning, osteomalacia, marked decalcification, etc [28]. Upon treatment of the textile effluent
with waste glass powder adsorbent, % adsorption efficiency of 81.69%, 82.2% and 89.5% was achieved for CV dye, Pb (II) and Cd (II) ions, respectively (Figure 10). \( \text{Pb}^{2+} (0.004 \pm 0.19 \text{ mg/L}) \) and \( \text{Cd}^{2+} (0.001 \pm 0.14 \text{ mg/L}) \) concentrations obtained after treatment are relatively much lower than the 0.01 mg/L and 0.003 mg/L World Health Organisation recommended values for Pb (II) and Cd (II) metal ions respectively in drinking water [6].

![Figure 10: Adsorption efficiency of waste glass for the removal of CV dye at 592 nm, Pb\(^{2+}\) and Cd\(^{2+}\) ions in a textile dye wastewater (effluent volume= 40 mL, stirring speed = 300 rpm, temperature = 30°C, contact time = 30 min, dosage = 0.5 g).](image)

### 4. Conclusion

Waste glass from local environment was pulverized, characterized and successfully applied for the removal of \( \text{Pb}^{2+}, \text{Cd}^{2+} \) ions and CV dye in waste water. Structural crystallinity, surface morphology, functional groups and surface area analyses of the porous waste glass showed predominantly silica crystalline phases with highly active \(-\text{OH}\) group on the dense, coarse, porous glass adsorbent. The pH of the medium strongly influenced the adsorption process via electrostatic interaction and precipitation. The adsorption isotherm and kinetics of the process favoured Freundlich isotherm and pseudo-first order models, respectively. The waste glass adsorbent displayed remarkable efficiency and effectiveness in the practical treatment of textile effluent and can thus be recommended for the removal of other environmental pollutants.

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