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RESEARCH ARTICLE

Adsorptive Removal of Malachite Green Dye from Aqueous Solution Using Chemically Modified Charred and Xanthated Wheat Bran

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Abstract

Adsorptive removal of Malachite Green (MB) dye from aqueous solution using chemically modified Wheat Bran has been investigated. Wheat bran was chemically modified to charred through charring process and it was further modified to Xanthated through xanthation process. Batch experiments were carried out to study the experimental parameters such as effect of pH, effect of concentration and effect of contact time for both Charred Wheat Bran (CWB) and Xanthated Wheat Bran (XWB) simultaneously. The concentrations of dye ions before and after the adsorption were determined by using UV-Visible Spectrophotometer. The dye uptake was maximum for the initial pH of 4 for both CWB and XWB but the percentage removal for XWB was found to be effective in comparison with CWB i.e., 98.45% and 93.45% respectively with adsorbent dose of 0.025g and agitation speed of 190 rpm. The applicability of Langmuir isotherm was tested. The adsorption capacity of MG dye into CWB and XWB was found to be 69 mg/g and 112.9 mg/g, respectively. Similarly, the kinetic data best fitted for pseudo-second order. Hence, the result showed that XWB may be an attractive alternative for the removal of MG dye from aqueous solution in comparison to CWB as bioadsorbent.

Keywords: Malachite green, xanthated wheat bran, adsorption-isotherm, charred wheat bran

Introduction

Industries like textile, paper, food technology, hair colorings, leather tanning etc. use synthetic dyes extensively. Wastewaters discharged from these industries are usually polluted by residual dyes [1]. Such residual dyes are non-biodegradable due to their complex molecular structures making them more stable and harder to biodegrade. They pollute water, inhibit penetration of sunlight into the water bodies and constitute a serious threat to the environment. Many of them are also toxic in nature and can cause direct destruction or can affect catalytic capabilities of various microorganisms [2].

Malachite green (MG) is available in various forms, namely oxalate or hydrochloride salt [4]. Malachite green a triphenylmethane cationic dye, is most commonly used for dyeing cotton silk, paper, leather and also in manufacturing of paint and printing inks. [5] In addition, MG also is used as a fungicide and antiseptic in aquaculture industry to control fish parasites and disease [6]. The MG is highly toxic to mammalian cells and causes detrimental effects in liver, kidney, intestine, gonads and pituitary gonadotropic cells. In addition, they may enter into the food chain and could possibly causes carcinogenic, mutagenic, teratogenic effects on humans. Therefore, the treatment of effluent containing MG pollutants being discharged to the environment is essential [7].

Several methods are used to remove these dyes from waste water, namely chemical oxidation and membrane separation. Other processes include aerobic/anaerobic processes, electrochemical method, agglomeration etc. Because of being expensive and inefficient, most of these processes are not used on large scale. Coagulations and chemical and electrochemical oxidations have low practicability on large scale plants. Adsorption is preferable over

these processes and is widely used because of low cost and high performance. Activated carbon is the most common adsorbent used. Economic benefits, performance efficiencies and environment are primary considerations while choosing an adsorbent, therefore researchers usually cheap and best adsorbents, which are usually waste materials such as activated carbon prepared from the agricultural wastes/by-products such as saw dust, maize cob, rice husk, tea leaves, potato peel, orange peel, coconuts heel, etc. [13]. Hence, herein we report wheat bran as a low cost, ecofriendly, rapid and cost-efficient bio-adsorbent for the removal of malachite green dye.

Wheat bran collected was converted into carbon by the chemical treatments adding conc. sulphuric acid. That charred bio-waste can be further activated by chemical modification. With the help of activation of the surface functional group, adsorption increases gradually.

Materials and Methods

Materials

Wheat bran were collected from Punarbash municipality-09, Kanchanpur Nepal. All chemical used were LA/AR grade.

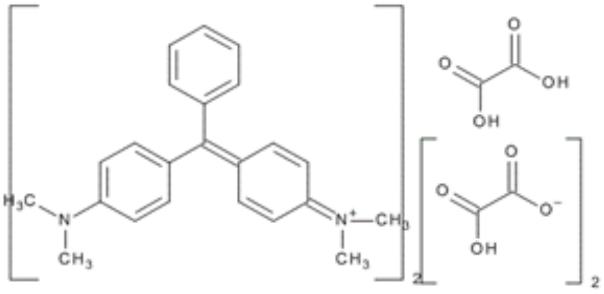

Figure 1: *Molecular structure of malachite green.*

Preparation of charred wheat bran

For the preparation of Charred wheat bran, 400 g of the powered wheat bran was taken in a 5L bucket and 100 mL of conc. sulphuric acid (H_2SO_4) was added in it and stirred thoroughly with wooden spatula until it turns completely black. Addition of conc. H_2SO_4added in a little amount at first followed by stirring and again added in small amount. This process was repeated several times till it turns completely black in color. This process is called charring. In this process ring opening of cellulose takes place. Thus, obtained black power of wheat bran was left over for 24 hrs. Charred sample was washed several times with deionized water until the pH of the water obtained after washing reached to neutral i.e. pH 7 then it was air dried for 4 days and 6 hrs. in hot air oven for complete removal of moisture from it. Thus, obtained charcoal was grinded thoroughly and sieved with 150 µm. Finally, 200 g charred sample obtained was kept in desiccator for 1hr and stored in air tight bottle.

Preparation of xanthated wheat bran

100 g of the charred wheat bran was taken in a reagent bottle, 100 mL of 1N NaOH was added in it and shaked in a mechanical shaker for 3 hrs. Then 20 mL of carbon disulphite was added in it and again shaked in mechanical shaker for 6 hrs and left overnight, after which the solution was filtered with Whatman filter paper. It was washed with several times with deionized water for making the sample neutral. Thus, obtained charcoal was air dried for 3 days and oven dried for 6 hrs. for complete removal of moisture. Finally, 70 g Xanthated wheat bran was kept in desiccator for 1hrand stored in air tight bottle.

Instrumentation

SEM images of samples were taken to investigate the surface morphology of the activated carbons using FE-SEM, HITACHI, SUB 8230, Tokyo, Japan.The FTIR spectrum of CWB and XWB before and after adsorption of MG dye were analyzed by SHIMADZU, IRPrestige-21, Pacific Commercial Company (P) Ltd.

A calibration curve for MG dye was constructed by using UV-Vis double beam spectrophotometer (Model LT-2802) at maximum wavelength of 617nm at pH 4. Similarly, the pH of the solution was monitored using Digital pH meter, Labtronics-11, Hyderabad, India.

Experimental procedure

Batch adsorption experiments were carried out in a mechanical shaker at room temperature. 25 mL of MG dye solution of known concentration (25-800 mg/L) was shaken at constant agitation speed (190 rpm) with constant adsorbent dose 25 mg for specific period of contact time in mechanical shaker. The pH of the solution was adjusted by contacting with HCl or NaOH solution. After equilibrium, the final concentrations (Ce) were measured and the amount of MG dye adsorbed in mg/g at equilibrium was computed by the equation.

$$q_e = \frac{C_i - C_e}{W} \times V \qquad (1)$$

where, and are the initial and equilibrium concentration of MG dye in mg/L, respectively. V is volume of MG dye solution in liter and W is the weight of adsorbent in gram.

And the percentage adsorption of dye was calculated using the following expression,

$$\%A = \frac{C_i - C_e}{W} \times 100 \qquad (2)$$

Where A% is the percentage of dye adsorption from the solution.

Results and Discussion

Characterization of adsorbents: FTIR analysis

The combined FTIR spectrum of RWB, CWB and XWB is shown in the following figure separately. The FTIR spectrum of adsorbents were taken between 300-4500 cm^{-1} using IR tracer. By the close inspection of FTIR spectrum the samples showed clear and broad centered at 3200-3600 cm^{-1} which is because of vibrations of OH groups of alcohols, phenols or carboxylic acids. The band is very intense in RAW and decreased on modified samples.

This means the moisture content after treatment drastically decreased hence, the amount of carbon remains in the sample. The broad peak observed at 3348.42 cm^{-1} are due o the existence of bounded hydroxyl groups. The peak observed at 2924.09 cm^{-1} can be assigned to C-H group. The peak around 1018.41 cm^{-1} (RWB), 1041.56 cm^{-1} (CWB) and 1026.13 cm^{-1} (XWB) are the characteristics of C-O group of primary hydroxyl group also the peaks at 1635 cm^{-1} (RWB), 1612.49 cm^{-1} (CWB) and 1558.48 cm^{-1} (XWB) shows N-H group present at all adsorbent. The sulfate is found between the range of 1380-1415 cm^{-1} and the result extracted is found the band 1365.60 cm^{-1} at CWB is depicted to 1381.03 cm^{-1} revealing that sulfate group.

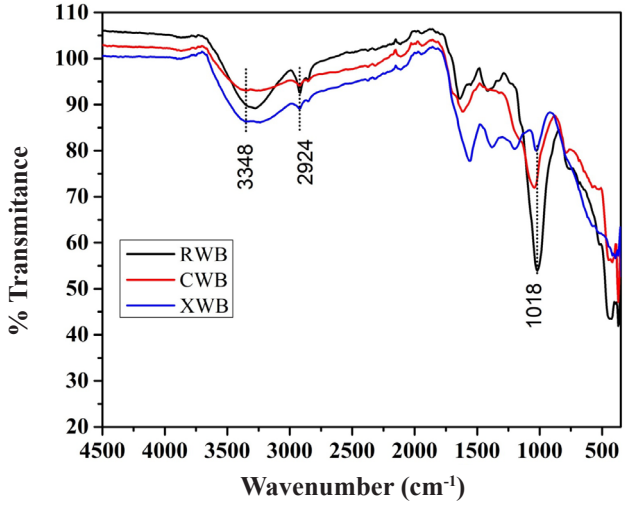

Figure 2: *FTIR spectra of RWB, XWB and CWB*

FE-SEM analysis

The SEM images of RWB, CWB and XWB are shown in Figure 3a, 3b and 3c respectively. The SEM image of RWB appears to be rough whereas those of CWB and XWB appear to be micro porous, showing high surface area. Surface structure of the XWB was found more enhanced morphology, rough and non-uniform then that of CWB because of the further modification from CWB. Its SEM structure looks like honeycomb with full of cavities having capacity to adsorb more adsorbate in its cavities. SEM images revealed that adsorption capacity of the CWB is less than that of XWB.

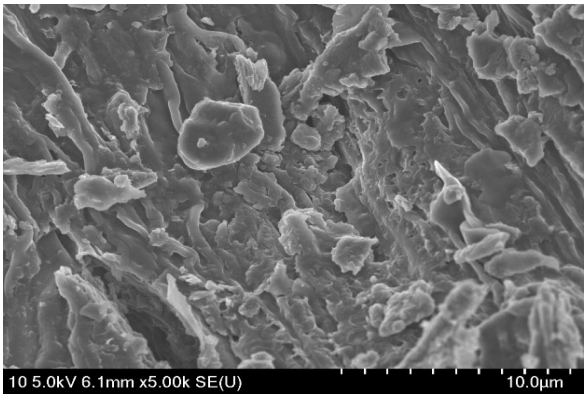

Figure 3a*: SEM image of RWB*

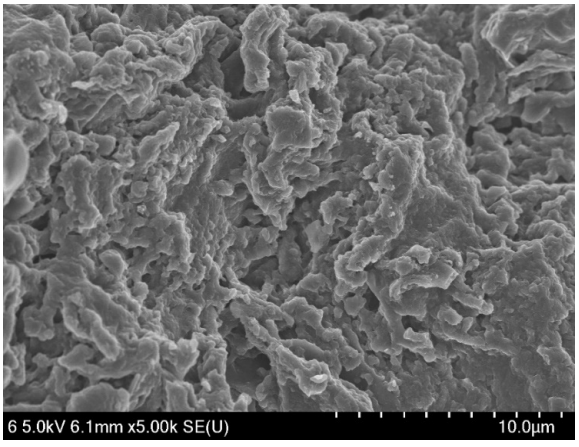

Figure 3b*: SEM image of CWB*

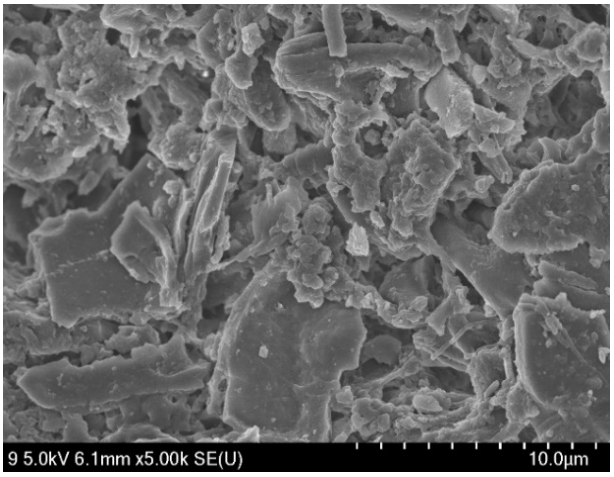

***Figure 3c**: SEM image of XWB*

Effect of pH on MG dye removal

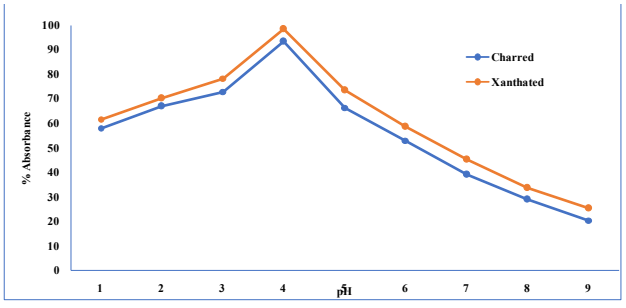

***Figure 4:** Effect of pH on CWB and XWB*

From the pH studies it is seen that the optimum pH for CWB and XWB was found to be 4.0. It can be seen from above figure that the maximum percentage removal of the MG dye was found to be 93.35% and 98.45% for CWB and XWB respectively. The dye removing ability of the XWB was found more efficient than that of the CWB because of the addition of the chelating group i.e. S-atom of sulphur group which enhance the ability of the activated carbon to adsorb more dye onto the XWB. Thus, it can be concluded that the optimum pH for adsorption of MG dye by CWB and XWB is 4.0.

Kinetic studies of adsorption of MG dye

Kinetics studies of the adsorption of MG dye onto CWB and XWB was represented as a function of time at initial dye concentration of 25 ppm. The concentration of the dye was analyzed as the function of time at optimum pH 4 by using UV-Visible spectrophotometer. In batch adsorption process, kinetic studies provide information about optimum conditions, mechanism of sorption, and possible rate controlling step. For this purpose, linear and nonlinear form of pseudo-first- and pseudo-second-order kinetics is applied on adsorption data.

The pseudo-first-order equation is given as:

$$log\ (q_e - q_t) = log\ q_e - \frac{K_f}{2.303}\ t \qquad (3)$$

The value of will be calculated from the linear plot of vs t for the adsorption of dye. Hence, the plot of against t gives the straight line from which and can be calculated from slope and intercept respectively.

The pseudo-second order model is represented as:

$$\frac{t}{q_t} = \frac{1}{k_s q_e^2} + \frac{1}{q_e}\ t \qquad (4)$$

The equilibrium adsorption capacity, is obtained from the slope and is obtained from the intercept of linear plot of t/q_t versus t.

Pseudo second order plot of t/qt vs time give the perfect straight line for the adsorption of MG dye onto CWB and XWB simultaneously indicating that the adsorption reaction can be followed from the pseudo order kinetic model. Correlation coefficient R^2 value is nearly unity foe both CWB and XWB which shows that the pseudo second order model can be applied for the adsorption of the MG dye onto CWB and XWB

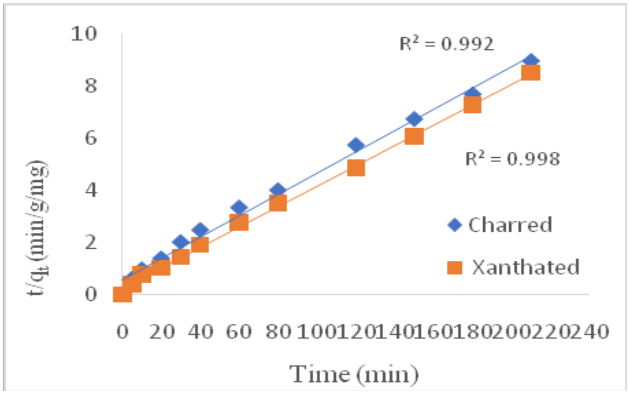

***Figure 5:** Pseudo-second order kinetic model for the adsorption of MG dye by using CWB and XWB*

***Table 1:** Pseudo-second order kinetics for adsorption of MG dye onto CWB and XWB*

Dye (MG)	Slope	R^2
MG onto CWB	0.0409	0.9922
MG onto XWB	0.0393	0.9985

also confirms the chemisorption of dye ions. All the above phenomenon is shown in the fig. below.

Adsorption isotherm studies

Adsorption isotherm experiment was conducted by taking various amounts of adsorbents in reagent glass bottles. Different amounts of adsorbents were added to each case of dye solution prepared in our laboratory. After an equilibration period of four hours absorbance of the supernatant clear liquid is determined spectrophotometrically at maximum wavelengh of dye. The amount of dye adsorbed was calculated using the graph. The applicability of the isotherms was judged from the values of the correlation coefficient for Freundlich and Langmuir isotherms. The Langmuir equation can be written in the following linear form:

$$\frac{C_e}{q_e} = \frac{1}{q_m K_L} + \frac{C_e}{q_m} \quad (5)$$

Where, is the amount of adsorbate per unit mass of adsorbent at equilibrium in (mg/g), is the equilibrium concentration of adsorbate in (mg/L), is the maximum adsorption capacity (mg/g) and is the Langmuir adsorption equilibrium constant (L/mg). The linear form of the Freundlich isotherm is as follows:

$$log\ q_e = log\ K_F + \frac{1}{n}\ log\ C_e \quad (6)$$

Where, is the amount adsorbed per unit mass of adsorbent (mg/g), is the equilibrium concentration of the adsorbate (mg/L). and are Freundlich equilibrium coefficients, which are considered to be the relative indicators of adsorption capacity and adsorption intensity.

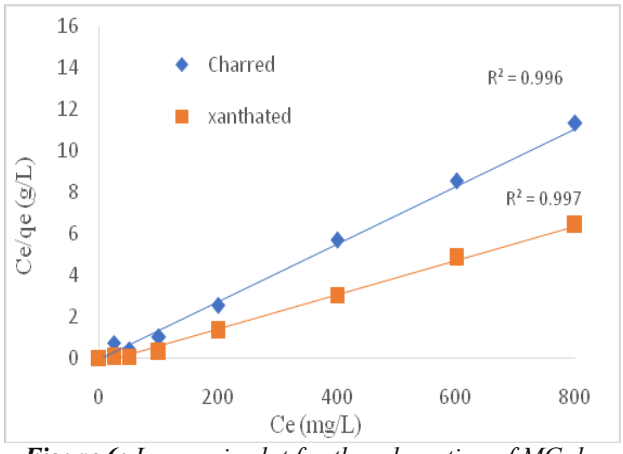

Figure 6: *Langmuir plot for the adsorption of MG dye onto CWB and XWB*

Langmuir and Freundlich adsorption isotherm were plotted from the data obtained from the effect of the initial concentration. Among them Langmuir adsorption isotherm best fitted the by both adsorbents because Langmuir plot have correlation coefficient R^2 nearly equal to 1 than that of the Freundlich plot.

Table 2: *Langmuir parameter for the adsorption of MG dye onto CWB and XWB*

Dye	q_e (mg/g)	R^2
MG onto CWB	69	0.9969
MG onto XWB	112.9	0.9971

Conclusion

The maximum adsorption of dye was at initial pH of 4 for both CWB and XWB which illustrate that the optimum pH for both CWB and XWB is pH 4. The percentage of adsorption was found to be 93.35% and 98.45% respectively with adsorbent dose of 0.025 g and agitation speed of 190 rpm. Different adsorption isotherms plot was plotted using the data calculated mathematically using the adsorption data obtained from the initial concentration of dye. The data best fitted for the Langmuir isotherm which has higher correlation coefficient for both CWB and XWB i.e. 0.9922 and 0.9985 respectively. Similarly, while studying the effect of contact time; it is concluded that CWB and XWB follows Pseudo-second order kinetics which is also verified by higher correlation coefficient values 0.9969 and 99.71 for CWB and XWB respectively.

Thus, it can be concluded that the CWB and XWB can be used for the adsorption of MG dye from the aqueous solution also XWB may be a better alternative for the removal of MG dye from aqueous solution in comparison to CWB as bioadsorbent.

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