

Use of *Cordia myxa* seed biomass as biosorbent for methylene blue dye removal from wastewater

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Abstract

Management of agriculture and horticulture waste biomass are of great concern in achieving circular economy and sustainable development goals. Use of plant biomass in sorption and biosorption is being explored for wastewater remediation cum agricultural and horticultural solid waste management. This study presents the use of the seeds of lasura/lasoda (*Cordia myxa*) as biosorbent for azo dye Methylene Blue (MB) from wastewater. The proximate and ultimate characterization of *Cordia myxa* seed powder (CMSP) was carried out for the bulk and surface characterization. Batch biosorption experiments were performed changing the range of parameters *i.e.* initial concentration of MB (C_0), and contact time (t) at room temperature. In order to know the nature and behaviour of biosorption process, equilibrium isotherms and kinetics were deciphered by employing commonly used models *i.e.* Langmuir, Freundlich, PFO (pseudo first order) and PSO (pseudo second order). The maximum MB dye uptake capacity of CMSP was determined as 21.93 mg/g at room temperature (398K). The Freundlich isotherm model and pseudo second order kinetic models were found to be the best fit ($R^2 > 0.99$) for the biosorption process. This study shows the applicability of *Cordia myxa* seed waste as low-cost treatment of dye wastewater.

Key words: Biowaste, *Cordia myxa*, biosorption, dye removal, adsorption, Langmuir model, Freundlich model

Introduction

Increase in the human population and energy consumption surges huge demand to increase the agroindustrial and horticulture production globally (Doering and Sorensen, 2018). More than three folds increase in the agricultural production has been observed in the last five decades (Duque-Acevedo *et al.*, 2020). Most of the agricultural and horticultural production are linked to the generation of huge amount of solid biomass residue / solid biowaste *i.e.* rice straw, husk, wheat straw, biomass from ornamental plants after pruning, fruit waste, non-edible seeds etc. (Obi *et al.*, 2016). This biomass can be used as feed stock, fuel for heating and in biofuel production. But efficient use of these biowaste is very less compared to its amount generated due to its less economic value linked to high investment in the collecting, processing, and transporting to the end user. In most of the cases waste biomass is burnt in the open fields, left as such or dumped, which creates nuisance as well as loss of available energy resource.

Use of biomass in the environmental pollution control is well evident. (Bharathi and Ramesh, 2013; Hubbe *et al.*, 2011; Mohamad Nor *et al.*, 2013). Sizeable work on the use of biomass for the wastewater treatment by sorption and biosorption process has been accomplished by researchers (Anastopoulos *et al.*, 2019; Gadd, 2009; Man mohan *et al.*, 2018; Wong *et al.*, 2018). Adsorption considered to be the physicochemical process of pollutant removal from liquid or gas medium using high surface area and porous materials *i.e.* activated carbon, silica etc (Kannan and Sundaram, 2001). These traditional adsorbents are characterised with high removal efficiency but they have negative impact on environment and economy. In this regard biosorption

has been considered as an effective method which uses living or dead biomass for the removal of pollutants from waste water. It may be metabolism dependent or non-metabolism dependent such as microbial pollutant removal and dead plant biomass-based pollutant removal, respectively. The process of biosorption of pollutant may involve the precipitation, complexation, ion-exchange, adsorption and intracellular transport (in case of metabolism dependent biosorption) mechanisms on the surface of adsorbent materials. There is a constant research to explore the non-traditional adsorbents such as raw and modified biomass for the decontamination of polluted water due to their less cost, abundance and eco-friendly nature.

Presence of dyes in industrial effluent such as textile finishing, paper pulp, cosmetics, leather industries, dye manufacturing etc., imposes toxicity and carcinogenicity on the local fauna and flora (Vadivelan and Vasanth Kumar, 2005). Color removal from these effluents has been considered crucial in terms of economy and ecological aspects due to the recalcitrant nature of synthetic dyes. It is reported that there are more than 10^5 commercial dyes available with production of over 7×10^5 MT annually. Azo dyes are very common and it is produced more than 50 % of overall synthetic dyestuff production globally. Textile processing units release approximately 2 and 10-15 % of total dyestuff, respectively in the wastewater (Koumanova and Allen, 2005). Sorption process have been favored amongst other complex physical, chemical and biological processes due to the simplicity of operation, cost effective and comparable efficiencies (Dahri *et al.*, 2015). Efforts have been made to use agro-waste materials as a substitute for commercial adsorbents such as charcoal, active carbon etc. For the removal of azo dyes

many agro-waste materials have been exploited *e.g.*, garlic peel (Hameed *et al.*, 2009), grated copra (Simarani *et al.*, 2015), rice straw (Man mohan *et al.*, 2018), rice husk (Ahmad *et al.*, 2020), walnut shell powder (Uddin and Nasar, 2020), chestnut and hazelnut shell (Sarici Özdemir, 2018) waste, corn, agave, alfa-alfa waste (Rosas-Castor *et al.*, 2014), banana peel (Amel *et al.*, 2012), ground nut shell etc.

Cordia myxa is also known as asyrian plume, naruvelli, or lasura, which is the member of Boraginaceae family. *C. myxa* is globally distributed across the arid/semiarid regions including Asia, Africa, Europe, Australia and America (Sivalingam *et al.*, 2012). Significant abundance has been reported in the Indian states of Rajasthan, Gujarat, Madhya Pradesh, Punjab, Haryana, and Uttar Pradesh (Saini *et al.*, 2002). The fruit of the *C. myxa* contains huge amount of mucus and a very hard seed. The fruit is edible and used mostly in pickle making in India. Other than pickle and vegetable making, the fruits are reported to be used as nutrient supplement for protein, carbohydrates, minerals, dietary fibers etc., and pulp from the ripen fruit can be used as glue. Fruits have been found to be rich in antioxidants, poly phenols and chlorogenic acids compounds which can provide health benefits in the skin and stomach related ailments (Meghwal *et al.*, 2014). The seeds of *C. myxa* are generally discarded as waste after extraction of mucus or pickle making process. In this research *C. myxa* seeds waste has been explored for low-cost removal of MB dye from waste water. Biosorption was studied in batch experimental mode by changing C_0 of MB solutions and contact time to evaluate the impact of parameters on the process. Equilibrium isotherms models *i.e.*, Langmuir and Freundlich isotherms were used to understand the nature of biosorption process. Sorption kinetics were studied employing pseudo first order and pseudo second order kinetic models.

Materials and methods

Biosorbent preparation and characterization: The *Cordia myxa* fruits were purchased from a vegetable market Raniwara, Jalore (Rajasthan, India). The seeds were removed from fruits and cleaned using hot water and dilute acid treatment (0.01N HCl) for 24 hours to remove mucous layer from the surface. Seeds (Fig. 1a) were oven dried overnight at 80°C. Dried seeds were pulverized in flour mill and the powder was sieved to get ≤ 0.5 mm particle size. To avoid contamination, the biosorbent CMSP was kept in air tight jar.

The CMSP surface morphology was studied by scanning electron microscopy (SEM) taking carbon coated material from carbon sputtering technique. The major chemical constituents and diversity of surface functional groups on CMSP was determined using CHNS analyser (model- elemental vario El cube) and FTIR spectroscopy (model- Thermo Nicolet) respectively. The FTIR spectroscopy was done using KBr pellet method. The point zero charge (PZC) of CMSP surface was determined by salt-addition method (Bakatula *et al.*, 2018). In this method, the 0.01M KCl solution of different pH ranging from 2 to 11 were prepared using 1M HCl and 0.1M NaOH solutions. Fixed amount of CMSP (0.2g) was mixed in 20mL of 0.1MKCl solutions of all desired pH. The mixture was kept in incubator shaker at 120rpm for 24 hours. The PZC was determined by plotting graph between initial pH and Δ pH (initial pH~final pH) after 24hours. The CMSP

sample was analysed for the surface area and porosity by BET technique (model- Micromeritics' Gemini VII 2390) in nitrogen gas atmosphere.

Adsorbate preparation and characterization: The reagent grade azo dye MB (MW. 319.86gm/mole, Fig. 1b.) was obtained from Spectrochem Ltd., Mumbai, India. One litre stock of 1000mg/L MB concentration was made taking 1g MB in 1L distilled water and stored in amber glass bottle. This stock solution was used for the entire experimental work using dilution method. The qualitative and quantitative analysis were done using UV-Visible spectrophotometer (model- Lab India UV-3000⁺). All quantitative analysis of MB was done at λ_{max} 668nm and calibration curve were prepared for the range of 0-25mg/L dye concentration.

Batch biosorption experiment: Full factorial batch biosorption experiments were conducted taking 40 mL dye solution in 50mL conical flasks. Three C_i (50,100 and 200mg/L), four adsorbent dosage (0.25, 0.5, 1 and 1.5g) were taken at natural pH and room temperature (300K). One mL of sample was taken from each flask at fixed time interval (10 - 260 minutes) to study the sorption kinetics. All the observations were done using triplicate sampling. The amount of dye removal and removal efficiency (%) were determined using Eq. 1. and Eq. 2. To figure out the nature of biosorption process, the experimental data was employed to the Langmuir (Eq.3) and Freundlich (Eq.5) sorption isotherms models. The sorption equilibrium parameter or Langmuir separation constant (R_L) was determined from the Eq. 4. The PFO (Eq.6), and PSO (Eq.7) kinetic models were used to determine rate constants and kinetic behaviour of the biosorption process.

$$q_e = (V(C_0 - Ce))/m \quad (1)$$

$$\text{Dye removal (\%)} = ((C_0 - Ce)/C_0) 100 \quad (2)$$

$$(1/q_e) = \{1/(K_a q_m)\} (1/Ce) + (1/q_m) \quad (3)$$

$$R_L = 1 / (1 + K_a C_0) \quad (4)$$

$$\ln q_e = \ln K_f + (1/n) (\ln Ce) \quad (5)$$

$$\ln(q_e - q_i) = \ln q_e - K_1 t \quad (6)$$

$$(t/q_i) = (1/K_2 q_e^2) + (1/q_e) \quad (7)$$

where, q_e (mg/g) represents dye uptake at equilibrium, C_0 (mg/L) - the initial concentration of MB; C_e (mg/L) - equilibrium concentration of MB; m (g) - the dosage of CMSP used; K_1 (min^{-1}) - PFO rate constant; K_2 (g/mg.min) - PSO rate constant; q_m (mg/g) - dye uptake capacity (Max.); K_a (L/mg) - isotherm constant (Langmuir); K_f (mg/g) - isotherm constant (Freundlich) and n represents heterogeneity factor for sorption intensity.

Results and discussion

The SEM analysis provides fundamental structural morphology and property of adsorbent. The CMSP surface roughness associated with grooves and scaffolds was observed in the SEM analysis at 2700X magnification (Fig. 2a.), which in turns results in high surface area and porosity. The major elemental composition of CMSP contained 68.35 % carbon, 7.70 % hydrogen, 2.07 % nitrogen and 2.35 % sulphur. The FTIR spectrum (Fig. 2b) represents various functional moieties related to lignocellulosic matrix. The broad band around 3349 cm^{-1} show the presence of -OH groups and 3051 cm^{-1} , 2925 cm^{-1} , 2857 cm^{-1} suggest C-H stretch from alkane, alkene and amines. The band at

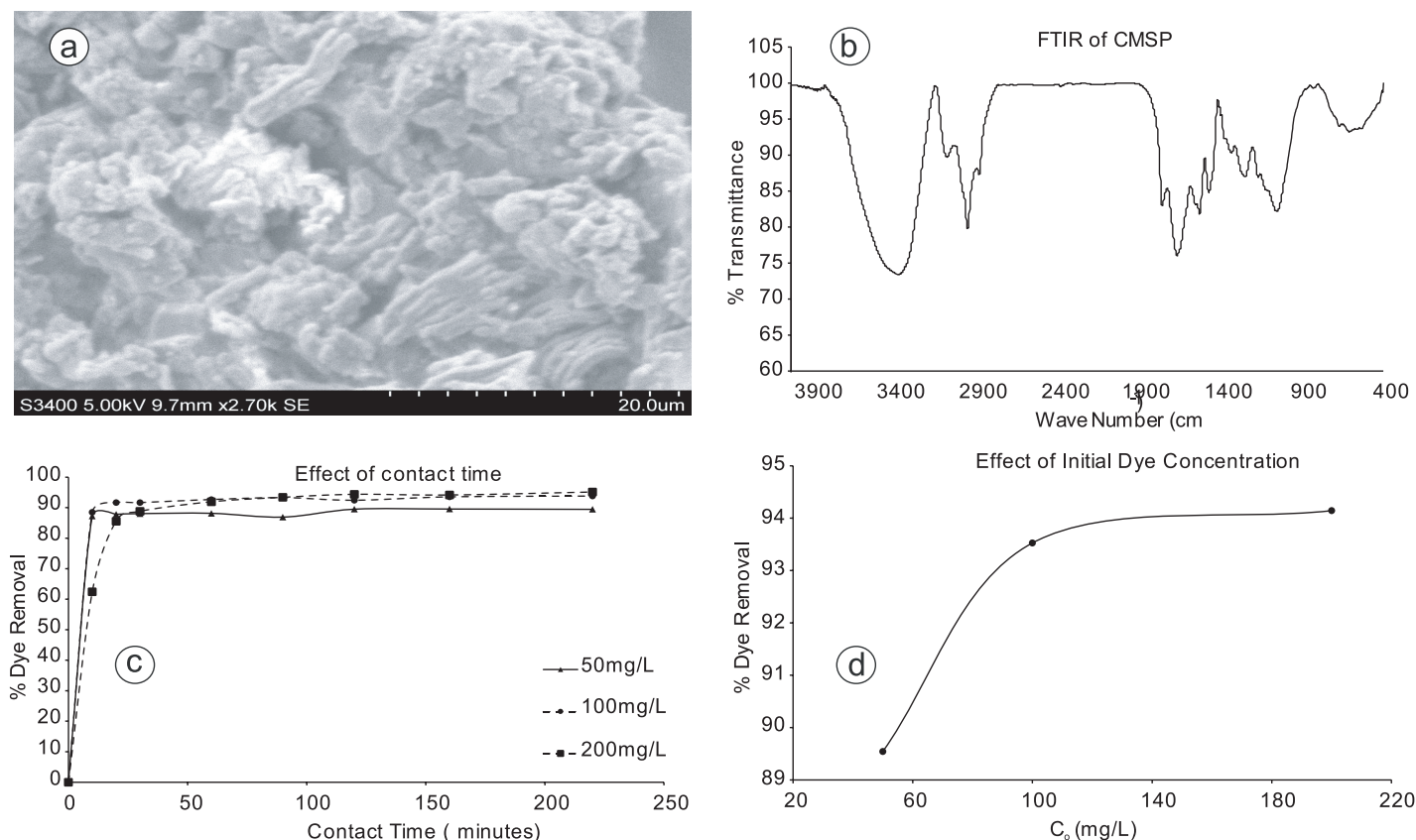


Fig. 2. (a) Scanning Electron Micrograph of *Cordia myxa* seed powder, (b) FTIR spectrum of CMSP, (c) Effect of t (contact time) on MB removal by CMSP and (d) Effect of C_0 (initial concentration) on MB removal by CMSP.

1743 cm^{-1} indicates presence of C=O esters and lactone structures and availability of glycosidic linkages. The signals related to nitro and amine groups were reported at 1654 cm^{-1} , 1516 cm^{-1} , 816 cm^{-1} and phenolic -OH, primary -OH at 1321 cm^{-1} , 1077 cm^{-1} which indicates presence of cellulose, hemicellulose and lignin containing structures. The PZC of the adsorbent material gives the information of the overall electrostatic nature of particle surface in accordance with the interaction of available functional groups and particular pH of liquid medium. Overall electrostatic charge of the adsorbent surface would be -ve, if the solution pH is higher than PZC and vice-versa. The PZC of the CMSP was observed to be 6.5 which indicates favourability of MB sorption on more than 6.5pH of the MB solution (Bakatula *et al.*, 2018; Rosas-Castor *et al.*, 2014). The BET surface area (SA), pore diameter (PD) and volume (PV) were found to be 64.113 m^2/g , 2.364nm and 0.135 cc/g respectively. The observed SA, and PV were comparatively higher than previously reported studies on corn (1.53 m^2/g , 0.007 cc/g), alfa-alfa (1.55 m^2/g , 0.008 cc/g) and agave (2.53 m^2/g , 0.010 cc/g) as biosorbent for MB (Rosas-Castor *et al.*, 2014). Higher porosity of CMSP can be attributed to its smaller PD with compare to corn (21.77nm), alfa-alfa (25.1nm), and agave (29.20nm) biomass.

The dye removal increases in response to increased contact time and process reaches at equilibrium after around 1 hour of contact time (Fig. 2c). Initial rapid increase indicates the higher interaction of dye molecules on the available sites of biosorbent surface and which decreases after achieving equilibrium state. It is observed that the increase in the C_0 have positive impact on the dye removal percentage (Fig. 2d). The dye removal (%) was observed to be increased from 89.54 % to 94.14 % with C_0 from

50mg/L to 200mg/L respectively and 0.5g CMSP dosage at the equilibrium. It can be associated with the increased molecular interactions of adsorbate and adsorbent surface which ultimately results to surpass the resistance of mass transfer for the dye molecules on the adsorbent (Idan *et al.*, 2018).

Adsorption equilibrium and reaction kinetic study provides an essential information for the design and optimization of the adsorption unit operation in the wastewater treatment process. Equilibrium isotherms gives the information of the interaction and adsorption capacity (mg/g) of particular sorbent for the specific pollutant at constant temperature. Applicability of isotherms models can be compared by comparing correlation coefficient (R^2) values of each applied model. The Langmuir and Freundlich models are widely tested to understand the nature and applicability

Table 1. Equilibrium isotherms and kinetics parameters of MB sorption on CMSP

	Adsorption isotherms		
	Langmuir model	Freundlich model	
q (mg/g)	21.9298	K_f (mg/g)	0.8263
K_a (L/mg)	0.03618	n	1.2206
R^2	0.9898	R	0.9996
R_L @ 50mg/L	0.3559		
R_L @ 100mg/L	0.2164		
R_L @ 200mg/L	0.1213		
Adsorption kinetics			
	PFO	PSO	
q_e (mg/g)	1.70131	q_e (mg/g)	7.4850
Rate constant K_1 (min^{-1})	0.0067	Rate constant K_2 (g/mg.min)	0.00139
R^2	0.7456	R^2	0.9999

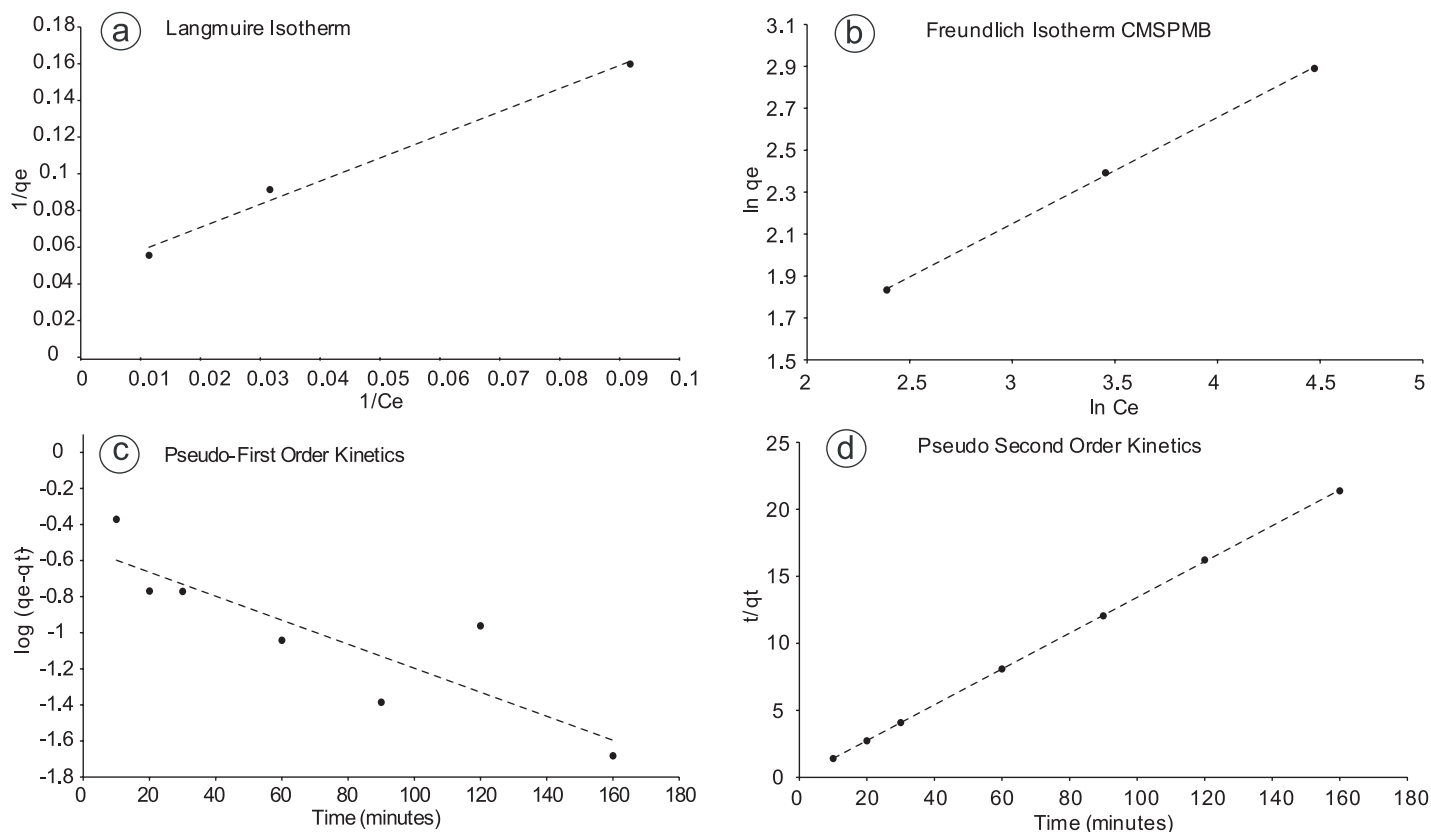


Fig. 2. (a) Langmuir isotherm of MB sorption on CMSP, (b) Freundlich isotherm for MB removal on CMSP, (c) PFO plot for MB removal on CMSP and (d) PSO plot for MB removal on CMSP.

of adsorption systems. Langmuir model assumes homogeneous adsorbent surface with non-interacting reaction sites and equal distribution of binding energies for monolayer formation. On the other hand, Freundlich model which is empirically derived, rely on the assumption of heterogeneous surface of the adsorbent for interaction. Table 1 presents the parameters of the equilibrium isotherms and sorption kinetic. Results of the equilibrium isotherms (Fig. 3a, 3b) shows better correlation to the Freundlich model ($R^2 = 0.9996$) than the Langmuir isotherm model ($R^2 = 0.9898$). It indicates the heterogeneous surface sorption process is taking place in this reaction. The Langmuir maximum uptake capacity was calculated to be 21.9298mg/g, which is comparable to some of the reported biosorbent *i.e.*, rice husk (4mg/g), wheat shells (16-22mg/g), date peats (15-50mg/g) banana and orange peel (12-14mg/g) waste (Hubbe *et al.*, 2011). The equilibrium constant (R_L) provides the nature of adsorption process. If the value of R_L falls zero, 1, from 0 to 1 and more than 1 can be assigned as irreversible, linear, favourable, and unfavourable sorption process, respectively (Hameed and Hakimi, 2008). The values of equilibrium constant ($R_L = 0.3559, 0.2164, 0.1213$) indicates the favourability of sorption process ($0 < R_L < 1$) at all three selected initial concentration of dye *i.e.* $C_0 = 50, 100, 200$ mg/L respectively. The higher value of Langmuir separation constant at higher C_0 suggests the favourability of sorption process at higher C_0 and, it is evident from the Fig. 3d. which shows the increase in the MB removal with increase in the C_0 of the solution. Freundlich isotherm constant (n) which is the function of sorption capacity and intensity is found to be 1.2206 which falls in the range of favourability ($1 < n < 10$) of the sorption process (Vadivelan and Vasanth Kumar, 2005). The kinetics study reveals the progression of dye uptake process

which is crucial for the development of reactor design criteria. From the Fig. 3c and 3d and Table 1, it is well evident that correlation coefficient value for PFO ($R^2 = 0.7456$) found to be very lower than the PSO ($R^2 = 0.99$). It predicts the chemisorption and intraparticle diffusion mechanism are predominant in MB sorption on CMSP. The equilibrium concentration C_e (7.48mg/L) and experimental data were approximately in accord for the PFO model. The values of rate constants for PFO and PSO were found to be $K_1 = 0.0067 \text{ min}^{-1}$ and $K_2 = 0.00565 \text{ g/mg.min}$ for the MB sorption on CMSP.

Agricultural waste has been identified as potential element for the circular bioeconomy development. It has been employed in many fields such as sustainable biofuel, development of nutritional products and pollution removal etc. The present study reported a novel biomass *C. myxa* seed powder for the decontamination of azo dye MB containing wastewater by batch biosorption process. It showed maximum dye uptake of 21.92mg/g which is comparable to the efficiency reported by other authors with another biomasses. The sorption process explained well with Freundlich isotherm and PFO kinetics models. This preliminary study suggests more research that need to be carried focussing on the surface modification and process parameter optimization for developing CMSP as alternative to available costly and ecologically negative options.

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