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Research Article

MALACHITE GREEN DYE REMOVAL ON BIOADSORBENT NUTRACEUTICAL INDUSTRIALPTEROCARPUS MARSUPIUMSPENT

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ABSTRACT

The Agro-based nutraceutical industrial Pterocarpus Marsupiumspent (NIPMS) adsorbent used to adsorb a cationic dye, Malachite Green (MG) from aqueous solution. The influence of initial dye concentration, pH, temperature, adsorbent dosage and particle size on dye adsorption was studied. The experimental equilibrium data obtained were analyzed by isotherm models of Langmuir, Freundlich. The experimental value of the adsorption capacity, q_e was 45mg/g. The models applied for adsorption kinetic studies fitted well to a pseudo-second order model. The thermodynamic parameter values like ΔG^o, ΔH^o and ΔS^o proved that the process of adsorption was exothermic. The FTIR spectra and images of SEM proved the MG being adsorbed onto NIPMS. Possible interaction that occurred in the MG-NIPMS system is discussed. NIPMS is an effective adsorbent to remove MG dye from aqueous solution.

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INTRODUCTION

The large-scale production and extensive application of synthetic dyes is causing environmental pollution leading to a serious public concern. Large amounts of dyeing wastewater containing a sizeable amount of residual dye (Verma *et al.,* 2012) have led to the legislation on the limits of color discharge which is becoming increasingly rigid. The presence of small amount of dyes is highly visible and undesirable. Thus, the necessity for dye-containing water to undergo treatment before disposal into the environment is highly imperative (Fernando *et al.,* 2013; Wang and Xu 2012). The adsorption technique has been proven to be an effective and attractive process for the treatment of dye-containing waste water (Fuand Wang 2011).

India is botanical garden of the world and the largest producer of medicinal herbs (Seth and Sharma 2004; Modak *et al.,* 2007). A number of medicinal plantshave been traditionally used for over 1000 years in herbal preparations of Indian traditional health care systems commonly known as *Rasayana* (Modak *et al.,* 2007; Scartezzini and Speroni 2000). Many parts of tree including wood have been reported to be useful in treating diabetes (Evans 2002; Devgun *et al.,* 2009), and display excellent cardiotonic activity (Mohire *et al.,* 2007).

Pterocarpus Marsupium Roxb, is one of the important tree species with medicinal value. The bark is used for the treatment of stomachache, cholera, dysentery, urinary complaints, tong disease and toothache (Tiwari *et al.,* 2004). It has antipyretic and anthelmintic properties and used as a tonic to liver. It is useful in all diseases of body and styptic vulnerant, ophthalmia. The flowers are bitter; improve the appetite and cause flatulence. The timber is also highly valued in the international market for its quality (Tiwari *et al.,* 2004; Guha Bakshi *et al.,* 1999). The gum exudes 'kino' derived from this tree is used as an astringent (Singh1965).

Pterocarpus Marsupium belong to the family Fabaceae and contains chemical constituents like pterostillbene, (-)-epicatechin, pterosupin, marsupin. Many parts of the plant have been used as cardiotonic and hepatoprotective agents. The chemical composition of pterocarpus marsupium is pterocarpol, marsupol, maesupin, carpusin (Marsupsin), (-) epicatechin, propterol, pterosupin, marsupinol, Lupeol (https://easyayurveda.com). Most of the constituents are extracted from different parts of the plants by chemical and

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thermal processes. The spent generated after the processes have no fertilizer and feed value.

This study is to investigate adsorption of Malachite Green (MG), a toxic dye from aqueous solution using Nutraceutical Industrial Pterocarpus Marsupium Spent (NIPMS) as low cost biosorbent. The spent in the present context is the material left after extraction of the principle component(s). The process involves thermal, mechanical and chemical steps. In order to understand the nature of the adsorption; optimization of the parameters such as pH, initial dye concentration, adsorbent dose and temperature have been studied. Kinetics and equilibrium isotherms have been evaluated.

MATERIALS AND METHOD

Materials

Malachite green hydrochloride [cationic basic dye with Molecular formula $C_{23}H_{26}N_2Cl$, Molecular weight of 364.92, λ_{max}=618nm, classified as C.I. 42,000 was purchased from Sigma Aldrich Private Limited, Mumbai, India. All chemicals used were of analytical-grade reagents. The adsorption experiments were carried out at room temperature (25±28°C),30^0,40^0C and 50^0C.(Figure 1) shows the molecular structure of MG.

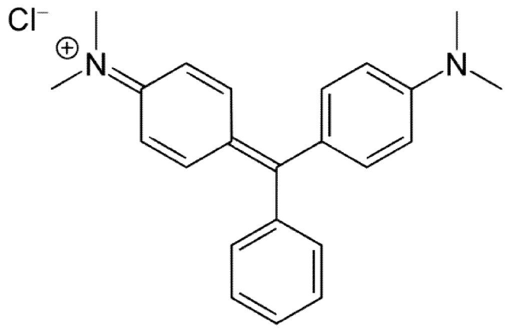

Figure 1 Molecular structure of Malachite green

Preparation of adsorbent

The NIPMS used in this study was procured from a local industry. It was dried in an oven at 60^0 C for 24 h. The dried spent material was ground to fine powder and sieved through ASTM 80 mesh to obtain particle size of ≤ 177 µm, and stored in plastic bottles for further use. No other chemical or physical treatments were done prior to adsorption process.

Surface characterization

The surface morphology of NIPMS was visualized by a Scanning Electronic Microscope (Carl Zeiss Scanning Electron Microscope EvoLS15, Germany). The functional groups present in the adsorbent were identified by FTIR. Infrared spectra of the NIPMS before adsorption and after the MG-loaded samples adsorption were obtained using an ATR-FTIR spectrometer (Perkin Elmer Spectrum two**)**. Point of zero charge (pH_z) was determined to ascertain the surface charge of NIPMS.

Adsorption experiment

Adsorption of MG from aqueous solution by NIPMS was investigated by batch method. The effects of various parameters affecting adsorption such as initial dye concentratio n, pH, temperature and adsorbent dosage were studied.

Batch adsorption experiments were carried out by adding a fixed amount 50 mg of adsorbent into 250 mL Erlenmeyer flasks containing 50 mL of initial dye concentration (25-200mg/L). The flasks were agitated (Kemi Orbital Shaker, India) at 150 rpm at 28±30°Cfor 180 min, until equilibrium was reached. Later, the sample was centrifuged for 10 minutes. The supernatant liquid containing un-adsorbed dye solution was removed using micropipette and the absorbance of the colored solution was measured by a double beam UV/Vis spectrophotometer (Systronics 166) at 618 nm. The amount of MG adsorbed at equilibrium, q_e(mg/g) was calculated using following equation (1).

$$q_e = (C_0 - C_e)\frac{V}{W} \quad \ldots\ldots(1)$$

Where, C_0 and C_e are concentrations (mg/L) of MG at initial and at equilibrium respectively, V is solution volume (L) and W is adsorbent weight (g). For kinetic studies, the same procedure was followed, but the aqueous samples monitored at pre-set time intervals. The concentrations of MG were similarly measured. The amount of MG adsorbed at any time, q_t(mg/g), was calculated using equation (2).

$$q_t = (C_0 - C_t)\frac{V}{W} \quad \ldots\ldots(2)$$

Where, C_t(mg/L) is the concentration of MG measured at time t. Initial concentrations of 25, 50 and 100 mg/L of the dye and adsorption time of 15 min (2 min intervals) performed in temperature controlled Orbital incubator. For determining optimum amount of adsorbent per unit mass of adsorbate, 50 mL of dye solution with NIPMS (50mg) till equilibrium was attained. To find out the influence of pH on dye adsorption, 50mg of NIPMS along with 50mL of dye solution of concentration 100mg/L were agitated using orbital shaker. The experiment was done with pH values of 2-12. The pH was adjusted with 0.1Ndilute HCl and 0.1NNaOH solution. Solution pH was determined by pH meter (Systronics 802, India). Agitation was continued for 180 min;in 140-150 min equilibrium was reached with constant agitation speed of 150 rpm. Agitation speed of 150rpm for 180min was fixed for all studies. At equilibrium, the dye concentration was measured using double beam UV/Vis spectrophotometer at 618nm. The extent of removal of dye was determined by following equation (3).

$$Dye\ removal\ efficiency\ \% = \frac{(C_0 - C_e)}{C_o} \times 100 \quad \ldots\ldots(3)$$

Modeling studies

Adsorption isotherms

The most accepted models for single solute system with two parameters are Langmuir and Freundlich (Ahmad and kumar 2010). The models were used to test the equilibrium adsorption at ambient temperature.

The non-linear forms of isotherm models studied are shown below.

$$langmuir\ isotherm\ :\ {}^{1}/_{x/m} = {}^{1}/_{\theta^\circ bc} + {}^{1}/_{\theta^\circ} \quad \ldots\ldots\ldots(4)$$

$$Freundlich\ isotherm : \log {}^{x}/_{m} = {}^{1}/_{n}\ \log C + \log kf \ldots\ldots\ldots (5)$$

Where, q_e is the amount of dye at equilibrium in unit mass of adsorbent (mg/g), C_e is

Concentration of dye solution at equilibrium (mg/L), $\theta^{\circ}$and b are the Langmuirco efficient related to adsorption capacity (mg/g) and adsorption energy (L/mg), respectively. K_f and n are the Freundlich coefficient related to adsorption capacity [(mg/g) / (mg/L) $^{1/n}$] and adsorption intensity of adsorbent, respectively. In our present study on NIPMS with MG both Langmuir and Freundlich model fits to the data. Langmuir best fits to the experimental results exhibits good correlation.

Adsorption kinetics

The controlling of the adsorption process was done by fitting experimental data with pseudo-second-order. The models were fitted according to linear least–square method. The controlling mechanism of the adsorption process was found by fitting the experimental data with the respective kinetic equations.

Thermodynamic parameters

Energy and entropy enable to understand the feasibility of mechanism. In the present study, thermodynamic parameters, including standard free energy (ΔG^0), enthalpy change (ΔH^0) and entropy change (ΔS^0) were estimated by using rate law and also kinetic data to find out the extent and enthalpy of the adsorption process.

RESULTS AND DISCUSSION

Surface characterization

Scanning electron microscope

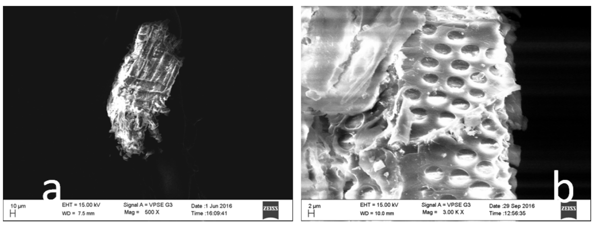

Figure 2a SEM image of NIPMS before adsorption**2b**NIPMS with MG after adsorption.

Point of zero charge

To determine pH_z, 0.1M KCl was prepared, and its initial pH was adjusted to 2.0 - 12.0 using 0.1NNaOH and HCl.

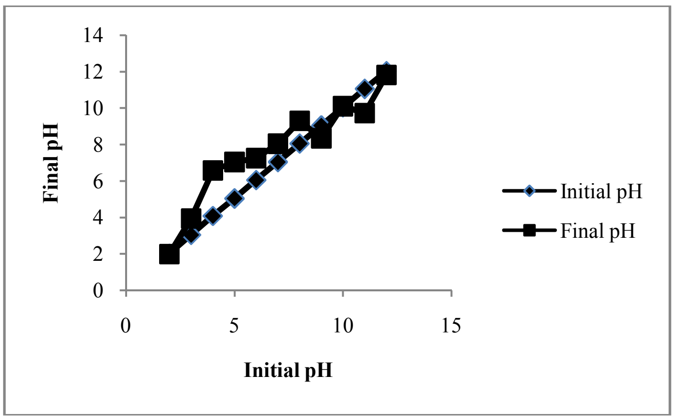

Figure 3 Point of zero charge of NIPMS

An amount of 50 mg of NIPMS was added to each 250 ml conical flask containing 50 ml of 0.1 M KCl with presetpH. After 24 h, the final solution pH was measured. Graphs were plotted between pH_{final} and $pH_{initial}$ (Ahmad and kumar 2010; Chaya and khatoon 2017)(Figure3).

FTIR characterization of MG adsorbed on to NIPMS

As seen in (Figure 4) The FTIR spectroscopy analyzes the interaction between NIPMS and MG. The FTIR Spectra of NIPMS recorded shows the vibrational frequency of the functional groups in the adsorbent3354cm^{-1} 0-H stretching vibrations presence of carboxylic acid,1594cm^{-1} presence of stretching vibrations C=C aromatic compounds carboxylic acid, alcohols, esters C-O stretching vibrations ranges 1222cm^{-1} to 1035 cm^{-1}, presence of 612 cm^{-1};624 cm^{-1}; 633 cm^{-1}; 674 cm^{-1} C-Cl stretching vibrations. The FTIR of MG-NIPMS shows functional groups after adsorption lies shows 2971cm^{-1} C-H stretching vibrations, 1739 cm^{-1} C=O stretching vibrations,1033 cm^{-1} 1217 cm^{-1},1229 cm^{-1} C-O stretching vibrations, presence of C-Cl stretching vibrations at 531-637 cm^{-1}, 900 cm^{-1}C=C bending vibrations.

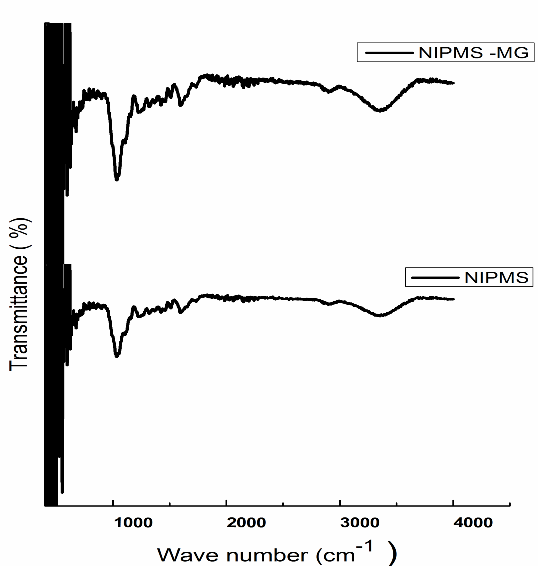

Figure 4 FTIR spectra of NIPMS before adsorption and NIPMS-MG after adsorption.

Adsorption Of MG

Effect of initial dye concentration

The dye uptake increased from17 to60mg/g of NIPMS with the increase in dye concentration from 25 to2 00mg/L. There is an increase in the initial dye concentration due to the increase on the driving force of the concentration gradient. Adsorption was rapid initially on the surfaces; Later, the dye molecules, probably entered into pores (interior surface), which is relatively a slow process. The adsorption of MG was more with higher concentration and remained almost constant after equilibrium time.

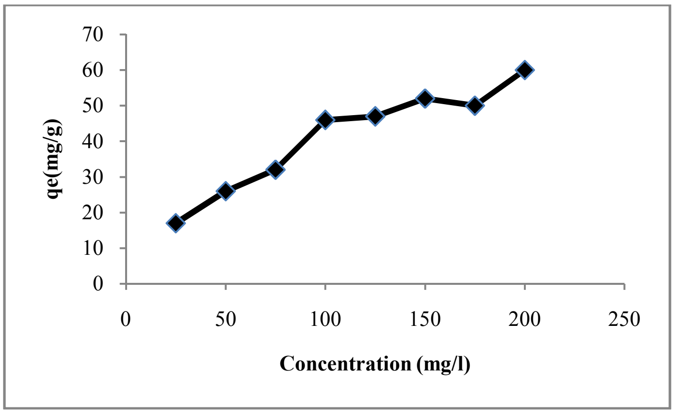

Figure 5 Effect of initial dye concentration on adsorption of MG

Influence of adsorbent dosage

The influence of adsorption of the dye onto NIPMS increased with the enhancement in the adsorbent dosage from 0.010 to 0.100 g. This may be due to the binding of almost all dye molecules on the adsorbent surface and establishing equilibrium of dye molecules and the adsorbent (Chowdhury and Das 2012)as shown in (Figure 6).

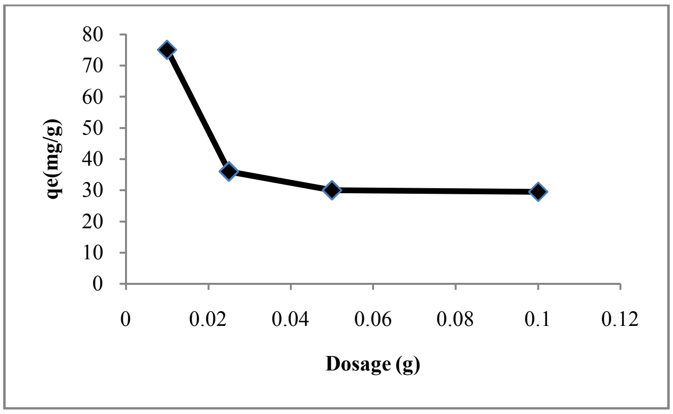

Figure 6 Effect of adsorbent dosage on adsorption of MG -NIPMS

Effect of temperature

The temperature was studied at different variants 30^{o}C, 40^{o}C and50^{o}C and the results are shown in (Figure7).

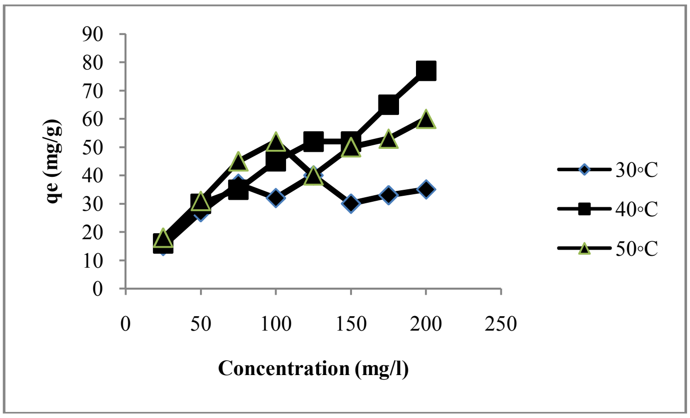

Figure 7 Effect of temperature on adsorption of MG – NIPMS

It can be observed that with the increase in temperature, the adsorption capacity increases marginally, which indicates that the process is exothermic in nature. The increase in adsorption with temperature is may be due to the increase in the mobility of the dye molecule with increase in their kinetic energy and the enhanced rate of intra-particle diffusion of adsorbate with the rise in temperature. The slight increase in removal of dye due to increasing temperature may be due to higher interaction between adsorbate and adsorbent.

Effect of pH

The pH is one of the most important parameters in the adsorption process. It controls the adsorption capacity by influencing adsorbent surface properties and ionic forms of dye. The adsorption capacity of NIPMS slightly increased with increase in solution pH and maximum adsorption capacity of MG was under acidic condition. In acidic pH an excess of H^+ ions compete with cations of the dye for adsorption sites. When the surface charge density gets lowered with increased solution pH, the repulsion between the positively charged dye and the surface of the adsorbent gets lowered. This results in increased adsorption as depicted in (Figure 8).

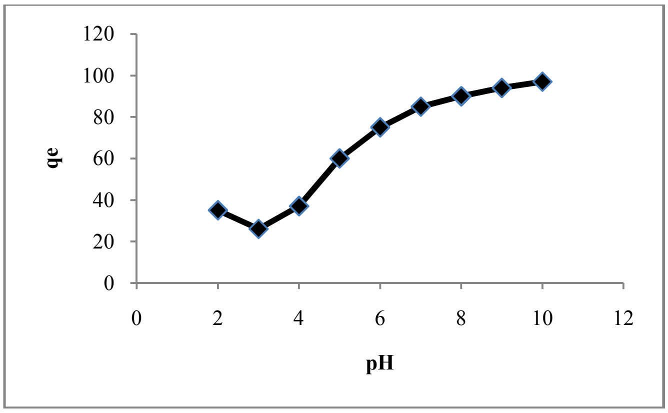

Figure 8 Effect of pH on adsorption at 100mg/Lconcentration of MG

Adsorption isotherms

The Langmuir adsorption isotherm exhibits monolayer phenomena of adsorption on MG-NIPMS system. Langmuir constants θ∘andbrelated asadsorption capacity and adsorption energy. Langmuir parameters are listed in (Table 1).The experimental data shows a straight line to the model MG-NIPMS adsorption indicates a good correlation coefficient (R^2) indicating best applicability of Langmuir adsorption isotherm.

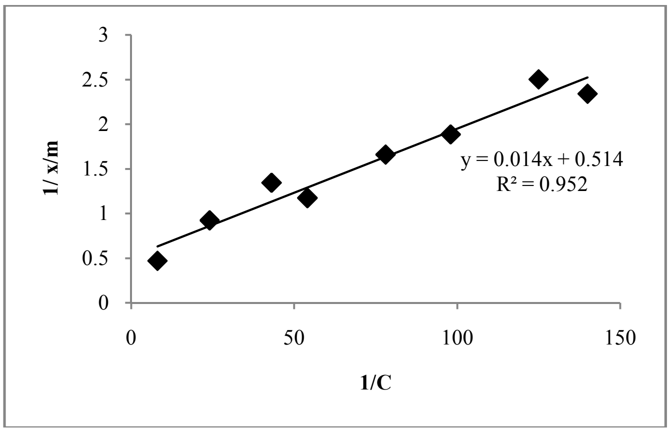

Figure 9 Langmuir adsorption isotherm for MG adsorption on NIPMS

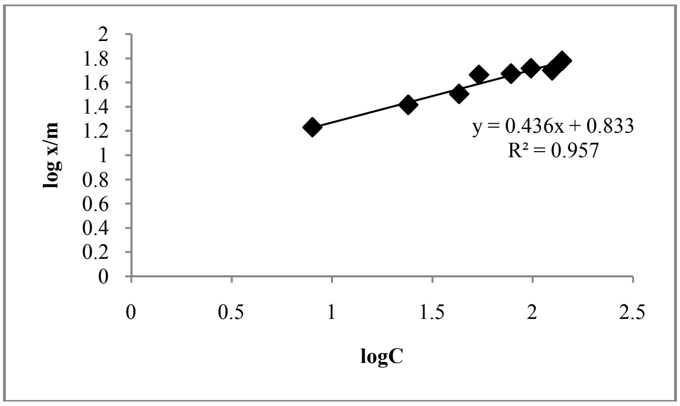

Figure 10 Freundlich adsorption isotherm for MG adsorption on NIPMS

Table 1 Langmuir & Freundlich isotherm constants for MG adsorption on to NIPMS

Temp (◦C)	Langmuir constants			Freundlich constants		
	θ◦(mg/g)	b(l/mg)	R^2	K_f(mg/g) (mg/l)$^{1/n}$	n	R^2
30	71.42	1.945	0.952	6.807	2.293	0.957

Adsorption kinetics

The rate constants for the adsorption of MG on NIPMS were obtained using the pseudo-second orderkinetic models.

Pseudo-second order kinetic model

The pseudo-second order kinetic model (Ahmad and kumar 2010; Hoand McKay1999)is presented in equation (8)

$$t/q_t = 1/k_2 q_e^2 + t/q_e \qquad (8)$$

Where, q_t and q_e are the amount of dye adsorbed at time t (mg/g) and at equilibrium (mg/g), respectively and k_2 is the pseudo-second order rate constant (g/mg min).The values of k_2 and q_e were calculated from intercepts and slopes of the linear plots of t/q_t vs. t respectively and presented in (Table 2). The experiments performed at 25mg/L, 50mg/L and 100mg/L with different temperature variants. The 100mg/L exhibits good correlation as seen in (Table 2) shows that the calculated q_e values are very close to that of experimentally obtained q_e and the values of correlation coefficients (R^2)are closer to unity confirms that adsorption of MG onto NIPMS follows pseudo-second order kinetics.

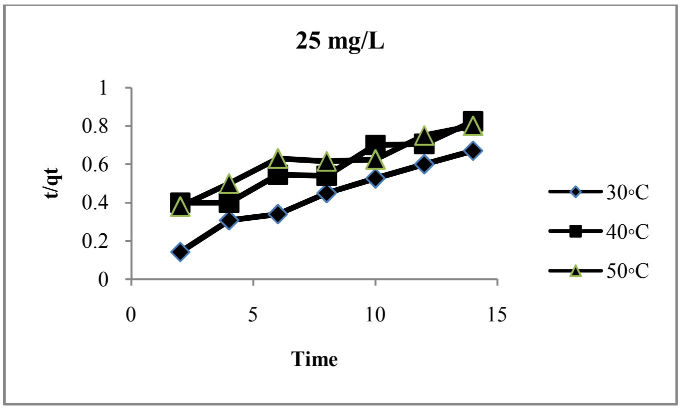

Figure 11c Pseudo-second order kinetic model of NIPMS on MG at 25mg/L

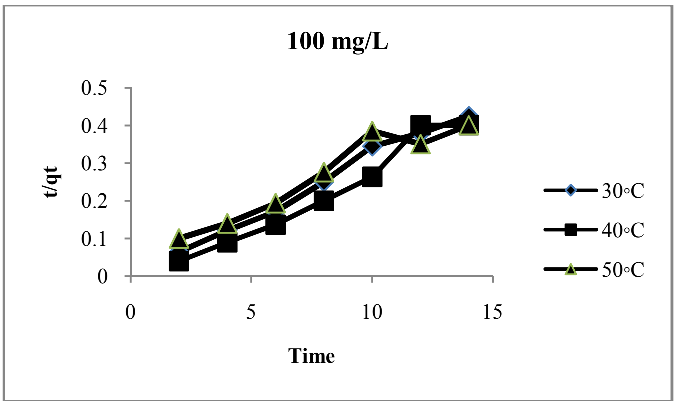

Figure11d Pseudo-second order kinetic model of NIPMS on MG at 100mg/L.

Table 2 Pseudo second order kinetic constants for MG adsorption on to NITTS

concentration (mg/L)	Temp (◦C)	qe (exp) (mg/g)	k_2 (g/mg min)	qe (cal) (mg/g)	R^2
	30		10.3	23.80	0.980
25	40	17.0	3.37	27.77	0.945
	50		2.75	32.25	0.907
	30		1000	32.25	0.986
100	40	45.0	23.80	31.25	0.966
	50		21.27	37.03	0.928

Effect of adsorption thermodynamics

The Activation energy (Ea) for the reaction using Arrhenius equations by the method of least squares(Low *et al.*, 1973;Feller and Gerday1997;Chaya and khatoon2017) a relationship between the rate constant k and temperature T,

$$k = A.e^{-Ea/RT} \qquad \ldots\ldots\ldots\ldots\ldots\ldots (9)$$

It follows in form

$$\text{as:} \log k = -\frac{Ea}{2.303RT} + \log A \qquad \ldots\ldots\ldots\ldots (10)$$

$$K_c = \frac{C_{ac}}{C_e} \qquad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (11)$$

$$\Delta G° = -RT \ln K_c \qquad \ldots\ldots\ldots\ldots\ldots\ldots (12)$$

$$\ln K_c = \Delta S°/R - \Delta H°/RT \qquad \ldots\ldots\ldots\ldots (13)$$

Where, K_c is the thermodynamic equilibrium constant, C_{ac} and C_e are the initial and equilibrium concentration(mg/L) of dye in solution, ΔH°, ΔG° and ΔS° can be determined from the slope and the intercept of the linear plot of $\ln K_c$ vs. 1/T(Ahmad and kumar 2010).

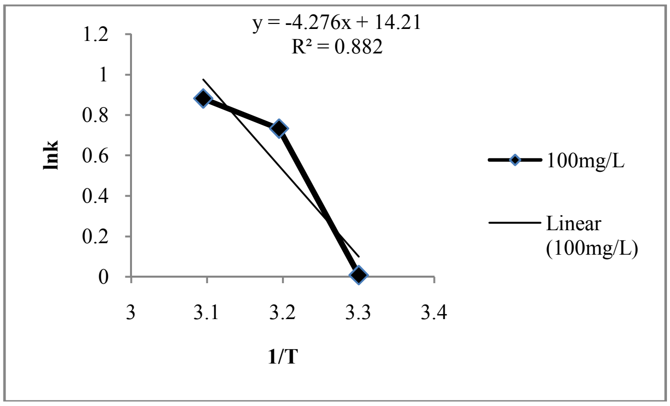

Figure 12a plot of Van't Hoff equation for adsorption Malachite green on NIPMS

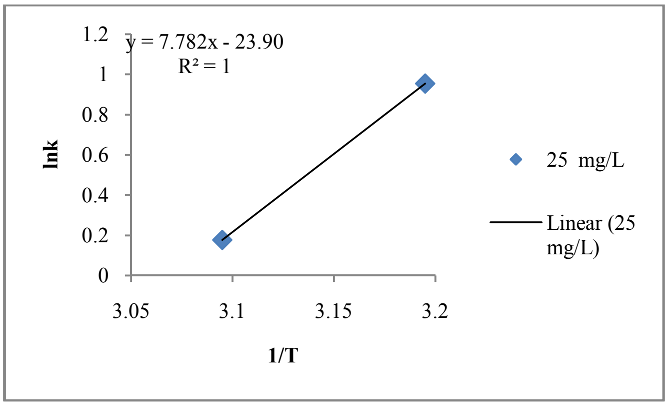

Figure12b plot of Van't Hoff equation for adsorption Malachite green on NIPMS

Table 3 Thermodynamic parameters for MG adsorption onto NIPMS Spent

concentration (mg/L)	Temperature(K)	ΔH° (KJ /mol)	ΔS °(KJ/mol K)	ΔG° (KJ/mol)
25	303 K	-248.69	-213.10	-269.11
	313 K	-257.02	-248.06	-326.10
	323 K	-265.34	-213.81	-290.03
100	303 K	-128.67	-205.88	260.46
	313 K	-136.98	-242.36	316.81
	323 K	-145.26	-244.93	330.36

The Gibbs free energy, entropy and enthalpy changes of adsorption were calculated by Van't Hoff and Gibbs-Helmholtz equations. As seen in (Table-3) the negative ΔH° value at 25mg/Lsuggests Spontaneous at low temperatures and the exothermicnature of adsorption. TheΔG° is positive for some studied temperatures indicating that the adsorption of MG ontoNIPMSfollows a Non-spontaneous at higher temperatures; 25mg/L ΔG° is negative indicatesSpontaneous at low temperatures. The Negative value of ΔS° suggests good affinity of MG towards the adsorbent and increased randomness at the solid solution surface.

CONCLUSION

The major conclusions of this study are:

- NIPMS has been developed as efficient, environmental-friendly and cost-effective biosorbent for the remediation of dye Malachite green.
- Operational parameters such as; initial dye concentration, adsorbent dose, temperature and pH, influenced the adsorption efficiency of NIPMS.
- Thermodynamic study demonstrated the Non-spontaneous and exothermic nature of biosorption process. It also confirmed that the adsorption is physical in nature.
- The concept demonstrated will help overcoming resource depletion through utilization of agro-based spent which has neither feed or fertilizer value.
- NIPMS is appropriate ready-to-use matrix in field of adsorption science.

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