Facile Fabrication of Quaternized Sisal Fiber by Electron Beam Radiation and its Effective Adsorption for Indigo Carmine from Aqueous Solution

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Abstract

The removal of Indigo carmine (IC) from the aquatic environment is necessary due to its high toxicity. In this study, methacryloxy ethyltrimethyl ammonium chloride (DMC) modified sisal fiber (SF-DMC) was prepared by a one-step process using radiation induced grafting polymerization. The adsorption performance of SF-DMC toward IC dye was investigated by batch adsorption experiments. The adsorption kinetic studies shows that adsorption equilibrium reached within 30 min, and it can be well described by pseudo-second-order model. The adsorption isotherms are well described by Langmuir model, and the theoretical maximum adsorption capacity are 709.22 to 892.86 mg/g at different temperature. The adsorption of IC onto SF-DMC is a spontaneous and exothermic reaction and low temperature is favorable for adsorption. Besides, SF-DMC has good selective adsorption for IC in the mixed anionic dyes and in high-salt solution. The dyes on SF-DMC can be desorbed by 2mol/L HCl solution. Therefore, the SF-DMC exhibits excellent adsorption performance, which is suitable for IC removal from high-salt wastewater.

1. Introduction

Indigo Carmine (IC) is a synthesized anionic dye that is widely used as a colorant in foods, cosmetics and dyeing of clothes (blue jeans) (Ketes et al., 2020). The inappropriate release of IC from large-scale use throughout dyeing process will cause environmental problem and severe harm on human health due to its high toxicity (Gopi et al., 2017; Ahmed et al., 2017). Thus, the removal of IC from the aquatic environment is considered to be necessary (Ahmad et al., 2021). Various methods are used to remove IC from effluents, including membranes (Gopi et al., 2017), chemical oxidation (Shu et al., 2016), photo-catalysis (Secula et al., 2020), electro-catalytic (Lei et al., 2021) and biological methods (Borba et al., 2020). However, the application of the mentioned processes is limited because of the relatively high operational costs, low efficiency, greater energy consumption, and sludge generation.

Adsorption is considered as one of the most simple and attractive methods to purify polluted water (Chowdhury et al., 2020; Du et al., 2021). In recent years, the low cost adsorbents based on natural fibers have been recognized as efficient, cost-effective, and environmental friendly for contamination removal purposes (Candido et al., 2021). Owing to small fiber diameter and excellent osmotic stability, the plant based cellulose fibers have been widely investigated for the adsorption of pollutants, including radionuclides (Bai et al., 2020; Li et al., 2020), heavy metals (Huang et al., 2021; Pang et al., 2020), humic acid (Du et al., 2019), phosphate (Du et al., 2019), dyes (Shao et al., 2021), gold ions (Mostofa et al., 2021) and oil/water separation (Meng et al., 2020; Liu et al., 2018) et al. Plant-based cellulose fibers such as sisal, jute, hemp, coir, kapok, cattail, and cotton linter display lots of advantages over synthetic fibers like low-cost, low density, biodegradability, recyclability, environment friendly, and less hazardous over synthetic fibers (Chokshi et al., 2020; Cui et al., 2021; Zheng et al., 2020; Silva et al., 2020). Among them, sisal fiber (SF) is a most noteworthy and industrially promising fibers have attracted particular interest due to its fairly coarse, moderate high specific strength and stiffness, durability, and resistance to deterioration in saltwater (Tesfamariam et al., 2019). The main component of SF is cellulose, which mass

proportion of cellulose accounts for 73% (Filho et al., 2020). Cellulose possesses a reactive surface which bears hydroxyl groups make it very suitable to be chemical modified and used as adsorbent.

The modification of fiber matrix has attracted increasing attention, which are relevant strategies to improve the adsorption performance of plant fibers. Various methods such as carbonization (Melike et al., 2018), plasma treatment (Silva et al., 2020), chemical deposition (Teshamariam et al., 2019), microwave treatment (Gao et al., 2015) and electron or γ -ray radiation techniques (Dong et al., 2021) are used for fiber modification. Specifically, radiation-induced graft polymerization (RIGP) can conveniently introduce the required functional groups onto polymer surface and then endow the polymers with densely accessible adsorption sites (Du et al., 2018).

Electrostatic attractions have been shown to be an effective and versatile force to treat ionic contamination. The quaternary amine type adsorbents are the most commonly used adsorbent for anionic dye removal because of the strong electrostatic reaction between the amine group and the anionic sulfonate group of the dye molecule. The synthesis routes are usually carried out by the modification of primary amine, followed by quaternization (Du et al., 2019; Talet et al. 2008; Wang et al., 2019; Song et al., 2019). The syntheses of these adsorbents are cumbersome and require several steps, which need to be simplified. To our knowledge, if appropriate monomer and synthesis method were devised, facile fabrication of adsorbent will be obtained.

In this paper, we attempted to synthesize a low cost and environmental friendly adsorbent by an one step process through grafting methacryloxy ethyltrimethyl ammonium chloride (DMC) onto SF. Considering the unique advantages of the high stability, ordered hydrophobic pore channels and densely accessible cationic sites, the adsorption performance of SF-DMC are expected to be good for anionic dyes. So, batch adsorption experiments of SF-DMC toward IC were conducted. The effects of solution pH, dosage, adsorption time, initial concentration, ion strength and the coexisted dyes on the adsorption of SF-DMC for IC were studied. The adsorbent may provide a useful alternative for industrial dye removal.

2. Experimental

2.1 Materials

Sisal fiber was obtained from commercial. DMC, HCl, NaOH and IC were obtained from a commercial supplier (Macklin Chemical Reagent Co., Ltd).

2.2 Preparation of SF-DMC

SF was boiling treated by 5% NaOH for 30 min, washed and dried at 40 °C. SF with mass of 2 g was vacuum sealed in polyethylene bags. 30 wt% DMC aqueous solution was nitrogen-bubbled to remove oxygen in solution. Then 50 mL DMC solution were extracted using a medical syringe and injected into the bags. The bags were irradiated by the electron beam by an accelerator (1 MeV, Wasik Associates Inc.,

USA) with the dose rate 10 kGy/pass. The total dose ranged from 0 to 60 kGy. After irradiation, the SF were filtered, washed and dried at 40 °C. Thus, SF was functionalized by DMC (SF-DMC).

The grafting yield (GY) was calculated using the mass increase using equation (1):

$$GY = \frac{W_g - W_0}{W_0} \times 100\%$$

1

where W_0 and W_g are the mass of SF before and after grafting, respectively.

2.3 Characterization

Fourier transform infrared spectroscopy (FTIR) spectra was tested by spectrophotometer (Bruker Tensor 27). The morphologies were observed by scanning electron microscope (SEM) (Tescan Vega 3).

Thermogravimetric analysis (TG) curves were recorded using a TGA 55 (TA instruments) with heating rate 10 °C/min. Zeta potential was tested on a 90 Plus PALS system. The concentration of IC and MO was determined using a UV-vis spectrophotometer (UV-3600, Shimadzu) at 610 nm and 468 nm, respectively.

2.4 Batch Adsorption Experiments

In the pH effect studies, 0.03 g of dry SF-DMC was placed in a glass bottle containing 100 mL of 100 mg/L IC solution. Different pH values were adjusted by using 0.5 M HCl or NaOH. In the adsorption kinetic studies, 0.02 g SF-DMC was used and the adsorption times were 1, 3, 5, 10, 15, 20, 30, 60, 90, and 120 min. In the adsorption isotherm studies, 0.02 g and 100mL was used and the IC concentration ranged from 100 to 700 mg/L. The adsorption capacity (Q) and removal percent (R) of SF-DMC to IC was calculated using equation (2) and (3):

$$Q_t = \frac{(C_0 - C_t) \times V}{m}$$

2

$$R = \frac{C_0 - C_e}{C_0} \times 100\%$$

3

where C_0 and C_t are the IC concentrations before and after adsorption, respectively; V is the solution volume, and m is the weight of SF-DMC. The averages of triplicate measurements were used as the final adsorption data.

3 Results And Discussion

3.1 Preparation

Figure 1 shows the effect of radiation dose on the *GY* of DMC grafting onto SF. The *GY* increased with increasing radiation dose, and reached a maximum value of 73% at 60 kGy. This results can be explained by the decay mechanism of trapped radicals. The mechanism of RIGP is mainly a free radical reaction, and the grafting yield is determined by the total free radicals formed both in the monomer solution and the substrate (Zhang et al., 2012). The total free radicals increased with the increasing radiation dose. The grafting polymerization mainly occurs at the interface between monomer and polymers, so, *GY* finally reached at 60 kGy. Moreover, a higher radiation dose will result in the decomposition of cellulose content, thereafter lead the *GY* decreasing. Then, the SF-DMC with a *GY* of 73% was used for further characterization and adsorption studies.

3.2 Characterization

FTIR spectra were used to verify the grafting of DMC onto SF (Figure 2a). The spectral bands at 3335, 2917, 1637, and 1036 cm^{-1} was due to O-H, C-H, H-O-H, and C-O bonds of cellulose, respectively (Du et al., 2019b; Chattopadhyay et al., 2017). The band at 1726 cm^{-1} is the characteristic of the carboxyl group (C=O) (Li et al., 2017). The bands at 1470 and 948 cm^{-1} are the bending vibration of the C-N⁺(CH₃) in methylene group and DMC, respectively (Yang et al., 2015; Udoetok et al., 2016). The two peaks obtained from the SF-DMC sample confirmed the successfully grafting of DMC onto the SF.

Figure 2b shows the TG analysis of the SF and SF-DMC samples. The weight loss at low temperature (25-100°C) corresponded to water dehydration. For the SF, the weight loss zones were located at 320-380°C and 380-580°C. After DMC grafting, the two weight loss zones located at 250 to 300°C and 300 to 450°C, which appeared at low temperature compared to SF, which corresponding to the decomposition of DMC and cellulose, respectively.

Figure 3 shows the SEM images of SF and SF-DMC. The SF exhibited a fiber bundle structure with diameters about 250 μm . The lignin and hemi-cellulose were removed by NaOH treatment and the fiber shape is exposed. After DMC grafting, the fiber bundle splitted into several thinner fibers with diameter of 30-40 μm .

3.3 Dye uptake experiments

3.3.1 pH effect and Zeta potential

Solution pH plays a significant role in controlling the adsorption process. The IC adsorption capacity by SF-DMC was tested at various pH and shown in Figure 4 (a). The adsorption capacity was higher at all the pH range. The concentration of IC after equilibrium adsorption was nearly zero, which means that all the IC in aqueous solution was completely adsorbed. Zeta potential (mV) of SF-DMC was tested and shown in Figure 4 (b), which were positive at a wide pH range. So the positive adsorption site will generate strong electrostatic attraction to the anionic IC molecule at a wide pH range, thus resulted in the high adsorption capacity with pH independent.

3.3.2 Effect of dosage

Figure 5 (a) displays the dosage effect of SF-DMC on IC adsorption. The IC removal efficiency increased significantly with increase of SF-DMC dosage. This is attributed to the more functional groups of SF-DMC were worked for IC adsorption. The removal efficiency reached 99.8% at 0.015 g/L. The adsorption capacity (Q_e) was decreased with increase of the SF-DMC dosage and it was 500 mg/g at dose 0.2 g/L. In this study, the dosage (0.02 g SF-DMC in 100 mL IC) was conducted for further experiment.

3.3.3 Adsorption Kinetics: Effect of Contact Time

Figure 5 (b) shows the adsorption kinetics with the initial concentration of 100 mg/L. It can be seen that the adsorption capacity increases with increasing contact time and the adsorption process reached equilibrium at 30 min. Three models, pseudo-first-order (equation 4), pseudo-second-order model (equation 5), and intra-particle diffusion model (equation 6) were used to evaluate the adsorption kinetics. The initial adsorption rate h_0 (mg/g·min) ($t \rightarrow 0$) was expressed as equation (7) (Hadid et al., 2021).

$$Q_t = Q_e(1 - e^{-k_1 t}) \quad (4)$$

$$Q_t = \frac{k_2 Q_e^2 t}{1 + k_2 Q_e t} \quad (5)$$

$$Q_t = k_p t^{1/2} + I \quad (6)$$

$$h_0 = k_2 Q_e^2 \quad (7)$$

where Q_t and Q_e are the amounts of IC adsorbed per gram SF-DMC at time t and at the equilibrium time, respectively.

Table 1 presents the values of the linear correlation coefficient (R^2), Q_e , k_1 , and k_2 . Comparison of these values showed that the R^2 of the pseudo-second-order model was higher than that of the pseudo-first-order model. The pseudo-second-order model described the kinetic data well (Figure 5c), suggesting that the IC adsorption onto the SF-DMC was a chemical adsorption process (Wu et al., 2021). The adsorption capacity was 500 mg/g, calculated using the pseudo-second-order model, which was in accordance with the experimental data.

The kinetic data is also described using the intra-particle diffusion model and the Weber-Morris plots are shown in Figure 5 (d). The plot is not straight, but presents multiply distinct regions. According to this model, the first and second linear parts corresponded to surface diffusion and intra-particle diffusion and the third region mean equilibrium adsorption. The first linear part did not pass through the origin, suggesting that intra-particle diffusion is not the sole rate determining step (Du et al., 2020).

Table 1
Kinetic parameters obtained from kinetics model
of the SF-DMC

Kinetic model	Parameters	Values
Pseudo-first-order	K_1	0.7068
	Q_e	473.13
	R^2	0.9346
Pseudo-second-order	K_2	0.0023
	Q_e	500.00
	R^2	0.9998
	ho	571.43
Intra-particle diffusion	R^2	0.9982
	K_{p1}	164.20
	I	7.2148
	R^2	0.9171
	K_{p2}	30.484
	I	357.08

3.3.4 Adsorption Isotherms: Effect of initial concentration

The adsorption isotherms were used to describe the distribution of target compounds between the solid and liquid phase at equilibrium. Figure 6 (a) shows the experimental data along with the fitted isotherm model curves of IC adsorption onto SF-DMC conducted at 298, 308, and 328 K.

Modeling of the experimental data using appropriate isotherm model is often used for prediction of the adsorption mechanism. In this study, Langmuir, and Freundlich models were fitted to the adsorption isotherms, which can be expressed as equation (8) and (9) (Hu et al., 2021):

$$Q_e = \frac{Q_m K_L C_e}{1 + K_L C_e}$$

$$Q_e = K_F C_e^{1/n}$$

where Q_e (mg/g) and C_e (mg/l) are the equilibrium adsorption capacity and equilibrium concentration, Q_m (mg/g) is the maximum adsorption capacity.

The isotherm constants obtained in this study are given in Table 2. Langmuir model have highest R^2 value (>0.99) at all temperatures, which suggested the adsorption of IC formed a monolayer on SF-DMC (Yang et al., 2021). The theoretical maximum adsorption capacity of IC onto SF-DMC was from 709.22 to 892.86 mg/g at temperature ranged from 298 to 328 K. In addition, it is observed that the adsorption capacity is negative correlated with temperature. According to Freundlich model, constant n gives an idea about the favor of the adsorption process. The n is greater than 1, indicating that IC was very favorable adsorbed by the SF-DMC (Hossain et al., 2021).

The maximum adsorption capacities of SF-DMC for IC were compared with other reported adsorbents given in Table 3. The SF-DMC had higher adsorption capacities than most of the other adsorbents. The small specific gravity and fiber diameter of SF combined with the advantage of RIGP, causing the large amounts of DMC monomer onto the surface of SF, which made SF-DMC very suitable for IC dye removal from aqueous solution.

Table 2
Key parameters and correlation coefficients of isotherm models for IC adsorption by SF-DMC

Temperature (°C)	Langmuir			Freundlich		
	Q_m	K_L	R^2	K_F	n	R^2
298 K	892.86	0.0225	0.9986	398.25	8.2977	0.9827
308 K	793.65	0.0292	0.9984	456.66	12.898	0.9000
323 K	709.22	0.0844	0.9999	569.89	32.237	0.9350

Table 3
comparisons of adsorption capacity of SF-DMC with other available adsorbents.

Adsorbent	Q _{max} (mg/g)	Reference
Brazil nut shells	1.09	(Brito et al., 2009)
Pistia stratiotes dry biomass	41.2	(Ferreira et al., 2019)
Rice Husk Ash	29.3- 65.9	(Lakshmi et al., 2019)
Mg/Fe LDH	62.8	(Ahmed et al., 2021)
Chitosan aerogels	168.6	(Luna et al., 2019)
Activated Carbon	298.3	(Zahia et al., 2019)
Nanofiber membranes	266.8	(Li et al., 2012)
carbonaceous nanofillers (graphite, graphene and graphene oxide)	380	(Galzerano et al., 2021)
chitosan/graphene oxide aerogels	534.4	(Luna et al., 2019)
CS/STPP	500	(Kekes et al., 2020)
CS/β-CD STPP	1000	(Kekes et al., 2020)
SF-DMC	709.22- 892.86	This paper

3.3.5 Thermodynamics: effect of temperature on adsorption

A series of adsorption experiments are conducted at 298-323 K to determine the adsorption was endothermic or exothermic in nature. The change in free energy (ΔG), enthalpy (ΔH), and entropy (ΔS) for the IC adsorption process are evaluated by Equation (10) to (11) (Mladenovic et al., 2021)

$$\ln K_d = \frac{\Delta S}{R} - \frac{\Delta H}{RT} \quad (10)$$

$$\Delta G = \Delta H - T\Delta S \quad (11)$$

where, K_d is the equilibrium constant obtained from Q_e/C_e , R is universal gas constant (8.3144 J/mol × K) and T is the temperature (K).

Figure 6 (d) shows the linear plot of thermodynamics at different initial concentration. The values of ΔH , ΔS and ΔG at different temperature were calculated from the slope and intercept by the linear plot and listed in Table 4. $\Delta G < 0$, $\Delta H < 0$ means that the IC adsorption by SF-DMC was a spontaneous and exothermic reaction. The ΔG values decreased as the adsorption temperature increasing, which means that the adsorption was more favorable at lower temperature (Ata et al, 2012). The result was consistent with the results of Langmuir model.

Table 4
Thermodynamic parameters of the adsorption of IC on SF-DMC

C_0 (mg/L)	ΔH (KJ/mol)	ΔS (J/mol/K)	ΔG (KJ/mol)		
			298K	308K	323K
300	-5.35	-4.66	-3.96	-3.91	-3.84
500	-6.23	-14.14	-2.02	-1.87	-1.66
600	-10.42	-29.78	-1.55	-1.25	-0.80
800	-10.76	-33.05	-0.91	-0.58	-0.08

3.3.6 Effect of NaCl Concentration

The effect of NaCl on the adsorption performance is shown in Figure 7 (a). The initial concentration of the IC was 100 mg/L. C_{IC}/C_{NaCl} represents the molar concentration ratio of IC/NaCl. With increasing molar concentration of NaCl, the adsorption capacity of IC was little decreased. When the concentration of NaCl was 1000 times that of the IC, the adsorption capacity was 61.8% of the adsorption capacity without NaCl.

3.3.7 Regeneration and Reusability

The adsorption-desorption experiments were carried out for six cycles and shown in Figure 7 (b). After full adsorption (0.02 g SF-DMC added to 100 mL of 100 mg/L IC), the IC loaded SF-DMC was regenerated using HCl at different concentration. The desorption (%) by 1M HCl was 75%, and nearly 100% by 2 M HCl. So, 2 M HCl was selected to regenerate SF-DMC for further cycle adsorption. After 6 adsorption-desorption cycles, the removal of IC still remained 95% of the first use. The results suggested that 2 M HCl is a good elution solution and that the SF-DMC can be used repeatedly for IC removal.

3.3.8 Selective adsorption

It is essential to investigate the selective adsorption performance, thus to well understand the relationship between the adsorbent and dyes to guide the design and fabrication. The competitive adsorption of IC and methyl orange (MO) in binary mixture solution was investigated. Figure 8 (a) shows the UV-vis spectra of IC and MO. The absorption of IC at λ_{max} 468 nm is negligible compared to MO and the absorption of MO at 610 nm is negligible compared to IC. The selectivity coefficient $\alpha_{IC/MO}$ was calculated using equation (12) (Lin et al., 2015; Zhou et al., 2018):

$$\alpha_{IC/MO} = \left(\frac{Q_{IC}}{Q_{MO}} \right) \left(\frac{C_{MO}}{C_{IC}} \right)$$

where Q_i and C_i ($i = \text{IC or MO}$) are the adsorption amounts (mg/g) of i , and the equilibrium concentrations (mg/L).

The $\alpha_{\text{IC}/\text{MO}}$ values of SF-DMC for IC versus MO adsorption at different molar concentration ratio are shown in Figure 8. (b). $\alpha_{\text{IC}/\text{MO}}$ decreased when the molar ratio of MO increased and reached 72.55 at the molar concentration ratio 1:1. $\alpha_{\text{IC}/\text{MO}}$ was 11.29 when the molar ratio of IC/MO was 1:5, indicating that SF-DMC have more preferential adsorption for IC than MO. The electrostatic attraction between the quaternary ammonium group of SF-DMC and the sulfonic acid group of anion dyes IC molecule induced the adsorption. IC molecules had two sulfonic acid groups while MO had only one, which caused the competitive adsorption for IC than MO.

Conclusion

SF-DMC was synthesized by one-step grafting DMC onto SF using electron beam radiation. The results showed that SF was successfully quaternized. The maximum GY value obtained 73% at 60 kGy. The SF-DMC have good adsorption performance to IC such as pH independent, fast adsorption rate, high adsorption capacity, selective adsorption and repeated use. The adsorption kinetics and isotherms of SF-DMC were well obeyed the pseudo-second-order kinetics and Langmuir model respectively. The theoretical maximum adsorption capacity of IC onto SF-DMC was from 709.22 to 892.86 mg/g. The IC adsorption by SF-DMC was a spontaneous and exothermic, which was more favorable at lower temperature. SF-DMC have more preferential adsorption for IC than MO with a selectivity coefficient 72.55 at the molar ratio 1:1 of IC than MO. SF-DMC can be efficiently regenerated by 2 M HCl and repeated use at least for 6 times without the adsorption capacity obvious decrease.

Declarations

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Conflict of interest The authors have no competing interests to declare that are relevant to the content of this article.

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Figures

Figure 1

Effect of radiation dose on GY

Figure 2

FTIR spectra (a) and TG analysis (b) of SF and SF-DMC

Figure 3

Surface morphology of SF (a) and SF-DMC (b)

Figure 4

Effect of pH for IC adsorption (a) and Zeta potential of SF (b)

Figure 5

Effect of dosage on the adsorption (a); effect of adsorption time on adsorption IC (b); pseudo second-order (c) and intra-particle model (d)

Figure 6

Adsorption isotherms of IC onto SF-DMC; effect of initial concentration (a); Langmuir model (b) ; Freundlich model (c) ; and Van't Hoff plots at different concentration (d)

Figure 7

Effect of NaCl on the adsorption capacity (a) and removal efficiency of IC after 6 times regeneration (b)

Figure 8

UV-vis spectra of IC and MO (a) and effect of IC/MO molar ratio on the selective adsorption (b).