Adsorption of Brilliant Blue Dye on Biochar from the Macroalgae Cladophora glomerata

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Abstract

Here we describe the utilization of freshwater macroalgae *Cladophora glomerata* (*C. glomerata*) biochar at comparatively low cost for the removal of brilliant blue dye (BBD) from aqueous solutions. The macroalgae was successfully converted into biochar through thermal pyrolysis. The chemical and morphological nature of the biosorbent was analyzed through Fourier Transform Infrared (FTIR) spectroscopy, scanning electron microscopy and thermogravimetric analysis. The effect of pH, initial concentration and adsorbent dosage were investigated. The removal of brilliant blue increased linearly with increasing adsorbent dose, concentration, and contact duration. Optimized conditions for adsorption were pH 4, contact time of 150 minutes, and adsorbent dosage of 3.0 g. Langmuir isotherm best explained the adsorption data and required parameters and appropriate constants were calculated. These results suggest that *C. glomerata* may remove dye from aqueous solutions.

1. Introduction

Cladophora glomerata is the most omnipresent algae in freshwater habitats throughout the globe, existing in Europe, North America, Asia, Africa, Australia, New Zealand, Islands of the Pacific and Atlantic, and the Caribbean. *Cladophora* blooms were abundant in the lower North American Great Lakes in the 20th century, but they were mostly destroyed thanks to a multibillion-dollar phosphorus abatement drive [1, 2].

Biomass is seen as one of the most viable alternatives to fossil fuels, and it is attracting increasing attention. Construction waste, municipal solid waste, crop stems, various herbs, dead wood and forest biomaterial waste and other biomass can all be converted to biofuels. Following the production of first and second-generation biofuels from food crops and lignocellulose biomass, third-generation renewable fuel is mostly made from algal sources found in fresh and marine water [3–5].

C. glomerata is the most well-known and broadly distributed macroalgae in the Baltic Sea's coastal zone. *Cladophora*, a Cladophorophyceae member with green algae clusters, is distinguished by fast development and a highly bifurcated threaded multicellular thallus that can regrow and that can reach lengths of many tens of centimeters to several meters. This species habitat requirements are contingent on the presence of various salts of nitrogen and phosphorus, as well as enough water aeration [6]. Thermal stability is a critical consideration when dealing with biomass, as it is made up of hemicellulose, cellulose, and lignin. Lignocellulose structure and thermochemical behavior of marine biomasses differ greatly from that of terrestrial and lignocellulose biomasses, as well as each other. Protein, glucose, and simple fats are also present [6, 7].

The overproduction of macroalgae biomass makes them a suitable source for biochar production through various pyrolysis processes. At around 550°C, pyrolysis of Cladophora generated gas (30%), bio-oil (39%), and biochar (31%) [8, 9]. This biochar has potential use in agriculture because it is rich in elements like Ca, Mg, P, and K [8, 9]. The Cladophora biochar is helpful in soil enrichment, enhancing its

water holding potential, increasing overall nutrient presence, and augmenting the presence of useful microbes [8, 9]. Acid washed *C. glomerata* biomass when pyrolyzed in the presence of zinc chloride produces highly porous biochar [10]. For example, an olive size magnetically active biochar can be made from Cladophora using low heating rate for pyrolysis through iron based catalysts [11]. The particle size of this char has a larger surface area of 296.4 m^2 g^{-1} [11].

It has been proposed that algal biochar can be used as an electrode in several types of modern batteries. This suggest that algae biochar is a promising material for future technological advancements in various electric vehicles and large electric storage machines [12]. The prominent and desirable pore size of the magnetic biochar produced from *C. glomerata* in combination with iron oxide helps in the production of asymmetric super-capacitors [13]. The electrodes made with magnetically active biochar have more capacitance and a longer life when used in electric storage equipment [13]. Heavy metals also adsorb well on *C. glomerata* biochar [14, 15]. Moreover, *C. glomerata* biochar adsorbs cationic dyes and can be regenerated several times [16]. The aim of the current work is to determine the brilliant blue dye (BBD) adsorption capacity of *C. glomerata* biochar. This adsorption capacity was evaluated for different factors, to understand how contact time, pH, adsorbent dosage and concentration of the dye influence dye removal.

2. Materials And Methods

2.1 Collection and Pretreatment

Cladophora glomerata biomass was collected from the Jindi River in the Sherpao area in the Charsadda district of Pakistan. The species was identified by professor Arshad Iqbal in Department of Botany, Islamia College Peshawar. The collected algae was washed with tap water followed by distilled water to remove mud or sand particles as performed previously for various algae [17]. The biomass material was dried and powdered with a grinder and stored in airtight jars.

2.2 Conversion of Biomass into Biochar

For biochar preparation, known weight of dried powdered *Cladophora glomerata* was taken in crucibles and were placed in furnace, the temperature of the furnace was set at 350°C with 10° C per mint rise. After this, the furnace was turned off and the crucibles were allowed to cool down, the macroalgae was converted into black color biochar. Then all these biochar from the crucibles were shifted to a glass jar for storage and for later use in research.

2.3 Fourier Transform Infrared Spectroscopy

The FTIR spectrum was taken for the biochar to check the existence of different chemicals represented by various functional groups that will interact with the adsorbing dye. The spectrum was recorded from 500 cm^{-1} to 4000 cm^{-1} of frequency window.

2.4 Scanning Electron Microscopy

2.5 **Thermogravimetric Analysis**

2.6 **Adsorption Studies**

A series of solutions were prepared (10, 20, 30, 40, 50, 60, 70 ppm) in a 250 mL flask and 100 mg of biochar was taken for each solution. The pH was adjusted from 2–10 and temperature was in the range of 298-328K respectively. The mixtures were shaken for 2 hours with 150 rpm. After 2 hours the solutions were filtered via Whatman filter paper. Then absorbance of initial and final solutions of 10 to 70 ppm was recorded at wavelength of 629 nm via UV-Vis spectrophotometer.

3. Results And Discussion

3.1 Spectrophotometric Analysis

The spectrophotometric determination of brilliant blue dye was carried out using an already reported method. For this, we prepared various solutions of selected adsorbate with known concentrations (10, 20, 30, 40, 50, 60 and 70 ppm). The absorbance was determined at 629 nm, and showed that an increase in concentration led to an increase in absorbance. A straight-line equation was used in order to find the unknown concentration of adsorbate i.e. $y = mx + c$, where y is absorbance, m is the slope and c is the intercept. The slope m and correlation coefficient (R^2) were 0.0542 and 0.9835 respectively (Fig. 1 and Table 1).

Table 1
Absorption data of brilliant blue dye at different concentrations.

S. No	Concentration (ppm)	Absorbance
1	10	0.471
2	20	0.994
3	30	1.646
4	40	2.056
5	50	2.978
6	60	3.246
7	70	3.580

3.2 FTIR Spectrum of the Biochar

An FTIR spectrum is presented in Fig. 2, displaying intense characteristic bands of *Cladophora glomerata* biochar, indicating the presence of functional groups. Major peaks in *Cladophora glomerata* biochar were noted in the region of 1600 – 400 cm^{-1}, the fingerprint region (Fig. 2). The FTIR spectrum of *C. glomerata* biochar shows a peak at 3308 cm^{-1} which was assigned to the stretching vibrations of the O-H group. In

the range of 2850–2970 cm^{-1} in the spectrum, there was no peak found, but mostly at 2850–2970 cm^{-1} peaks are attributed to the stretching vibrations of C-H bonds in aliphatic CH_2 and CH groups. The peaks in the range of 1960 – 1680 cm^{-1} are attributed to C-C stretching, and the peaks in the range of 1000–1350 cm^{-1} are assigned to C-O stretching. Those observed in the range of 910 – 650 cm^{-1} correspond to aromatic C-H bonds (Table 2). The different functional groups present on the surface and pores of the algae biochar are suitable chelating agents not only for heavy metals but they also interact with organic pollutants through various hydrophobic interactions and help in maximum adsorption [18]. The activation of N-C bonds in algae biochar by some chemical agents make them suitable carbocatalysts to generate atomic oxygen that can oxidize various harmful dyes and thus initiate their degradation in the environment [19].

Table 2
FTIR bands and functional group attributions.

Wavenumber	Assignment
3308.07 cm^{-1}	O-H
1398.38 cm^{-1}	O-H
1105.53 cm^{-1}	C-O
1035.25 cm^{-1}	C-O
872.84 cm^{-1}	Aromatic C-H
779.39 cm^{-1}	Aromatic C-H
616.52 cm^{-1}	Aromatic C-H
463.58 cm^{-1}	C-I

3.2 Elemental Composition and SEM Analysis

The elemental composition of the *C. glomerata* biochar showed that it conatins carbon, oxygen, iron, sodium, magnesium, aluminium, silicon, sulphur, chlorine, calcium as the major elements. These results are in close agreement with our previous results of *Cladophora* biochar [5]. Figure 3 showed the SEM images of the biochar formed at 450°C. A non-uniformed particulated structure can be observed under the electron microscope. The biochar of the Cladophora varies when the biomass is treated with different chemicals or the addition of different metals caused different shape particles of the biochar [15]. These pre-treatment and addition of metals can enhance the adsorption capacity due to different pore size of the biochar formed.

3.3 Thermalgravimetric Analysis

The thermogrvimetric analysis showed the weight loss as temperature increase from 25°C to 750°C with a heating rate of 30°C min^{-1}. It can be observed from Fig. 4, that the weight loss before 100°C is that of the moisture present in the dried biomass that eveaportes when temperature rises from room temperature to 100°C. The major weight loss observed from 200–500°C range is that of polysaccharides, protein and lipids that were devolatilized to low molecular weight compounds. The other remaining bio-organic molecules are further degarded in the second major pyrolysis step from 600°C to 700°C. Thus from room temperature to around 750°C around 60% of the biomass was devoltilized. The TGA/DTG analysis showed that a temperature of 350°C is suiatble for Cladophora biomass transformation into biochar that can be used for adsorption purposes. The TGA data obatined here was in close agreement with previous TGA analysis of Cladophora biomass performed at various heating rates [3, 5, 11].

Factors Affecting Adsorption

3.2.1 Effect of Concentration

The adsorption of brilliant blue dye with different concentrations was investigated using a series of concentrations (10, 20, 30, 40, 50, 60 and 70 ppm). The optimum value for brilliant blue dye was 70 ppm (Fig. 5). The adsorption increased substantially with increased concentration, due to an increase in the number of available active sites for 70 ppm concentration. Hence, 70 ppm was selected as the optimum concentration for brilliant blue dye. The results were plotted as concentration vs qe vs % R. The average and standard deviation of triplicate values were taken and error bars were applied on both graphs (Fig. 6). The increase of the dye concentration always has a limit on the adsorption capacity of the algae biochar. The monolayer of the dye saturates the surface of the biochar and it cannot take up extra dye to adsorb on its surface [20]. Pretreatment like washing of the macroalgae also increases the capacity of biochar to release less amount of metals during the adsorption process and adsorb higher amounts of dye and organic pollutants [21].

3.2.2 Effect of pH

The pH has a substantial effect on adsorption, as pH impacts the properties of the adsorbent and adsorbate molecules, such as the adsorbent's surface charge and the adsorbate's ionization degree. Thus, the influence of pH on the adsorption of brilliant blue dye onto *C. glomerata* biochar was investigated in the pH range of 4 to 10. It was discovered that adsorption decreased with increasing pH and at pH 4 the *C. glomerata* biochar adsorbent exhibited a maximal removal rate of 98.3 percent for 70 ppm brilliant blue dye. The major chemical groups on the *C. glomerata* biochar adsorbent become protonated in the presence of H^+. Subsequently, the brilliant blue is dissolved in water, where its sulfonate groups (Dye-SO_3Na) are dissociated and transformed to anionic dye ions (Dye-SO_3Na). The cationic *C. glomerata* biochar and the anionic brilliant blue dye ions exhibit considerable electrostatic interactions in this scenario. When the pH of the solution was increased to 10, the adsorption efficiency reduced considerably. The decrease in dye adsorption at pH greater than 4 is due to the reduction of positively charged sites on the surface of the *C. glomerata* biochar in alkaline media. Additionally, when the pH of

the solution increased, competition for adsorption on the active sites of *C. glomerata* biochar occurred between anionic brilliant blue dye ions and hydroxyl ions. The primary adsorption mechanism may be assumed to be electrostatic attraction between the charged surface and the charged dye molecule. At low pH, the dye adsorption on the adsorbent *C. glomerata* charcoal may be enhanced due to the neutralization of the negative charge on its surface, increasing protonation. This improves diffusion by supplying more of the adsorbent's active surface, resulting in increased adsorption at the adsorbent's surface. The decrease in dye adsorption with increasing pH could be attributed to deprotonation, which inhibits diffusion and so decreases dye adsorption until pH 10. As a result, the dye is most readily absorbed at a pH of roughly 4.0 and least readily absorbed at a pH of 10. The maximum amount of dye eliminated (98.3 percent) occurred at pH 4 and decreased until pH 10. (49.8 percent). At acidic pH, the increased adsorption of brilliant blue dye may also be attributed to electrostatic interactions between the negatively charged dye molecules and the positively charged biochar surface. The surface charge density drops as the solution pH increases, the biochar surface becomes negatively charged, rejecting the negatively charged dye molecules, resulting in a decrease in dye adsorption rate at basic pH. Increased electrostatic attraction between the negatively charged dye and negatively charged activated carbon may result in a decrease in the degree of dye molecule adsorption. The result was plotted as pH vs qe and % removal (Figs. 7 and 8, Table 3). The average and standard deviation of triplicate values were taken and error bars were applied on both graphs.

Table 3
Effect of pH on adsorption of brilliant blue dye.

S. No	pH	q_e	%R
1	4	68.81	98.30
2	5	67.97	97.10
3	6	66.04	91.65
4	7	57.82	82.60
5	8	52.54	75.06
6	9	51.79	60.96
7	10	46.51	49.82

3.2.3 Effect of Dosage

The effect of dosage of adsorbent on adsorption of brilliant blue dye was studied using different *Cladophora glomerata* biochar dosages in the range of 0.05g, 0.1g, 0.15g, 0.2g, 0.25g and 0.3g. It was confirmed that the maximum adsorption of brilliant blue dye occurs at a dose of 0.3g adsorbent, which contains the maximum number of active sites. The amount of adsorbent used is a critical experimental parameter for determining the capacity of adsorbents to adsorb dyes. The percent removal of 70 ppm brilliant blue dye ions increased from 41.5 to 86.9 percent when the adsorbent dosage was raised from

0.15 to 0.3 g. The result is consistent with earlier investigations, and this can be attributed to the adsorbent's increased number of active binding sites and surface area due to the adsorbate, which is a brilliant blue dye. The dose of adsorbent for adsorption of brilliant blue dye on biochar was found to be 0.3 g, which removed 86.9 percent of the dye at a concentration of 70 ppm. The result was plotted as adsorbent dose vs qe and % R. Average and standard deviation of triplicate values were taken and error bars were applied on both graphs (Figs. 9 and 10, Table 4). A similar effect of dosage has been observed for the brown algae *Ascophyllum nodosum* biochar activated with zinc chloride, which can adsorb up to 400 mg per g of ciprofloxacin [22].

Table 4
Effect of dosage on the adsorption of brilliant blue dye.

S. No	Adsorbent dose	q_e	%R
1	0.05g	58.1	41.5
2	0.1g	54.4	77.7
3	0.15g	39.0	83.7
4	0.2g	29.6	84.5
5	0.25g	24.32	86.8
6	0.3g	20.38	86.91

3.2.4 Effect of Contact Time

Contact time is a critical aspect in the batch adsorption process since it has a significant effect on the dye removal. The effects of contact time on the adsorption of brilliant blue dye by *C. glomerata* biochar was investigated from 25 to 150 minutes at a dye concentration of 70 ppm using a fixed biochar dosage (0.1 g). The adsorption of brilliant blue dye increased with the contact duration and reached a maximum value at 150 minutes. When the contact time was increased from 25 to 150 minutes, the brilliant blue dye's adsorption percentage increased from 44 to 85 percent. Thus, a contact period of 150 minutes was determined to be ideal for studying the adsorption of brilliant blue dye. The result was plotted as contact time vs q_e vs % R (Figs. 11 and 12, Table 5). Average and standard deviation of triplicate values were taken and error bars were applied on both graphs. The biochar of *Chlorella pyrenoidosa* can adsorb about 80% of Direct Red 31, a diazo dye in 180 minutes of contact time [23]. This showed that Cladophora biochar is quite superior than other algae biochar for adsorption of dyes.

Table 5
Effect of contact time on adsorption of brilliant blue dye.

S. No	Time (min)	q_e	%R
1	25	30.77	44.0
2	50	45.6	65.2
3	75	51.2	73.1
4	100	53.6	76.67
5	125	58.4	83.5
6	150	59.5	85.019

3.4 Adsorption Isotherms

The experimental adsorption data can be described using equilibrium isotherm equations. The features provided by the various models show the adsorption mechanisms, as well as the surface properties and affinities of the adsorbent. The Langmuir and Freundlich models are the most commonly accepted surface adsorption models for single solute systems.

3.4.1 Langmuir Isotherm

This isotherm holds true for the adsorption of a solute from a liquid solution monolayer adsorption on a surface with a finite number of identical sites. The Langmuir isotherm model assumes uniform adsorption energies on the surface and the absence of adsorbate transmigration in the plane of the surface. The Langmuir isotherm model was used to calculate the highest adsorption capacity corresponding to complete monolayer coverage on the adsorbent surface. The Langmuir equation is frequently represented in the following manner:

$$\frac{1}{qe} = \frac{1}{Klqmax} \cdot \frac{1}{Ce} + \frac{1}{qmax} \quad \text{..... (1)}$$

Where, q_m is monolayer adsorption capacity, K_L is Langmuir isotherm constant which is related to binding site affinity and adsorption energy. By plotting 1/qe vs 1/ce, the values of q_m and K_L were calculated (Fig. 13). A dimensionless constant separation factor or equilibrium parameter, RL can be used to characterize the key properties of a Langmuir isotherm.

$$R_L = \frac{1}{1+KlCe} \quad \text{..... (2)}$$

The effect of isotherm shape on favorable and unfavorable adsorption has been studied. Unfavorable (RL > 1), linear (RL = 1), favorable (0 > 1), or irreversible (RL = 0) isotherm types are indicated by the RL values. In this experiment, the findings for RL were determined in Table 6 to be between 0.649 to 0.209, indicating that the adsorption was favorable. The Langmuir isotherm are mostly suitable model for

adsorption of azo dyes on macroalgae biochar as reported for char produced from *Eucheuma spinosum* biomass [24].

Table 6
Langmuir constants for adsorption of brilliant blue dye.

Langmuir Constants	
Intercept	0.01707
Slope	0.3116
q_{max}	58.5823
K_L	0.054
R^2	0.9613

3.4.2 Freundlich Isotherm

The Freundlich equation was used to determine the adsorption of brilliant blue dye on *C. glomerata* biochar. The Freundlich isotherm was represented by the following:

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

Where q_e is the amount of brilliant blue dye adsorbed, Ce is the equilibrium concentration of dye in solution, K_f and n are constants that take into account the parameters that affect adsorption capacity and intensity, respectively. The adsorption of brilliant blue dye follows the Freundlich adsorption isotherm as shown by linear graph, which is plotting of log q_e vs log C_e (Fig. 14). According to Table 7, the values of K_f and n for adsorption capacity and intensity of adsorption were 2.949 for adsorption capacity and 0.911 for intensity for adsorption. The results indicated that there is better fitting of the Langmuir isotherm model than the Freundlich isotherm. The R^2 will also describe which model is best for experimental data, the values of R must be close to 1, in case of Langmuir isotherm the R^2 is 0.9613 and in case of Freundlich isotherm the R^2 is 0.9575, so the R^2 is closer to 1 for Langmuir compared to Freundlich isotherm. Thus, the Langmuir isotherm best explains the obtained data.

Table 7
Freundlich constants for adsorption of brilliant blue dye.

Freundlich Constants	
Intercept	0.4698
Slope	0.911
1/n	0.911
K_f	2.949
R^2	0.9575

Conclusions

In this work, thermal pyrolysis was utilized to convert a freshwater macroalgae, *Cladophora glomerata*, into biochar. It was used as an adsorbent to remove brilliant blue dye from aqueous solution. Adsorption tests were conducted at various concentrations, contact time, dosage, and pH values. Except for pH, where adsorption reduces with increasing pH, the data indicated that the removal of brilliant blue increases linearly with increasing adsorbent dose, concentration, and contact duration. The efficiency was enhanced when the pH was set to 4, but decreased when the pH was set to 10. Because the adsorption capacity increases significantly with increasing concentration due to the increase in the number of accessible active sites, a concentration of 70 ppm was chosen as the optimal concentration. It was observed that as the adsorbent dosage was increased from 0.15 to 0.3 g, the %R of 70 ppm brilliant blue dye ions increased from 41.5 to 86.9 percent. It was confirmed that the highest adsorption of brilliant blue dye occurs at a 0.3g adsorbent dosage. When the contact time was increased from 25 to 150 minutes, the brilliant blue dye's adsorption percentage increased from 44 to 85 percent. The adsorption of brilliant blue dye increases as the contact time increases and reaches a maximum value at 150 minutes. As a result, a contact time of 150 minutes was determined to be ideal. The adsorption isotherm studies revealed that the Langmuir isotherm model describes the adsorption of brilliant blue better than the Freundlich isotherm model. Additionally, estimated RL values indicated that brilliant blue adsorption onto biochar was favorable. The use of *C. glomerata* biochar as a potential adsorbent for dye removal from wastewater is promising and can be used for the removal of toxic dyes and other organic pollutants.

Declarations

Ethical Approval

Not Applicable

Competing interests

All the authors declare that they have no competing interests.

Authors' contributions

Azwa Areej, Maimoona Mumtaz and Fariha Aman performed the experimental work and write the initial manuscript with Syed Lal Badshah and Asfar Khan. Abdul-Hamid Emwas and Mauisz Jaremko improved the draft and final editing.

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Availability of data and materials

Data will be provided upon request from the authors.

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Figures

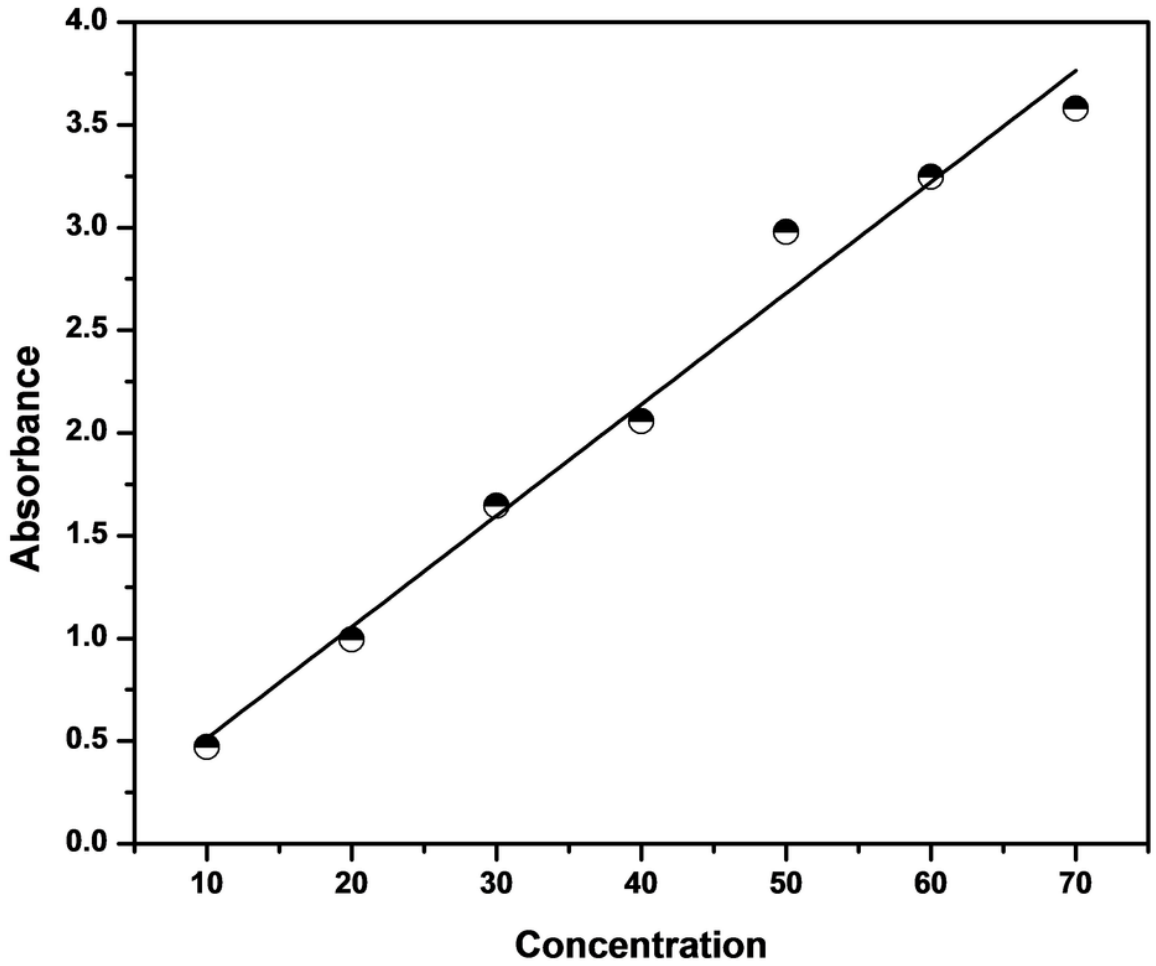

Figure 1

Spectrophotometric determination of brilliant blue dye absorbance vs concentration.

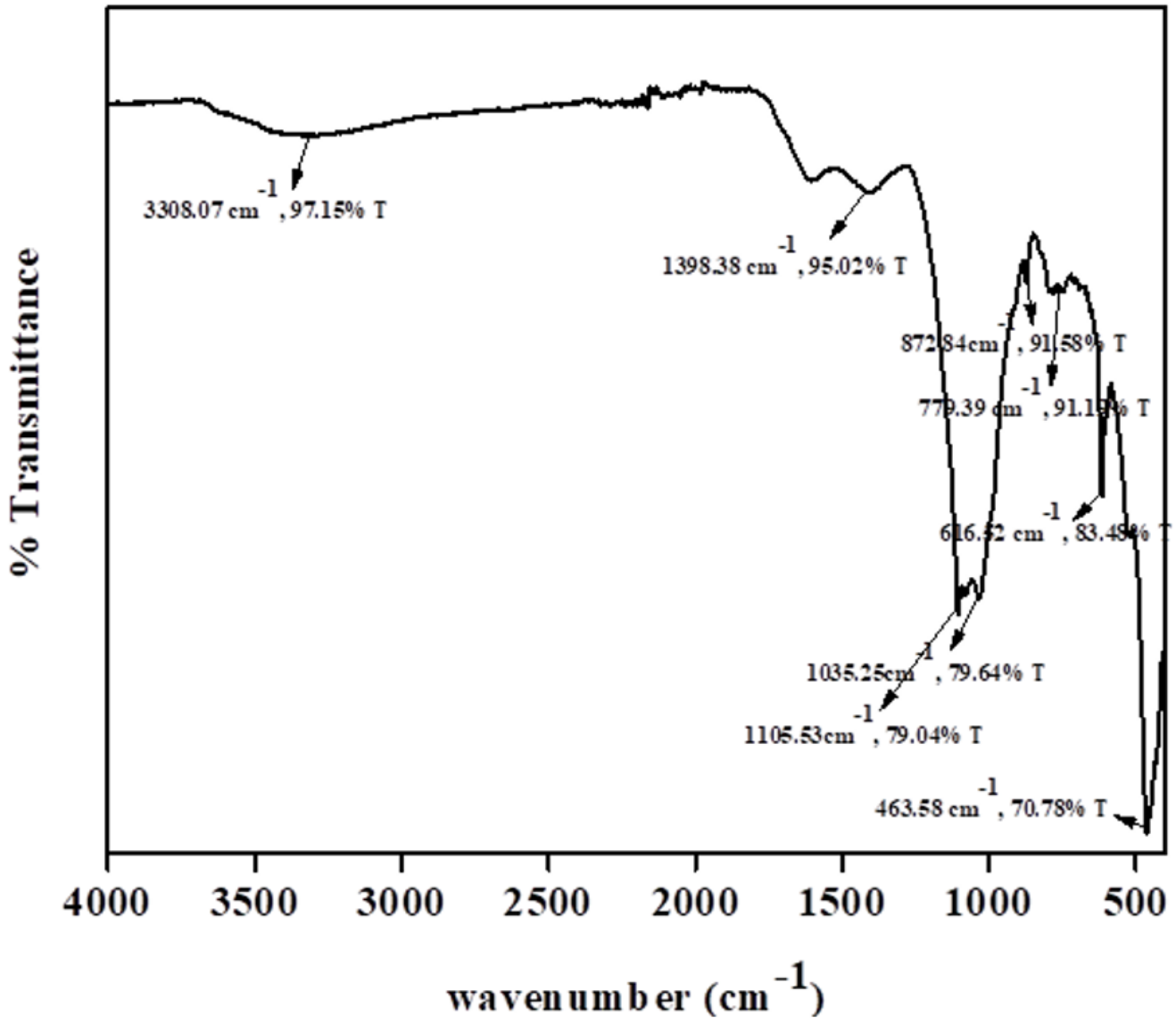

Figure 2

FTIR spectra of *Cladophora glomerata* biochar.

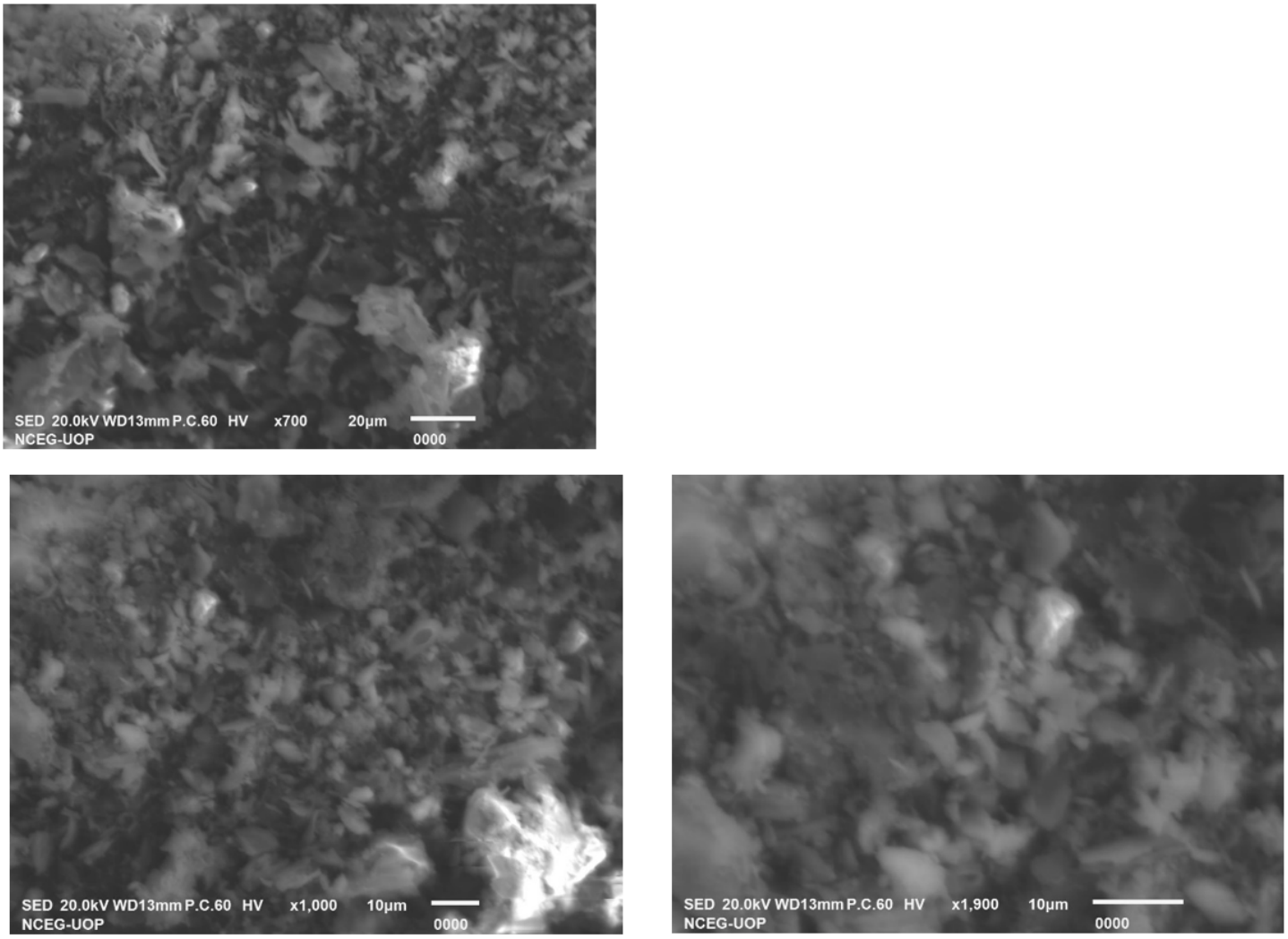

Figure 3

SEM images at different resolution for particle morphology of *C. glomerata*biochar.

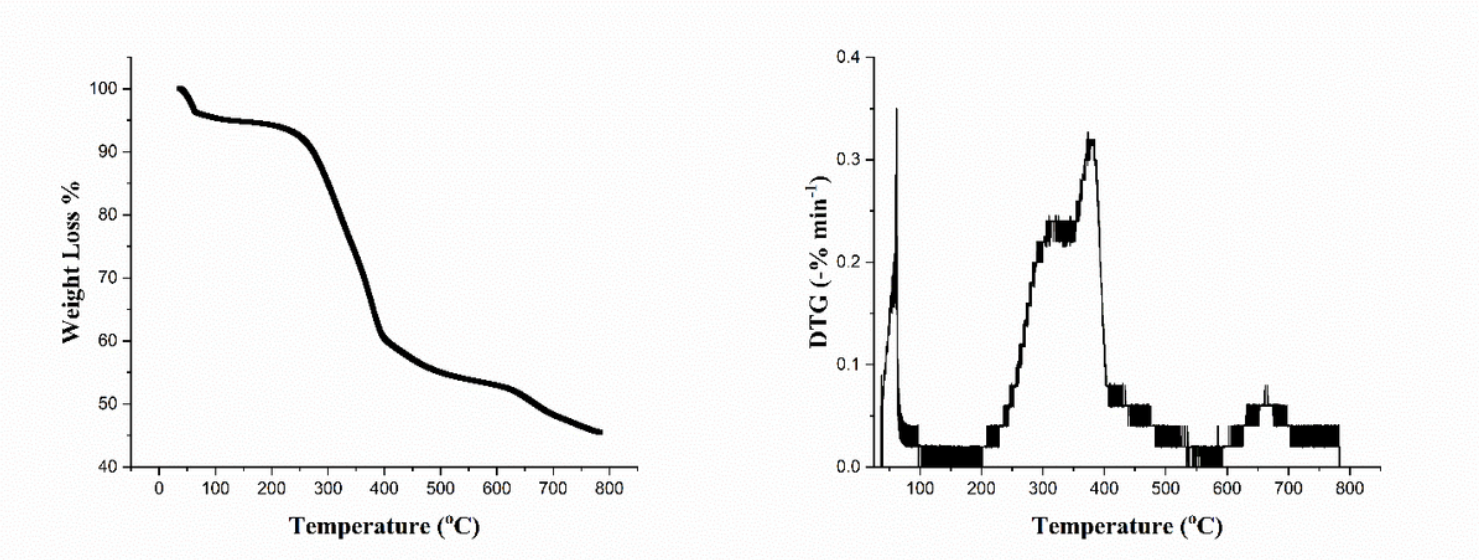

Figure 4

Thermogravimetric curve (Left) and first derivative of thermogravimetric curve (right) obtained at heating rate of 30 °C. min^{-1} from room temperature to above 750 °C temperature for the pyrolysis of *C. glomerata* biomass.

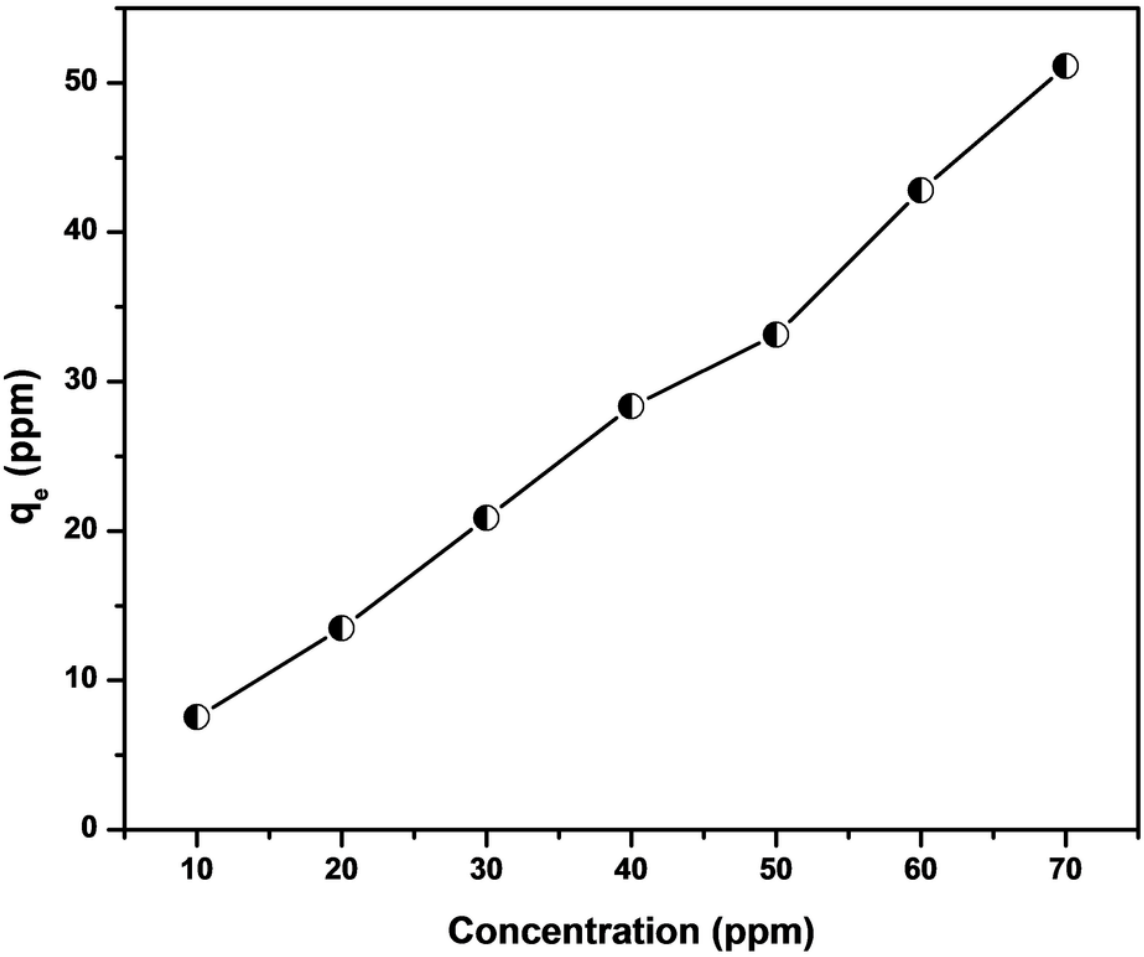

Figure 5

Effect of concentration on adsorption, plotting concentration vs qe.

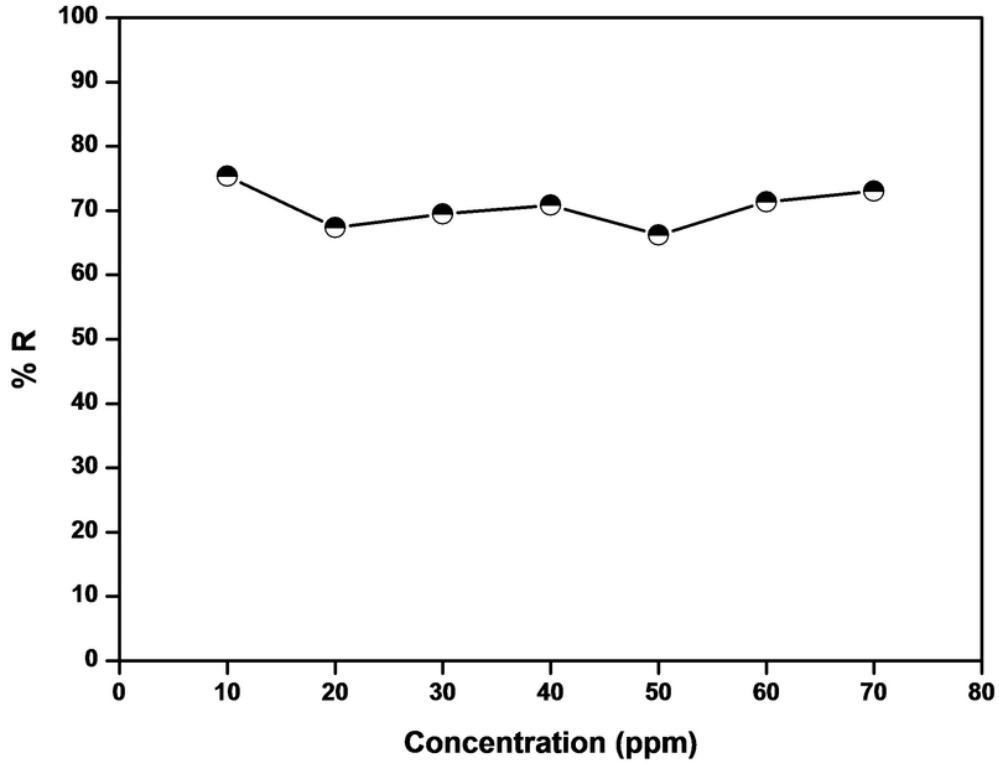

Figure 6

Effect of concentration on adsorption, plotting concentration vs %R.

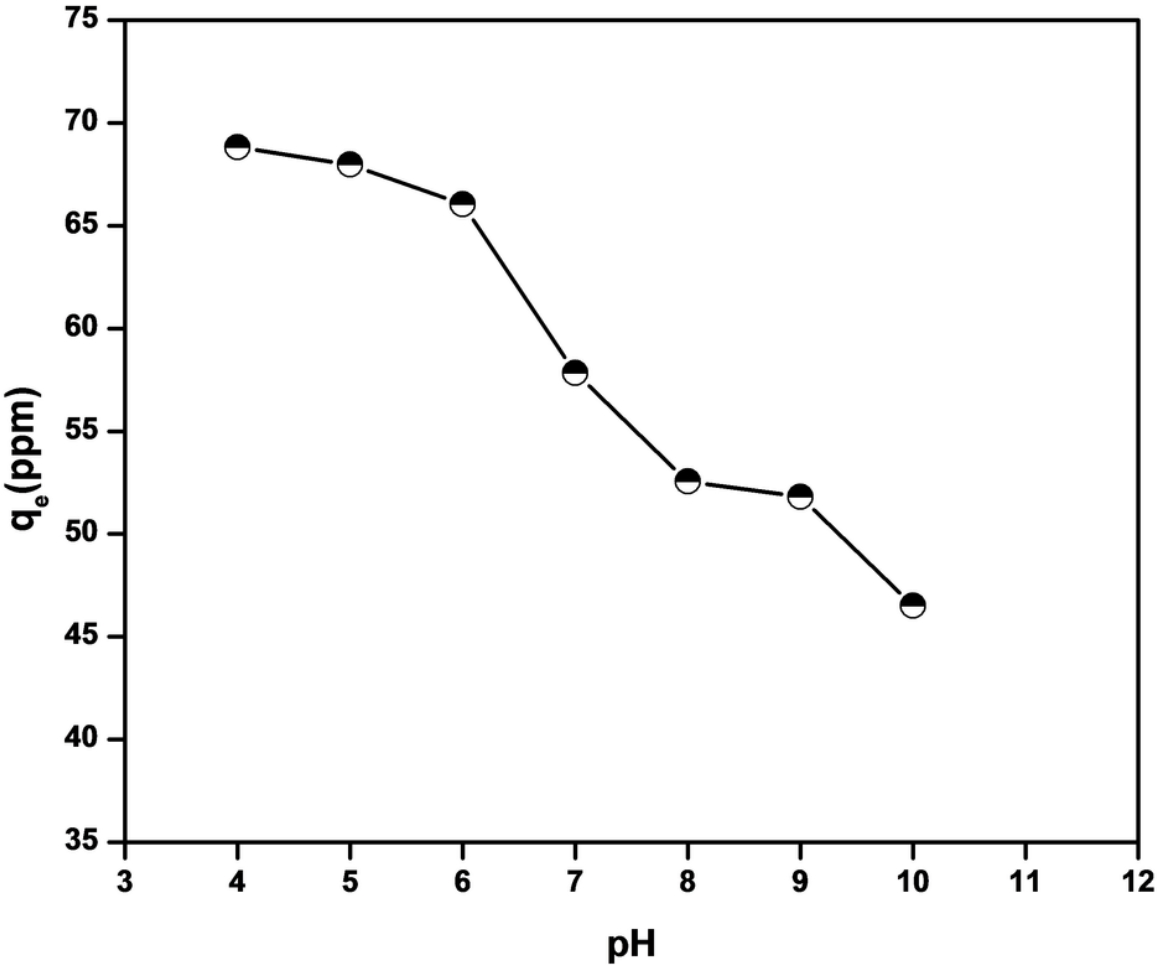

Figure 7

Effect of pH on adsorption, pH vs qe.

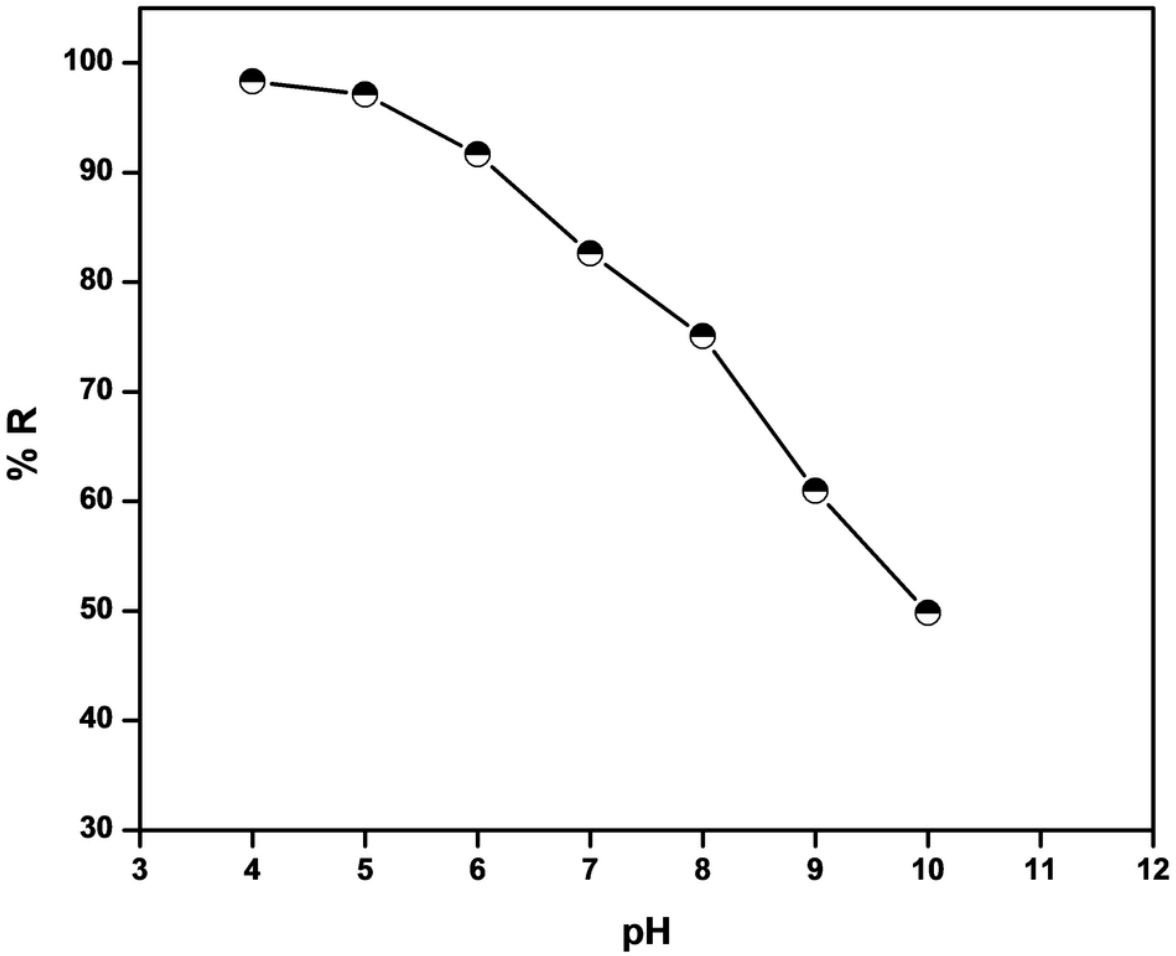

Figure 8

Effect of pH on adsorption, pH vs %R.

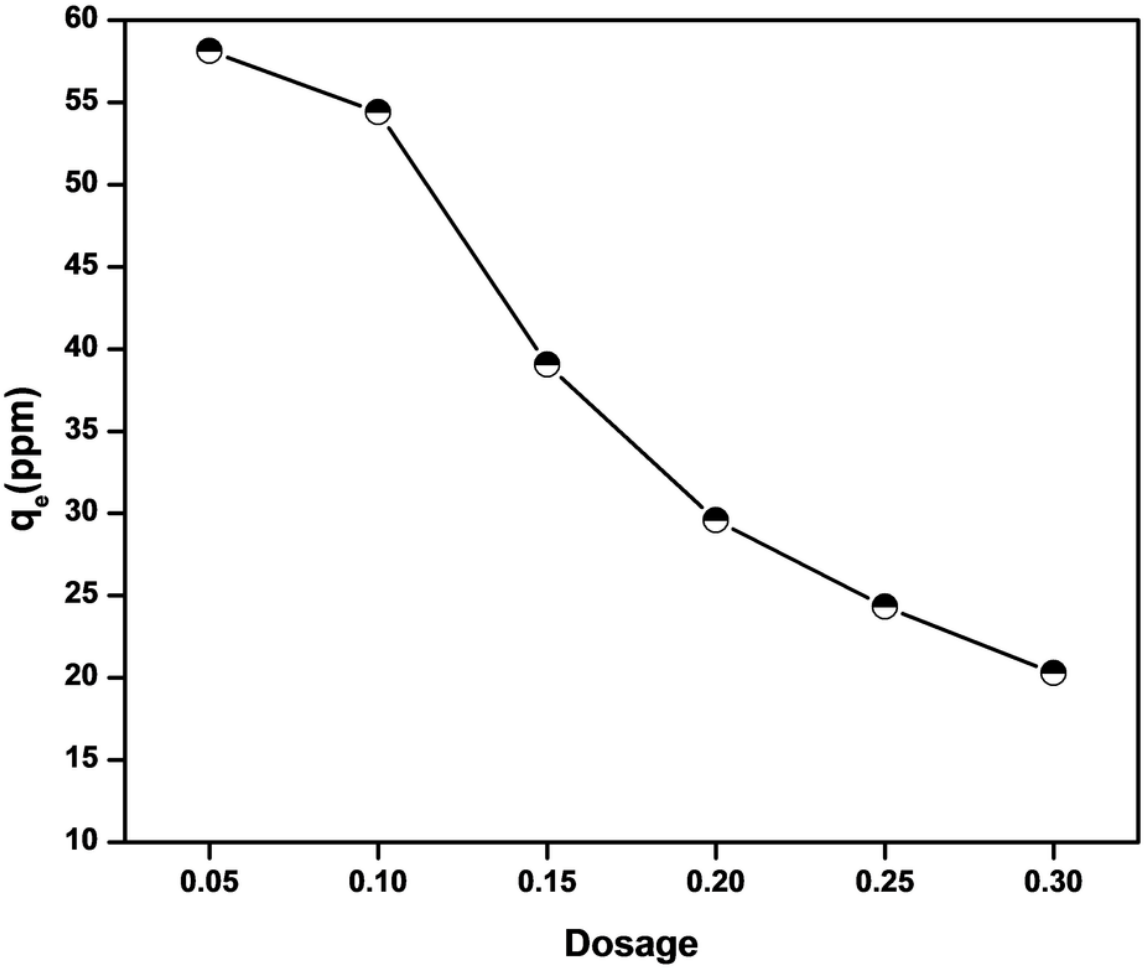

Figure 9

Effect of dosage on adsorption, dosage vs qe.

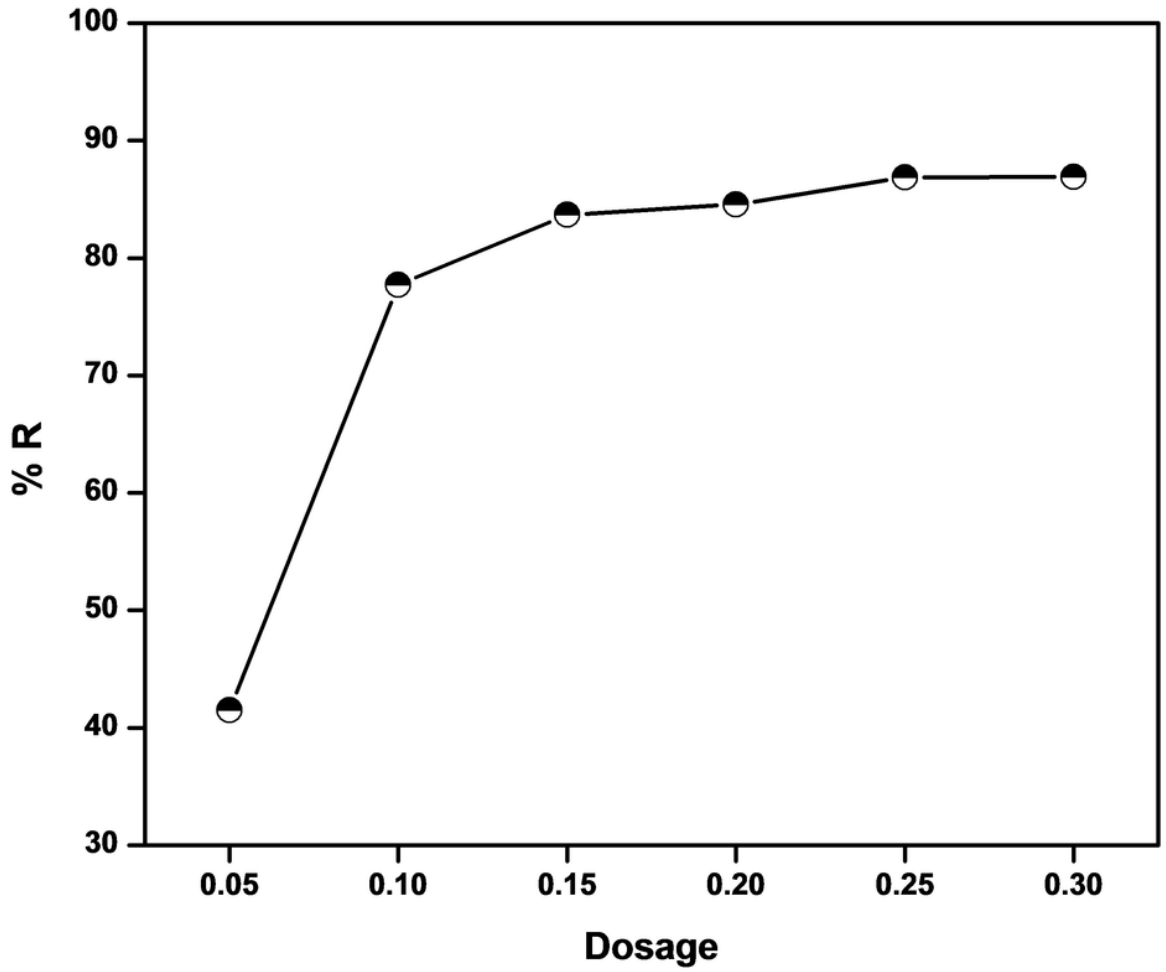

Figure 10

Effect of dosage on adsorption, dosage vs %R

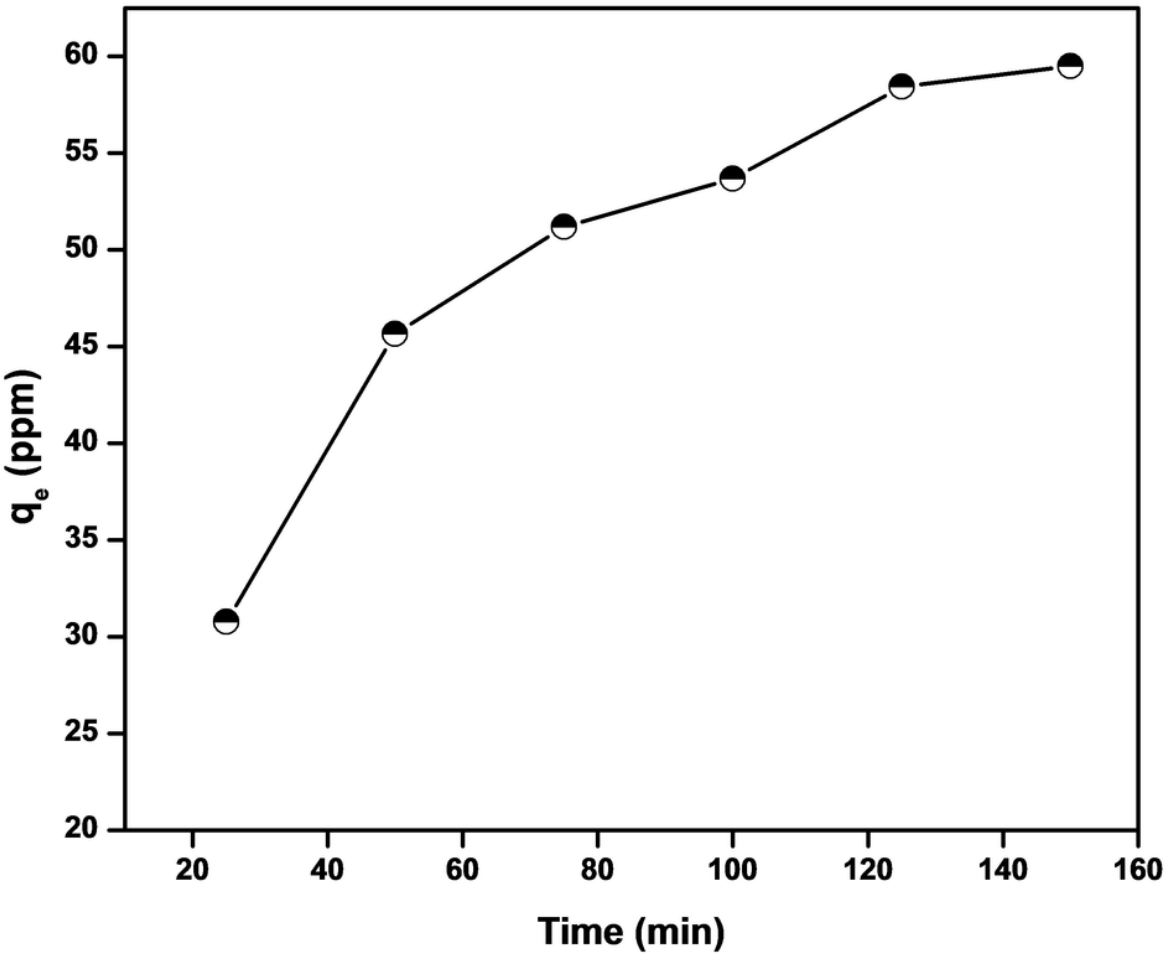

Figure 11

Effect of time on adsorption, time vs q_e.

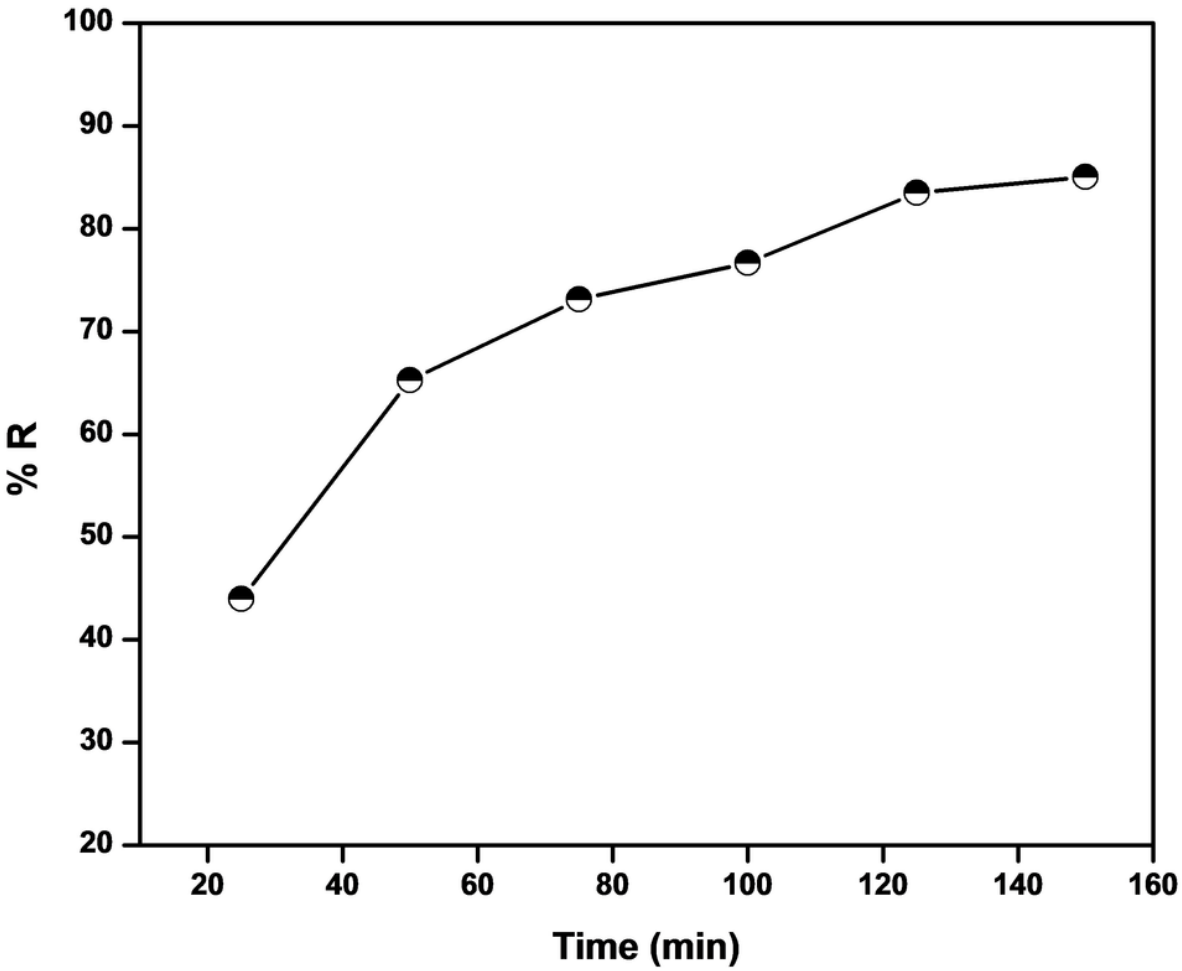

Figure 12

Effect of time on adsorption, time vs %R.

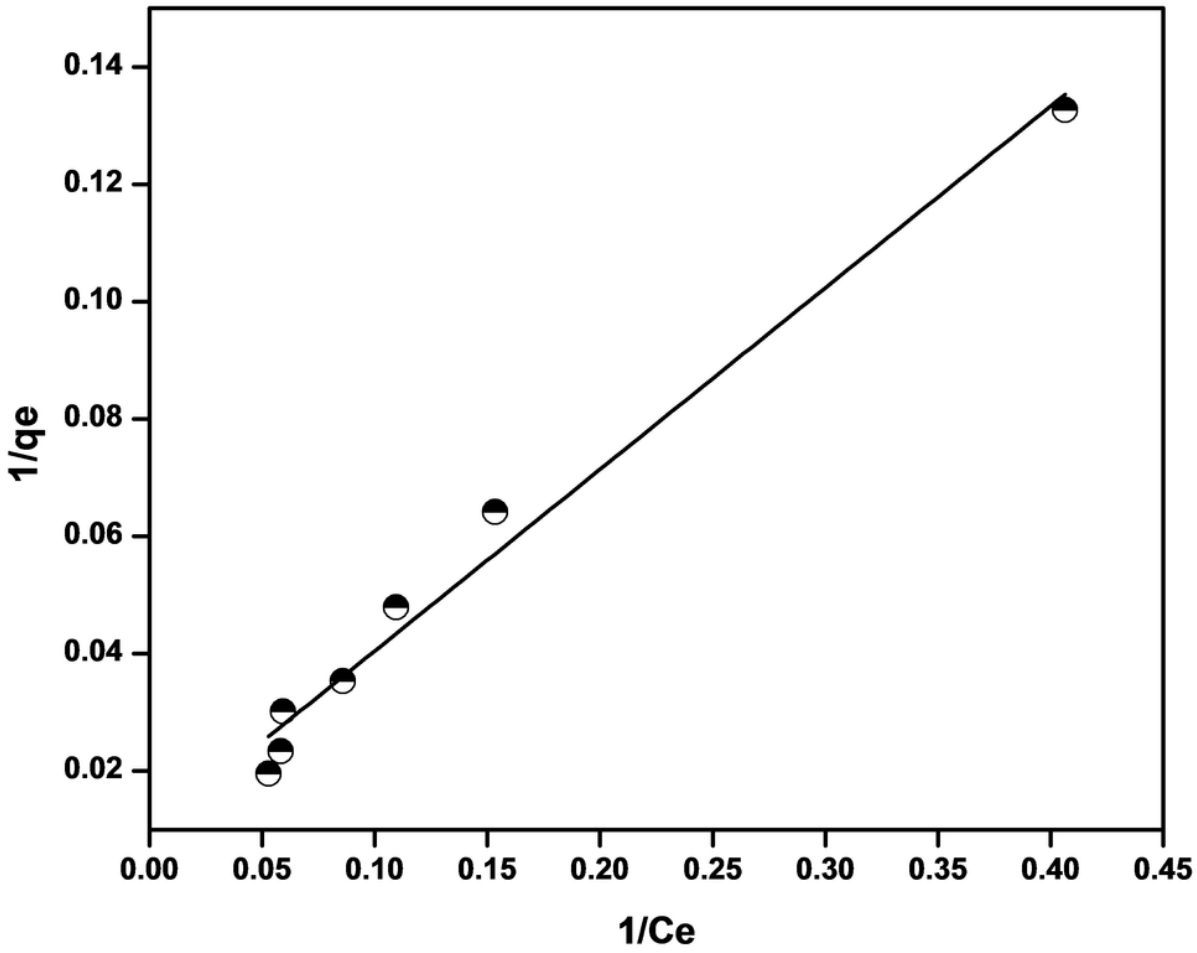

Figure 13

Langmuir isotherm for brilliant blue adsorption on *C glomerata* biochar.

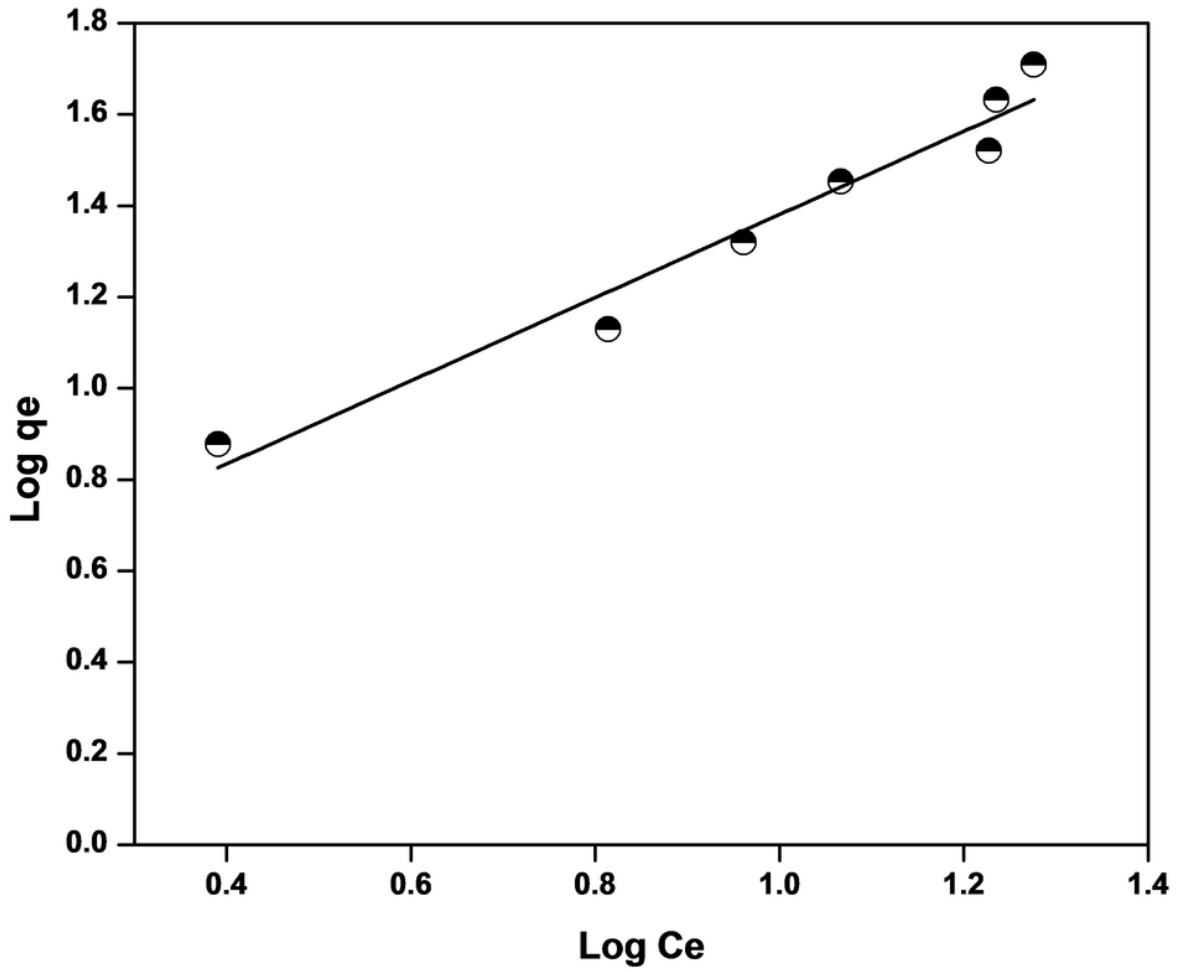

Figure 14

Freundlich isotherm for brilliant blue dye adsorption on *C. glomerata* biochar.