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(RESEARCH ARTICLE)

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Bio-composite of chitosan and polyethylene for the biosorption of dye in wastewater

Oluwaseun Adekoya Adelaja *

Department of Chemistry, School of physical sciences, Federal University of Technology Akure, P.M.B. 704, Akure, Nigeria.

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Abstract

This study investigated the potential of a biocomposite adsorbent made of low-density polyethylene-chitosan nanoparticles (LDPE/CHNP) for removing Congo red and Crystal violet dyes from wastewater. Batch experiments were conducted at room temperature to study the effects of pH, contact time, initial concentration, adsorbent dosage, and temperature on the adsorption process. The results showed that the maximum sorption of Congo red and Crystal violet occurred at pH 8. The adsorption process was rapid in the first 30 minutes of contact, with more than 90% uptake, and equilibrium was achieved with 150 rpm agitation. The biosorption of Congo red and Crystal violet dyes was described using Langmuir, Freundlich, and isotherm models. The Langmuir model was found to fit the equilibrium data better, with a correlation coefficient of 0.99 and a maximal adsorption capacity of 27.1 mg/g. Based on these results, it can be concluded that the biocomposite adsorbent made of low density polyethylene-chitosan nanoparticles (LDPE/CHNP) has the potential to be an effective and abundant alternative biomass for removing these dyes from wastewater. The LDPE/CHNP biocomposite adsorbent could be applied as a remediation method for color contamination in wastewater.

Keywords: Polyethylene; Chitosan; Nanoparticles; Dye; Biosorption

1. Introduction

Waste is an inevitable outcome resulting from physical, chemical, biological, and industrial activities, with waste water being a predominant contributor. The rapid pace of industrialization has a substantial effect on the emission of harmful substances as waste/byproducts, which are predominantly conveyed through water. As a consequence of the considerable usage of water for various procedures (such as chemical and industrial processes: textiles, pulp and paper, metallurgy, leather, paint and coatings, food, packaging, pharmacy, and plastics), waste water has become a significant concern with regard to environmental welfare. The discharge of industrial effluent, or wastewater, is a leading cause of environmental pollution. Dyeing industries are a significant contributor to this problem, as their effluents are highly colored and contain large suspended organic solids (Kanawade S.M., Gaikwad R.W., 2011). Dyes are among the many contaminants found in wastewater, particularly during dyeing and finishing operations, due to their high solubility. Until recent times, wastewater management was not a common practice, and wastes were often disposed of directly into rivers, uninhabited lands, seas, and dump sites. These practices have had adverse effects on human health and the ecosystem (Abioye et al., 2022). Currently, there is no single method capable of adequately treating industrial wastewater due to its complex nature. The techniques employed for removing toxic heavy metals from industrial wastewater range from ion-exchange to adsorption. However, Biosorption presents a more promising option due to its cost-effectiveness and eco-friendliness. Although biomass has been used for environmental cleanup for some time, there are expectations that biosorption, which involves the use of selective biomass or materials (biosorbents), will provide an economical alternative for effectively removing toxic heavy metals from industrial wastewater and aiding in environmental remediation. According to Wikipedia (2021), a dye is a substance that possesses color and chemically binds to the substrate onto which it is applied.

* Corresponding author: Oluwaseun Adekoya Adelaja

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Dyes are distinguishable from pigments in that they do not undergo chemical combination with the substrate to which they adhere. Consequently, the removal of dyes as waste water contaminants is a highly complex process with very few selective treatment methods having demonstrated efficacy (Rustamaji et al., 2023). Dyes are utilized extensively across a wide spectrum of applications, including industrial, printing, food, cosmetic, clinical, and textile domains. Notwithstanding their utility, the stability and toxicity of dyes, especially synthetic ones such as Azo dyes, can contribute to environmental pollution upon their release into effluents. Owing to the pervasiveness of dyes as impurities in wastewater and their potential menace to life, degradation and treatment pose certain challenges, often necessitating a sequence of physical, chemical and biological processes to ensure complete elimination of pollutants. Furthermore, given that some dyes are toxic and mutagenic, it is imperative to exercise caution in wastewater treatment selection. Physical-chemical treatments such as adsorption, coagulation/flocculation precipitation, etc. are frequently employed for industrial effluents (Hussein et al., 2023). However, despite their expeditiousness, these methodologies have proved inadequate in achieving the requisite standards. In light of this predicament, extant treatment procedures have undergone modification and have yielded promising results. At times, physical, biological and chemical processes are combined to optimize contaminant dye extraction (physicochemical). However, for the purposes of this study, biosorption processes have been embraced in light of superior removal strategies, enhanced industrial effectiveness, and ecological friendliness.

Biosorption refers to the process of accumulating or extracting heavy metals from wastewater through either metabolically mediated or spontaneous physicochemical pathways. A range of biosorbents are available, including fungi, chitin, sawdust, sugarcane bagasse, biological algae, and modified materials such as Chitosan and Polyethylene (high density). While the use of biomass for environmental cleanup has been employed for some time, scientists and engineers are optimistic that this approach could serve as a cost-effective alternative for eliminating toxic heavy metals from industrial wastewater and assisting in environmental remediation. Biosorption has the potential to function as an environmentally friendly filtering technique (Manikant et al., 2023). Additionally, Chitosan and polyethylene are among the biological adsorbents utilized for heavy metals removal, without causing detrimental effects on the environment (Pham, 2023). The utilization of biosorption as a means of removing toxic substances presents a promising opportunity in the commercial sphere. Within the field of water treatment technology, biosorption has emerged as a superior alternative due to its numerous advantages, including a minimal ratio of disposable sludge volume, low operating costs, high efficiency in detoxifying very dilute effluents, and in-situ remediation. Significant efforts have been devoted to the development of efficient, effective, economical, and eco-friendly biosorbents, either through direct use or modification with synthetic materials such as polymers, for their waste water treatment applications. The treatment of waste water remains a pressing concern, as dyes pose a continued threat to both human life and environmental well-being. In response to this, selective biosorbents such as Chitosan and Polyethylene bag absorbents have been employed, serving as the foundation for the present study.

2. Materials and method

The entirety of the reagents utilized in this experiment were of analytical grade and were employed without additional purification. These reagents were collected from physical and environmental chemistry department, Federal University of Technology Akure. Among the reagents utilized were Congo red, crystal violet dye, Xylene, Acetic acid, Hydrochloric acid, Sodium Hydroxide, and distilled water, which was utilized in the preparation of all solutions. The experimental apparatus employed included a laboratory vacuum oven, 100mL beaker, 250 mL conical flask, 100mL measuring cylinder, 1000mL standard flask, funnel, reflux heating, filter paper, electrical blender (QBL-18L40), orbital shaker (CETECH KJ-201BD), water bath with shaker, spatula, sample bottles, centrifuge tube, hand-held digital pH meter, UV-Visible spectrophotometer (PEC MEDICAL 721), micro pipette, analytical balance (OPD-104), and refrigerator.

2.1. Adsorbent preparation (low density polyethylene-chitosan nanoparticles biocomposite)

The study involved the synthesis of a biocomposite known as Low Density Polyethylene-Chitosan Nanoparticles (LDPE/CHNP), which was achieved through the modification of the solvent casting method as described by Sandeep et al. (2012). In this process, low density polyethylene (LDPE) pellets were subjected to reflux heating in 50 mL of xylene, while chitosan nanoparticle (CHNP) was dissolved in 10 mL of 0.02 M acetic acid. Gradual mixing of the two solutions in appropriate proportions was carried out, followed by stirring at 100 rpm to form a viscous solution, which was subsequently cooled to 37 ℃ and spread on a Teflon mold. The mold was then placed on a prepared cast and dried under vacuum oven at 35 ℃ to remove any remaining solvent. Once completely dried, the resulting Low Density Polyethylene-Chitosan Nanoparticle (LDPE/CHNP) was blended into powder particles.

2.2. Preparation of solutions

A stock solution of Congo red dye, with a concentration of 1000 mg/L, was created through the dissolution of 1g of the dye in 1000 mL of distilled water using a 1000mL standard flask. Similarly, a stock solution of crystal violet dye, also with a concentration of 1000 mg/L, was produced by dissolving 1g of the dye in 1000mL of distilled water using a 1000mL standard flask.Serial dilutions were performed on the stock solutions of Congo Red and Crystal Violet dyes to prepare working standard solutions. The desired concentrations of the solutions were 10 mg/L, 20 mg/L, 30 mg/L, 40 mg/L, and 50 mg/L. The stock solution was pipetted in volumes of 100µL, 200µL, 300µL, 400 µL, and 500 µL, respectively, and diluted with distilled water in volumes of 9.9 mL, 9.8 mL, 9.7 ml, 9.6mL, and 9.5 mL, respectively. These dilutions were carried out in 45ml centrifuge tubes.

An experiment in biosorption was conducted by means of agitation of varying dosages of biosorption composite (LDPE/CHNP) - 0.01g, 0.02g, 0.03g, 0.04g, 0.05g - with 50mL of methylene blue and bromocresol green solution at different concentrations (10 mg/L, 20 mg/L, 30 mg/L, 40 mg/L, 50 mg/L) over differing time intervals (15, 30, 60, 90, 120 minutes) at a rate of 150 rmp. The resultant solution was subjected to analysis via UV-Visible spectrophotometry.

2.3. Effect of pH

The present study investigates the impact of pH on the biosorption of Congo red and Crystal violet dyes. Six different beakers were used for each dye, into which 0.01g of biosorption composite was weighed. Subsequently, 1.2 mL of 30 mg/L dye solution and 38.79 mL of distilled water were added to each beaker. The pH of the solution was then adjusted within the range of 4.0 to 9.0, using 0.1M of HCl and 0.1M of NaOH. The solution was agitated using an orbital shaker at 150 rmp for an hour. Following this, the sample was removed from the orbital shaker, and the dye solution was separated from the adsorbent using filter paper. The absorbance of the dye solution was measured using UV-Visible spectrophotometer at a wavelength of 492nm for Congo red dye and 592nm for Crystal violet dye.

2.4. Effect of contact time

The present study explores the impact of contact time on the biosorption of Congo red and Crystal violet dyes. To begin, 0.01g of the biosorption composite was accurately weighed and added to 5 separate beakers. Following this, 1.2 mL of a 30 mg/L solution of each dye was added to their respective beakers along with 38.79mL of distilled water. The pH of each sample solution was adjusted to the optimum level using HCl and NaOH. The solutions were then agitated using an orbital shaker for varying time intervals of 15, 30, 60, 90, and 120 minutes, with each sample being withdrawn at the end of each interval. Subsequently, the dye solution was separated from the adsorbent using filter paper. The absorbance of the dye solution was determined using UV-Visible spectrophotometry at respective wavelengths of 492nm and 592nm for Congo red and Crystal violet dyes, respectively.

2.5. Effect of initial concentration

The study investigated the impact of initial concentration on the removal of Congo red and Crystal violet dyes using a biocomposite. To achieve this, 0.01g of the biocomposite was weighed into 5 labeled beakers, with each containing 0.6 mL, 1.2 mL, 1.8 mL, 2.4mL, and 3 mL of different initial dye concentrations of 15mg/L, 30 mg/L, 45 mg/L, 60 mg/L, and 75mg/L. The beakers were subsequently marked with 40 mL and 39.39mL, 38.79mL, 38.19 mL, 37.59 mL, and 36.99 mL of distilled water for Congo red and Crystal violet dyes, respectively. The samples were then adjusted to the optimum pH and shaken on an orbital shaker for 90 minutes and 60 minutes for Congo red and Crystal violet dyes, respectively. The samples were filtered using a filter paper and analyzed with a UV-Visible spectrophotometer at a wavelength of 492nm and 592nm, respectively.

2.6. Effect of adsorbent dosage

The present study examines the impact of adsorbent dosage on the removal of Congo red and Crystal violet dyes. To this end, five beakers were labeled differently with weighed biocomposite dosages of 0.01g, 0.02g, 0.03g, 0.04g, and 0.05g, respectively. Next, 2 mL of 50 mg/L dye solution was pipetted into each beaker and filled up with varying amounts of distilled water, ranging from 37.95 mL to 37.99 mL. The pH of each sample was adjusted using HCl and NaOH to achieve the optimum pH. Subsequently, the samples were agitated for 90 minutes each on the orbital shaker in the case of Congo red dye and for 60 minutes each in the case of Crystal violet dye. Finally, the samples were filtered and analyzed at a wavelength of 492nm and 592nm, respectively, using a UV-Visible spectrophotometer.

2.7. Effect of adsorption kinetic

The current investigation aimed to evaluate the impact of adsorption kinetics on the behavior of Congo red and Crystal violet dyes. To achieve this, 0.01g of the biocomposite was carefully weighed and placed into five distinct beakers.

Subsequently, 2 mL of the dye concentration of 50 mg/L was added to each beaker, and the content was topped up with 40ml of distilled water. The sample was then adjusted to its optimum pH level, followed by agitation using an orbital shaker at various time intervals (15, 30, 60, 90, and 120 minutes). Upon completion of agitation, the samples were filtered, and the filtrate was analyzed at a wavelength of 492nm for Congo red dye and 592nm for Crystal violet dye.

2.8. Effect of temperature

The impact of temperature on the behavior of Congo red and Crystal violet dyes has been studied. Specifically, 0.01g of LDPE/CHNP biocomposite was carefully weighed and placed in a beaker. Subsequently, 2 mL of the stock dye solution was dispensed into the beaker and the solution was filled with 40mL of distilled water. The resulting sample was then subjected to varying temperatures of 30 °C, 40 °C, 50 °C, 60 °C and 70 °C using a water bath for 90 minutes and 60 minutes for Congo red and Crystal violet dyes, respectively. The sample solutions were then filtered and the filtrate was analyzed at a wavelength of 492nm and 592nm for Congo red and Crystal violet dyes, respectively, using a UV-visible spectrophotometer.

3. Results and discussion

3.1. Effects OF pH

The impact of pH on dye biosorption is a crucial aspect that warrants scrutiny. The surface charge of the adsorbent is a pH-dependent factor that, in certain cases, affects the structure of the adsorbate species and, thus, the pH of the solution. Consequently, this study sought to investigate the effects of initial pH on dye adsorption across a pH range of 4 to 9 for both CR and CV. As depicted in Figure1, for CV, the percentage of dye removed decreased from pH 4 to 5, reaching a minimum at pH 4, and progressively increased from pH 4 to 9, where 61% of the dye was adsorbed. For CR, the minimum dye adsorption by the adsorbent was observed at pH 6, which then increased progressively from pH 6 to 7, and decreased from pH 7 to 9, dropping from 64% to 62%. Additionally, it was observed that CV had a higher adsorption rate than CR. A plausible explanation for this observation in pH-related adsorption is the point of zero charge (pHpzc) of the adsorbent. The pHpzc is the point at which the adsorbent is neutral, beyond which the material becomes either positively or negatively charged.

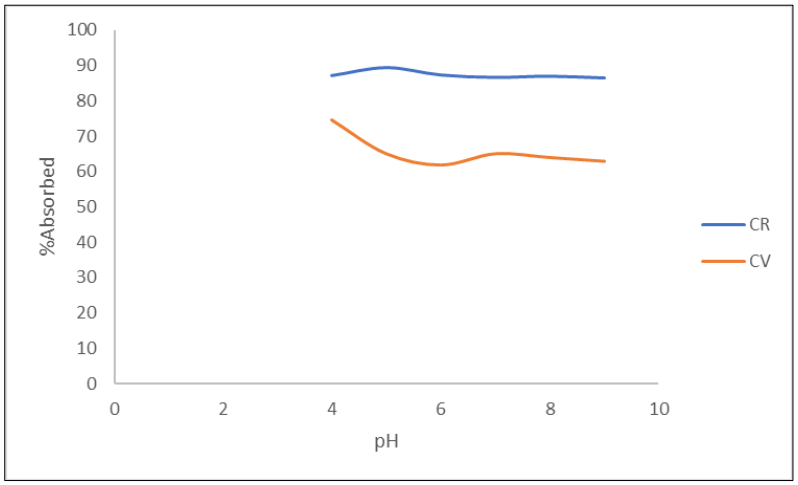


Figure 1 Effect of pH on the Biosorption of Congo Red and Crystal Violet using chitosan and polyethene bags as adsorbent. Initial concentration= 30 mg/L, Agitation speed = 150rpm, Contact time = 30min., Absorbent dosage = 0.1g. CR= Congo Red CV= Crystal violet

3.2. Effects of contact time on adsorption on congo red and crystal violet

The impact of duration of contact was scrutinized by utilizing various timeframes, a set adsorbent dose, and a fixed initial dye concentration. In general, the removal rate of dyes escalated with an increase in contact time until the establishment of equilibrium adsorption. Nonetheless, as depicted in Figure 2, each dye demonstrated a distinctive percentage of dye removal at equilibrium, whereby CR exhibited a lower percentage than CV, with the latter achieving its maximum percentage adsorption of 97.4%. Upon reaching equilibrium for CR and CV dyes, the adsorption process did not further augment due to the deposition of dyes on the available adsorption sites on the adsorbent material (Ansari and Mosayebzadeh, 2011). At this juncture, the quantity of desorbing dye from the adsorbent is in a dynamic

equilibrium state with the amount of dye being adsorbed onto the adsorbent. Moreover, for adsorption of CR and CV, the equilibrium point was attained in 50 minutes.

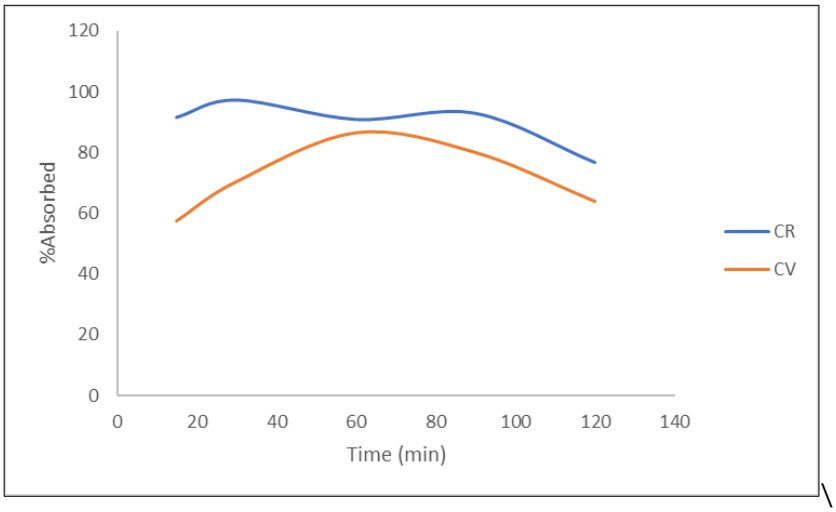


Figure 2 Effect of contact time on Biosorption of Congo Red and Crystal Violet using chitosan and polyethene bags as adsorbent. Initial concentration= 30 mg/L, Agitation speed = 150 rpm, Contact time = 30min., Absorbent dosage = 0.1g

3.3. Thermodynamics study on adsorption of cv and cr

Table 1 Thermodynamics parameters of adsorption of CR and CV onto LDPE/CHNP adsorbent

Thermodynamics parameter Δg(kj/mol}	**Δg congo red**	**Δg crystal violet**
303	-5.892	-4.691
313	-5.631	-4.873
323	-4.767	-5.747
333	-4.557	-7.01
343	-4.487	-7.353
ΔS (JK-1mol-1)	-39.4558	74.08508
ΔH (KJmol-1)	17.81131	-17.9947

The examination of the temperature-dependent behavior of adsorption reactions is a highly informative approach for determining the enthalpy and entropy alterations that occur during the adsorption process. The temperature is an essential indicator of the adsorption nature, as it provides insight into whether the process is exothermic or endothermic (Wongwailikhit and Horwongsakul, 2011). In this study, the thermodynamic parameters (ΔG, ΔH and ΔS) of the adsorption of CR and CV were ascertained. The values of ΔS and ΔH were computed from the slope and intercept of the graph plots listed in (Table 1). The observed negative values of free energy, ΔG, imply the feasibility of the process and its spontaneity in nature. However, the negative values of ΔH also suggest that the adsorption process is exothermic in nature. Conversely, the positive value of ΔS indicates that the randomness increases with the progress of the adsorption process. It was observed from the findings of the study that the ΔG of CV was higher than that of CR, indicating that the adsorption of CR is less spontaneous than CV.

3.4. Adsorption isotherm

The Langmuir model, which is associated with a monolayer homogeneous adsorbent surface, and the Freundlich model, which corresponds to a heterogeneous adsorbent surface, are the most commonly used adsorption models. However, in this particular experimental study, the isothermal results were analyzed using well-established expressions for Langmuir and Freundlich isotherms. As demonstrated in Figure 3, a plot of Ce/Qe against Ce aligns with the linearized Langmuir equation. The correlation coefficient R2 values for both CR and CV are 0.9998 and 0.9994, respectively. This

indicates that the adsorption of the dyes onto the LDPE/CHNP adsorbent conforms to the Langmuir isotherm, which suggests that the adsorption is a monolayer homogeneous adsorption. In addition, the Langmuir model, which corresponds to a monolayer homogeneous adsorbent surface, and the Freundlich model, which corresponds to a heterogeneous adsorbent surface, are the most frequently used adsorption models. Furthermore, in this particular experimental study, the isothermal results were analyzed using well-known expressions for Langmuir and Freundlich isotherms. As illustrated in Figure 3, a plot of Ce/Qe against Ce corresponds with the linearized Langmuir equation. The correlation coefficient R2 values for both CR and MR are 0.9995 and 0.9954, respectively. This confirms that the adsorption of the dyes onto the LDPE/CHNP adsorbent is consistent with the Langmuir isotherm, which may suggest that the adsorption is a monolayer homogeneous adsorption.

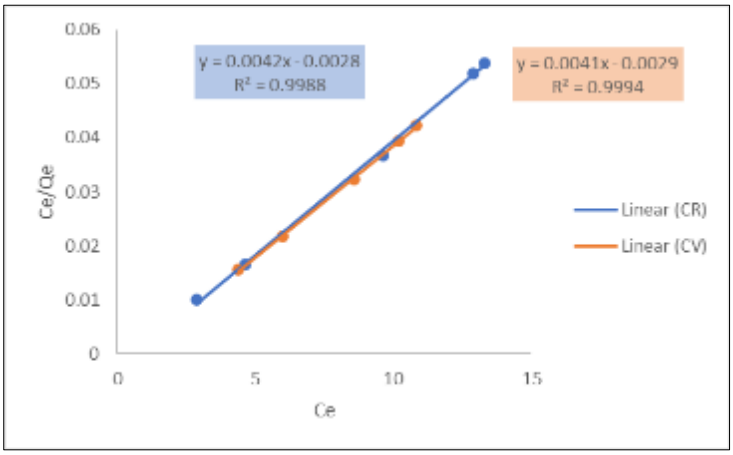


Figure 3 langmuir isotherm for removal of CR and CV (Adsorbent dosage 0f 0.1g, temperature of 300k, agitation speed of 150 RPM, contact time: 15, 30, 45, 60 and 75)

3.5. Adsorpton kinetics

3.5.1. Pseudo first order kinetics

The Lagergren pseudo first-order, Lagergren pseudo second-order, and intra particle diffusion models are widely accepted as the most popular kinetic models. Upon examining the experimental data (Figure 4), it was observed that the pseudo-first-order model did not fit well with an R2 value of less than 0.7923. The calculated qe values were not in agreement with the experimental qe values, which leads to the conclusion that the adsorption of both dyes onto LDPE/CHNP does not conform to first-order kinetics. This finding is further supported by Ashoka and Inamdar's (2010) research on the adsorption removal of methyl red from aqueous solutions using treated sugar cane bagasse and activated carbon.

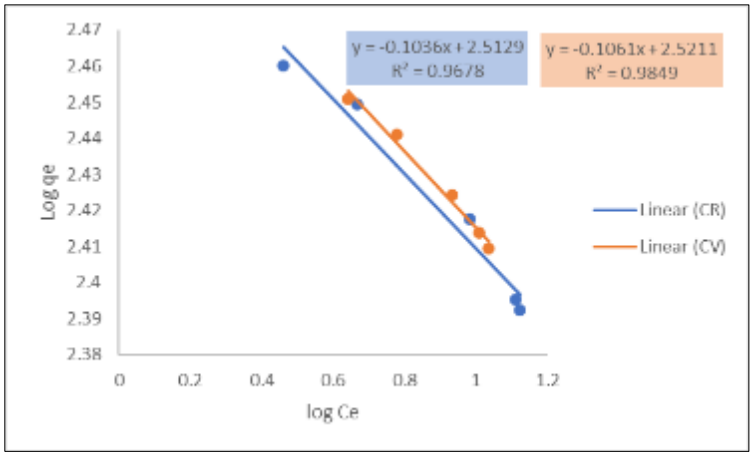


Figure 4 Freundich isotherm for removal of CR and CV (Adsorbent dosage 0f 0.1g, temperature of 300k, agitation speed of 150RPM, contact time: 15, 30, 45, 60 and 75)

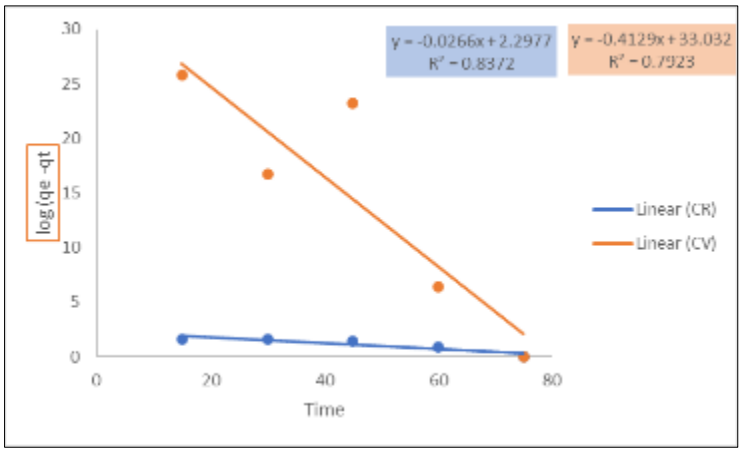


Figure 5 Pseudo first order kinetics for the removal of CR and CV (Adsorbent dosage 0f 0.1g, temperature of 300k, agitation speed of 150RPM, contact time: 15, 30, 45, 60 and 75)

3.5.2. Pseudo second-order kinetics

The adsorption of CR and MR onto LDPE/CHNP was analyzed through kinetic modelling, employing the Lagergren pseudo-second-order model as shown in Figure 6. This model is based on the premise that the rate-limiting step is likely to involve chemisorption, which may occur through the sharing or exchange of electrons with the N atom of the adsorbent. The second-order kinetics are commonly referred to as the two-site or bicontinuum models, where two reactions are assumed to occur either in parallel or series. One of the reactions is characterized as rapid, while the other is slower until it reaches equilibrium, which may occur over an extended period of time (Yeşim and Başak, 2016).

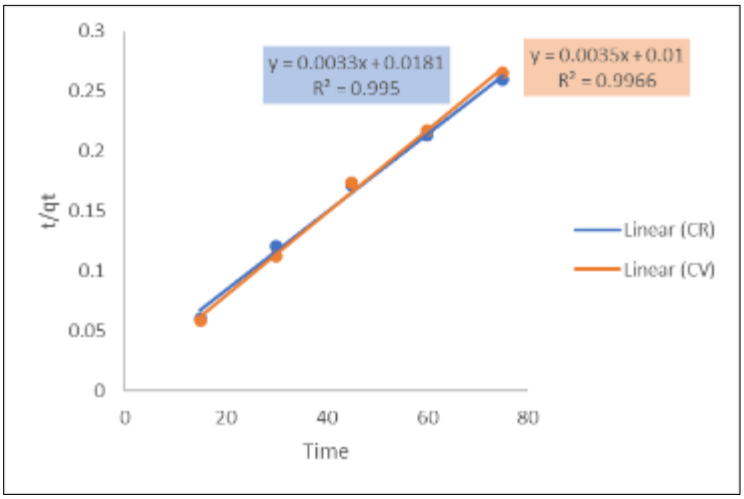


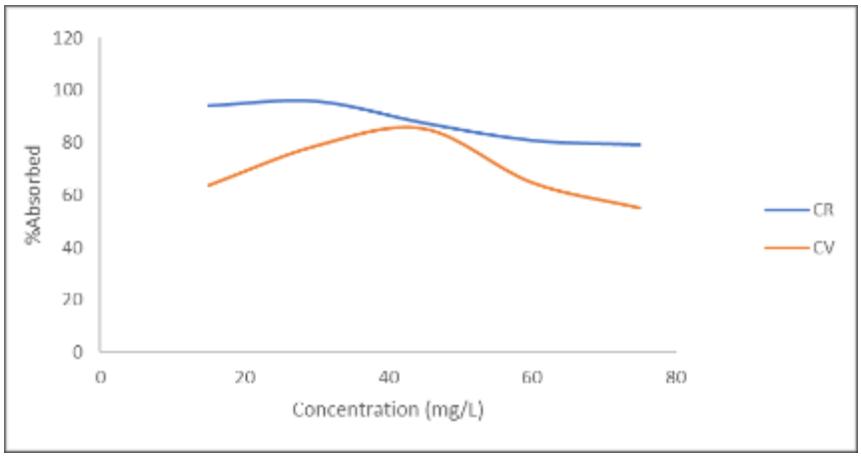
Figure 6. Pseudo second order kinetics for the removal of CR and CV (Adsorbent dosage 0f 0.1g, temperature of 300k, agitation speed of 150RPM, contact time: 15, 30, 45, 60 and 75)

Table 2 Kinetics study and adsorption of CR and CV

	Pseudo first order				**pseudo second order**	
	K1	**R2**	**K2**	**qe**	**h**	**R2**
CV	0.027	0.8372	10.9	0.259	0.045	0.0995
CR	0.392	0.7923	12.89	0.248	0.03	0.9966

3.6. Effect of initial concentration

In Figure 7, it was observed that an increase in the initial concentration of Congo red resulted in a relationship between %removal and adsorption with untreated adsorbent, while demonstrating a common relationship with treated adsorbent. The decrease in %removal can be attributed to a limited number of available sites on the adsorbent at higher concentrations. Conversely, the adsorption capacity increased as the dye concentration increased due to the utilization of all available adsorption sites at higher concentrations (Khan et al., 2011). As for CV, the amount adsorbed increased as the concentration increased, although the %removal adsorbed decreased from 95.8% to 79.1%. At lower initial concentrations, CV tends to bind to the surface of negatively charged ions, resulting in a significant amount of adsorption.

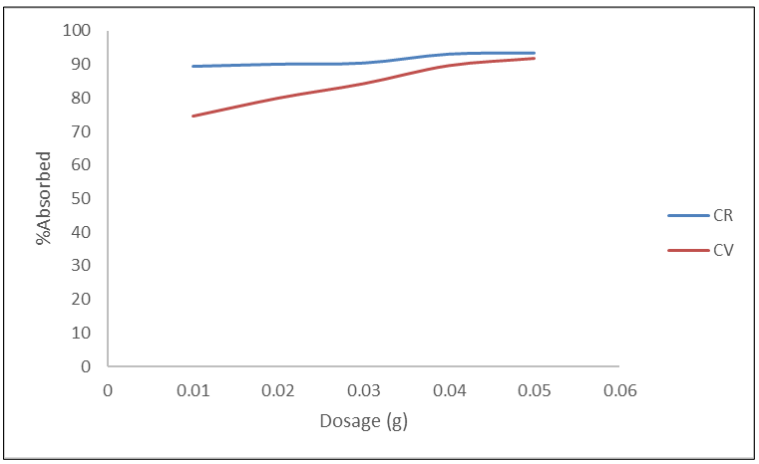


pH = 8.0, agitation speed 150rpm, contact time 30min, adsorbent dosage =0.1g.

Figure 7 Effect of the initial concentration on removal of Congo red dye and crystal violet

3.7. Effect of adsorbent dosage

The effect of adsorbent dosage on the adsorption of Congo red dye was investigated. An increase in adsorbent dosage was found to result in an increase in the amount of adsorbed dye due to the corresponding increase in the available surface area of the adsorbent, which in turn increases the number of available binding sites. This observation is supported by Figure 7, which shows that the adsorption of Congo red dye decreases only slightly with increasing adsorbent dosage from 0.01g to 0.05g. However, at an adsorbent dosage of 0.02g, a drop in %removal was observed, which can be attributed to the overlapping of the adsorption sites. Additionally, the results from the study of crystal violet indicate that as the adsorbent dosage increases, the affinity to bind with the binding sites decreases, which may be due to the overlapping of negatively charged ions on the surface of the adsorbent (Gebrehawaria, 2016).



pH = 8.0, agitation speed 150rpm, contact time 30min, adsorbent dosage =0.1g.

Figure 8 Effect of adsorbent dosage on removal of congo red dye and crystal violet

4. Conclusion

Chitosan, a minor derivative of a biodegradable natural polymer, was incorporated into synthetic polymer LDPE through the solvent casting method. The LDPE/CHNP biocomposite was subsequently examined for its efficacy in the removal of Congo red dye and crystal violet from waste water. The impact of various experimental parameters that affect the efficiency of the adsorbent were assessed and optimized. Given that waste water from textile and dye industries typically contains numerous organic compounds, many of which are colored, this study sought to remediate both crystal violet and Congo red dye simultaneously from waste water. The results of the investigation demonstrate that the LDPE/CHNP adsorption capacity is effective in removing congo red dye and crystal violet. Kinetic data obtained from the adsorption of Congo red and crystal violet were well-fitted with the pseudo first order model, while the kinetic data obtained from the adsorption was also well-fitted with the pseudo second order model.

Compliance with ethical standard

Acknowledgement

This work enjoys the cooperation of Babafemi Raphael Babaniyi.

Disclosure of Disclosure of conflict

I do not have any financial benefit to declare.

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