



GRATED COCONUT RESIDUE MODIFIED BY A CATIONIC SURFACTANT FOR REMOVAL OF REACTIVE ORANGE 16 DYE

(Sisa Kelapa Parut Diubahsuai oleh Surfaktan Kationik untuk Menyingkirkan Pewarna Reaktif Oren 16)

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Abstract

Hexadecylpyridinium chloride monohydrate, a cationic surfactant, was employed to chemically modify coconut-grated residue to adsorb reactive orange 16 (RO16). The pH point of zero charge (pHPZC) and surface area were used to characterise the raw coconut grated residue (RGC) and the surfactant-modified grated coconut residue (SMGC). The pHPZC was 5.5, and the Brunauer-Emmett-Teller surface area was $1.86 \text{ m}^2 \text{ g}^{-1}$. The dye solution pH and dose effects on RO16 adsorption were tested in a batch adsorption system. Maximum sorption was seen at an SGM concentration of 10 g L^{-1} , while optimal adsorption was observed at a low pH. When dye-loaded SGM was exposed to a basic solution, the desorption percentage increased to almost 70%, whereas low desorption was observed under acidic conditions. Furthermore, the isotherm study discovered that the Freundlich model explained RO16 adsorption of SMGC better than the Langmuir model. The Langmuir isotherm recorded a maximum adsorption capacity of 9.72 mg g^{-1} .

Keywords: cationic surfactant, coconut, reactive dye, adsorption

Abstract

Heksadekilpiridinium klorida monohidrat, sejenis surfaktan kationik, telah digunakan untuk memodifikasi kimia sisa parut kelapa untuk menyerap reaktif oren 16 (RO16). pH titik cas sifar (pHPZC) dan luas permukaan digunakan untuk pencirian sisa parut kelapa mentah (RGC) dan sisa parut kelapa yang dimodifikasi dengan surfaktan (SMGC). Nilai pHPZC adalah 5.5, dan luas permukaan Brunauer-Emmett-Teller adalah $1.86 \text{ m}^2 \text{ g}^{-1}$. Kesan pH larutan pewarna dan dos terhadap penyerapan RO16 telah diuji dalam sistem penyerapan kelompok. Penyerapan maksimum dilihat pada kepekatan SGM 10 g L^{-1} , manakala penyerapan optimum diperhatikan pada pH rendah. Apabila SGM yang memuatkan pewarna terdedah kepada larutan alkali, peratus penyerapan semula meningkat hampir ke 70%, manakala penyerapan rendah diperhatikan dalam keadaan berasid. Selain itu, kajian isotherm menemui bahawa model Freundlich menjelaskan penyerapan RO16 pada SMGC dengan lebih baik daripada model Langmuir. Isotherma Langmuir mencatatkan kapasiti penyerapan maksimum sebanyak 9.72 mg g^{-1} .

Kata kunci: surfaktan kationik, kelapa, pewarna reaktif, penyerapan

Introduction

Many dye effluents are harmful to the environment and wildlife [1]. Global production of dye chemicals for commercial and industrial use totals approximately 700,000 tons annually, representing over 100,000 different colours [2]. Reactive dyes are among the popular choices for dyeing cotton and other cellulose fabrics due to their bright colour shades [3] and are readily water-soluble and easy to apply [4]. According to Allègre et al. [5], over 80,000 tons of reactive chemicals are produced annually, enabling pollution estimates given their extensive application, and after dyeing processes, treatment baths are discharged, including an initial dye bath with extremely high salt concentration and dark hue from substantial organic material resulting from additives used to facilitate dye uptake during fabric treatment. This causes environmental concerns as only 30% of reactive dye typically binds with fabric, meaning that the remaining unfixed dye gives colour to their effluent [6].

Dyes have been removed from effluent using various methods [7]. However, adsorption remains one of the

best methods for removing various colour substances [8]. Due to some limitations associated with using activated carbon as an adsorbent, such as processes for regeneration and reactivation [9], alternative adsorbents sourced from plants have been actively explored. Many works have found the potential of plant-based adsorbents to remediate various water and wastewater pollutants [10].

Agricultural waste applied to treat wastewater has several advantages, including chemical stability, an abundant source, low cost, and environmental friendliness [11]. Many have reported that modifying raw agricultural waste improves sorption capacity [12]. Meanwhile, the successful modification of biomass with cationic surfactant in removing reactive dye has been extensively reviewed [13]. According to Kosaiyakanon and Kungsanant [14], cationic surfactants increased the adsorption affinity of negative dyes and introduced a positive charge to adsorbent surfaces. This enables the biosorbent surface and negatively charged anionic dye to attract electrostatically [15], as illustrated in Figure 1.

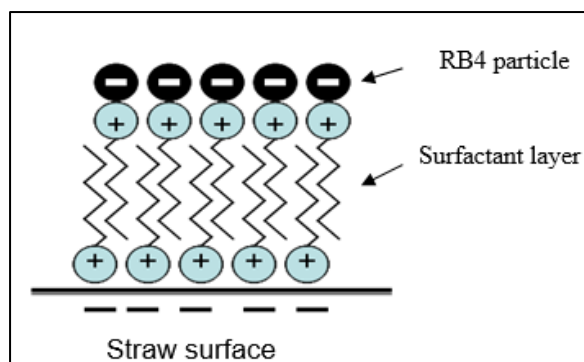


Figure 1. Schematic diagram showing reactive dye attracted to opposite charges on modified straw [15]

This study investigated the effectiveness of the cationic surfactant hexadecylpyridinium chloride monohydrate (CPC, $C_{21}H_{38}NCl$) in removing the anionic dye reactive orange 16 (RO16) from an aqueous solution. According to earlier studies, CPC cations on the adsorbent surface would modify surface characteristics and improve anionic contaminant adsorption [15,16]. Coconut flesh residue has been extensively studied to remove heavy metals like Cd (II) ion [17], Pb(II) ion [18], Ni(II) ion [19] and removal of dyes [20].

Materials and Methods

Materials

Reactive orange 16 (Figure 2) and CPC (98% purity) were purchased from Aldrich. Meanwhile, hexane was obtained from R&M Chemicals (UK). Distilled water was used throughout the adsorption experiments.

Preparation of surfactant-modified grated coconut residue

The preparation of surfactant-modified grated coconut residue (SMGC) has been reported before [21]. In summary, SMGC was prepared by shaking 1 L (2.50 mmol/L) CPC with a specific amount of dried, hexane-pretreated raw grated coconut residue (RGC). The treated grated coconut residue was separated from the liquid, washed with distilled water, and dried overnight in an oven at 60 °C. An anionic dye solution was made by dissolving the RO16 dye in distilled water.

Characterisation of surfactant-modified grated coconut residue

For characterisation, the pH point of zero charge (pHPZC) was measured using the salt addition method

[22]. Briefly, NaCl solutions were prepared with volumes of 50 mL and a concentration of 0.01 M, covering pH values from 4 to 12. Each solution was supplemented with 0.15 g of SMGC and mixed for 48 hours at 150 rpm. The initial and final pH values were measured to determine the pHZPC, indicating the point of zero charge where ΔpH equals zero, signifying no surface charge. The surface area was measured using the Brunauer-Emmett-Teller (BET) method through N_2 adsorption, employing the Micromeritics 2010 instrument from Instrument Corporation, USA. Surface morphology testing was carried out using a field emission scanning electron microscope (FESEM-Zeiss Supra 40V) at 5000x magnification.

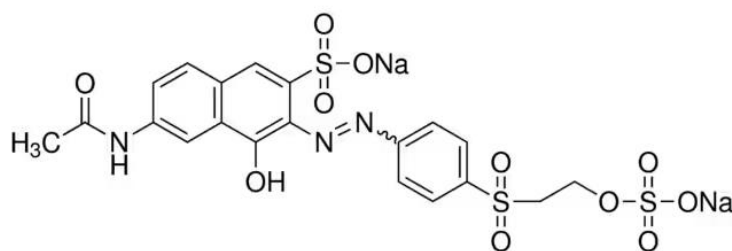


Figure 2. Chemical structure of RO16

Adsorption studies

The dynamic study involved agitating different concentrations of SMGC (10 and 20 g L⁻¹) with RO16 at a concentration of 100 mg L⁻¹. The solution was then sampled at specific time intervals for analysis. In the batch isotherm experiment, SMGC and RO16 were mixed and agitated for 60 minutes. While many parameters can influence the adsorption performance of the reactive dye on the biosorbent, this study focused on two important parameters: the pH of the dye and the dosage of the adsorbent. The SMGC dosage ranged from 5 to 50 g L⁻¹ to examine the impact of different doses, while the pH of RO16 was adjusted from 3 to 9 to assess the pH effect. Throughout the study, samples were shaken using an Innova 2100 platform shaker. The pH of the solution was measured with a Mettler Toledo S20

pH meter, and the concentration of dye samples was determined using a HACH DR2800 portable spectrophotometer (UK) at a maximum absorbance wavelength of 493 nm. The initial pH of the dye solution was 5.5 at a concentration of 100 mg L⁻¹, and the agitation speed, adsorbent size, and dosage were set at 200 rpm, 0.12–0.25 mm, and 10 g L⁻¹, respectively, unless specified otherwise.

Desorption of dye-loaded adsorbent

RO16 desorption from dye-loaded SMGC was achieved by dispersing it at 10 g L⁻¹ in deionised water at various pHs for 3 h. The mixtures were filtered and analysed at 493 nm using a HACH DR2800 portable spectrophotometer. Equation 1 calculates the desorption percentage:

$$\text{Percentage of desorption (\%)} = \frac{q_{e(\text{des})}}{q_{e(\text{ads})}} \times 100 \quad (1)$$

Where $q_{e(\text{des})}$ is the desorption of RO16 (mg g⁻¹) and $q_{e(\text{ads})}$ is the amount of RO16 adsorbed (mg g⁻¹).

Best fitting model estimation

The fit of the equation to the experimental data was established by regression correlation coefficients (R^2) and error estimation. The R^2 nearest to unity is assumed

to fit best, whereas a lower Marquardt's percentage standard deviation (MPSD) (Equation 2) is better for error estimation:

$$\text{MPSD} = 100 \left(\sqrt{\frac{1}{p-n} \sum_{i=1}^p \left[\frac{(q_{e,meas} - q_{e,calc})}{q_{e,meas}} \right]^2} \right) \quad (2)$$

Where p represents the number of experimental data, and n is the model equation's parameters. The experimental data are $q_{e,meas}$ and the model equation data are $q_{e,calc}$.

Results and Discussion

Adsorbent characterisation

The pHPZC of the adsorbents was used to determine the ideal pH for anion adsorption and the nature of the interaction [23]. The calculated pHPZC of the SMGC was 5.50. When the pH is less than the pHPZC, the adsorbent positive surface charge favours anion adsorption [24]. The removal of Remazol Brilliant Blue in industrial waste sludge exhibited an identical behaviour, with a notable increase in removal as the pH decreased [23]. Given the above, physisorption would be expected as the adsorption mechanism due to the anionic dyes' electrostatic interaction with the active centres of the adsorbents, as suggested by Karaman et al. [16] in their research of cationic surfactant-modified carbon for removing Congo Red dye.

The textural analysis showed that the SMGC had a relatively low BET surface area of $1.86 \text{ m}^2 \text{ g}^{-1}$ and pore volume of $2.04 \times 10^{-3} \text{ cm}^3 \text{ g}^{-1}$. These findings suggest that textural properties may not play a significant role in adsorption compared to other factors. For comparison, the reported BET surface area for acid-washed coconut grated in a separate study by Khalid and Hanafiah [20] was $23.673 \text{ m}^2 \text{ g}^{-1}$, significantly higher than SMGC as acid washing exposes more internal surface area by removing compounds and expanding pores. In contrast, Namasivayam and Sureshkumar [25] reported that surfactant modification coats and constricts pore networks of biomass, limiting accessible surface for nitrogen adsorption analysis.

The scanning electron microscopy (SEM) images (Figure 3), captured at 5,000x magnification, revealed that the SMGC exhibited a slightly more uniform surface than the untreated RGC. This coating effect resulted in a smoother morphology, as observed through the SEM analysis of SMGC. These findings are consistent with a study conducted by Chen et al. [26], in which they observed that untreated silkworm exuviae displayed a compact, uneven, and folded surface, while modified silkworm exuviae treated with hexadecyltrimethylammonium bromide, exhibited a porous and flexible surface.

In our previous work [21], we conducted an analysis to confirm the successful attachment of the cationic surfactant CPC to the surface of RGC. This was achieved through functional group analysis using Fourier transform infrared (FTIR) spectroscopy. The FTIR analysis revealed the presence of two distinct new peaks at wavenumbers 2921 and 2853 cm^{-1} in the spectrum of SMGC, which were attributed to the asymmetric and symmetric C-H stretching vibrations of the cationic surfactant CPC, respectively. The emergence of these characteristic peaks provided strong evidence in support of the effective binding of the cationic surfactant to the RGC surface, which was consistent with other reported surfactant-modified biomass studies [27].

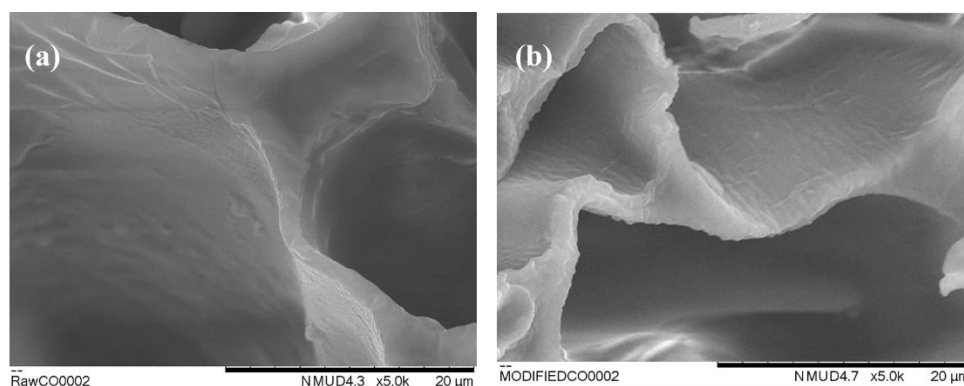


Figure 3. SEM micrographs of (a) RGC and (b) SMGC at 5000x magnification

Dynamic adsorption studies

A rapid initial phase and slow continuation are the characteristics of the adsorption reaction before it reaches equilibrium. For the SMGC dosage of 10 and 20 g L⁻¹, the equilibrium time was 30 min. Two widely used

$$q_t = q_e(1 - e^{-k_1 t}) \quad (3)$$

$$q_t = \frac{q_e^2 k_2 t}{(1 + q_e k_2 t)} \quad (4)$$

Adsorption capacities at time t and equilibrium are denoted by q_t and q_e , respectively, while the first-order and second-order rate constants are denoted by k_1 and k_2 , respectively. Equations 3 and 4 are non-linear equations that were fitted using Polymath software. The results of the fitting are shown in Fig. 4 and Table 1. For both dosages (10 and 20 g L⁻¹), the R^2 for the pseudo-second-order model (>0.98) was slightly higher than that for the pseudo-first-order model (>0.94). The

models, pseudo-first-order and pseudo-second-order, were used to investigate the dynamics of RO16 sorption onto SMGC, as given by Equations 3 and 4, respectively:

MPSD error value was also lower for the pseudo-second-order model (<6.64) than the pseudo-first-order model (<10.76). Furthermore, the pseudo-second-order model's calculated adsorption capacities ($q_{e,cal}$) and experimental adsorption capacities ($q_{e,exp}$) were nearly identical, indicating a better fit of this model to the experimental data. This finding is consistent with other surfactant-modified adsorbent for dye removal [28].

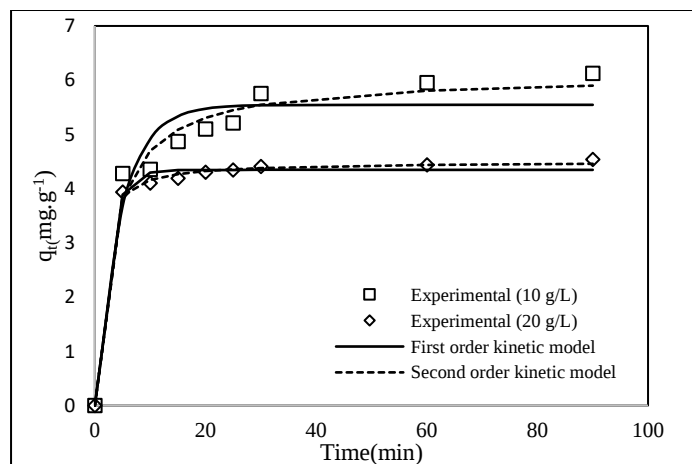


Figure 4. Adsorption dynamic by the nonlinear method for the adsorption of RO16 onto SMGC

Table. 1. Pseudo-first-order and pseudo-second-order rate constants and error analysis.

Experimental	Kinetic Model Constants					Error Analysis			
	Pseudo First Order	Pseudo Second Order	Pseudo First Order	Pseudo Second Order		Pseudo First Order	Pseudo Second Order		
Dosage (g L ⁻¹)	Exp. q _e (mg g ⁻¹)	k ₁ (min ⁻¹)	q _e (mg g ⁻¹)	k ₂ (min ⁻¹)	q _e (mg g ⁻¹)	R ²	MPSD	R ²	MPSD
10	5.75	0.22	5.54	0.05	6.09	0.94	10.76	0.98	6.64
20	4.41	0.45	4.35	0.27	4.50	0.98	3.23	0.99	1.42

Isotherm adsorption studies

Establishing the best equilibrium data correlations will help the adsorption system design [29]. Two well-known models are the Langmuir and Freundlich isotherms, which explain how adsorbate molecules are

distributed in solid phases at adsorption equilibrium. Equation 5 is the Langmuir isotherm model [30] for monolayer adsorption on a physically homogeneous surface without molecule interaction:

$$q = \frac{Q_{\max}}{1+bc_e} \quad (5)$$

Where $Q_{\max}$ is the maximum adsorption capacity, b is an adsorption energy constant, and C_e represents dye solution equilibrium.

The empirical Freundlich isotherm model (Equation 6) [31] describes interactions between adsorbed molecules on heterogeneous surfaces with multilayer adsorption:

$$q = K_F c_e^{1/n} \quad (6)$$

Where K_F is the adsorption capacity and n is a heterogeneity-dependent empirical formula. Fig. 5 shows Polymath's trial-and-error fitting of Equations 5 and 6. With its higher regression coefficients and lower

MPSD error value, the Freundlich isotherm more closely represents the experimental data (Table 2). The Langmuir model demonstrated the $Q_{\max}$ of 9.72 mg g⁻¹.

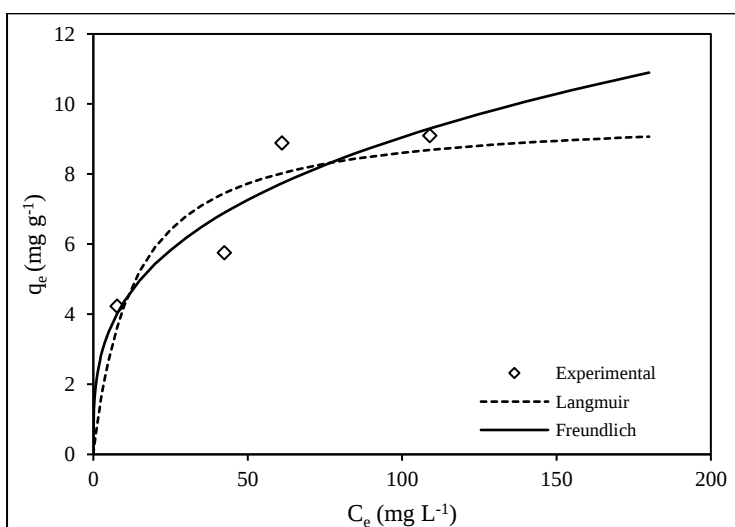


Figure 5. Adsorption isotherms by the nonlinear method for the adsorption of RO16 onto SMGC

Table 2. Freundlich and Langmuir isotherm constants and error analysis for RO16 adsorption

Isotherm Models Constant				Error Analysis			
Langmuir		Freundlich		Langmuir		Freundlich	
$Q_{\max}$ ($\text{mg}\cdot\text{g}^{-1}$)	b ($\text{L}\cdot\text{mg}^{-1}$)	K_F	n	R^2	MPSD	R^2	MPSD
9.72	0.08	2.11	3.16	0.92	24.40	0.95	17.25

Influence of pH

This study investigated the effect of pH on RO16 adsorption by varying the initial pH of the RO16 solution from 3 to 9. It was discovered that pH significantly impacts RO16 adsorption onto SGMC (Figure 6). The amount of RO16 removed decreased as the initial pH of the solution increased. At initial pH levels of 3, 5, 7, and 9, RO16 adsorption was 3.33, 3.25, 3.13, and 2.86 mg g^{-1} , equivalent to 100%, 97.63%,

93.84%, and 85.91% removal, respectively. Due to the negatively charged RO16 molecules' electrostatic repulsion with the adsorbent surface, RO16 removal was lower at pH 7 and 9. This was consistent with our data on pHPZC, which demonstrated that the SMGC became negatively charged when the pH of the dye solution exceeded 5.50, making it unfavourable for anionic contaminants such as RO16.

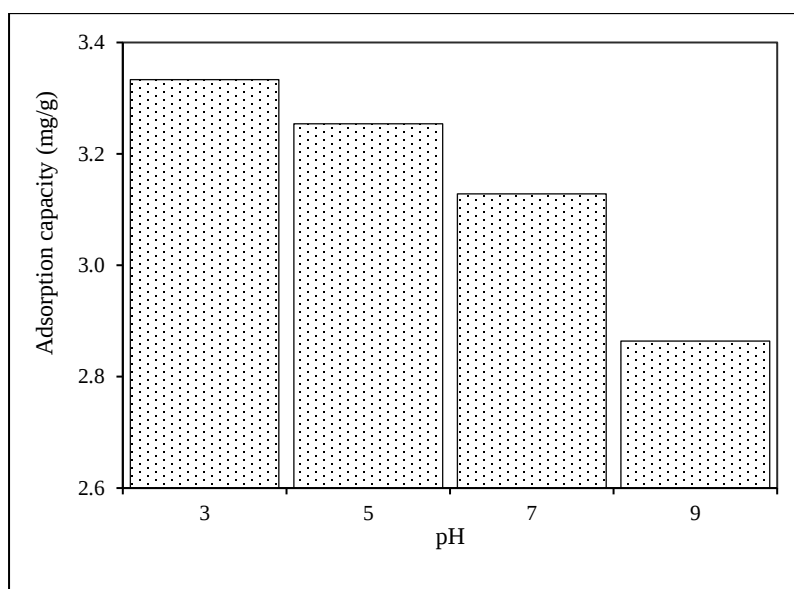


Figure 6. Effect of pH on the adsorption of RO16 onto SGMC

Influence of adsorbent dosage

As shown in Figure 7, as the amount of adsorbent used increased from 5 to 10.0 g L^{-1} , the RO16 dye adsorption capacity increased from 2.63 to 5.75 mg g^{-1} , equivalent to 13.17%-57.54% removal at equilibrium. The adsorption capacity for SGMC declined above 10.0 g L^{-1} to 1.99 mg g^{-1} , although at this point, the maximum removal of 100% was observed. The declining

adsorption capacity at adsorbent dosages greater than 10 g L^{-1} is mainly due to the unsaturation of adsorption sites through adsorption. This is in line with the findings reported by Suhaimi et al. [32] in their study, which asserted that the overcrowding of the adsorbent sites was responsible for the decreasing removal capacity of methylene blue using bamboo-derived biochar.

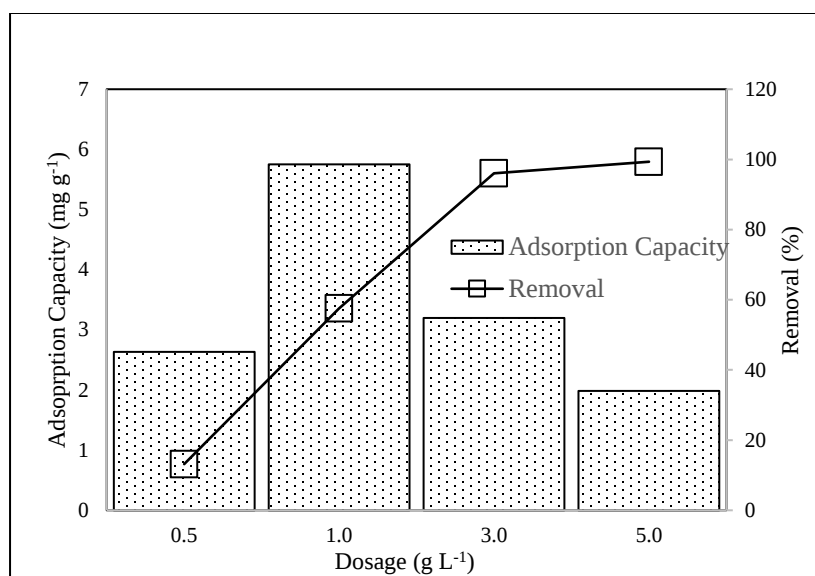


Figure 7. Effect of dosage on the adsorption of RO16 onto SGMC

Dye desorption

Figure 8 shows RO16 dye desorption from SMGC-loaded dye at different pH levels. As the pH increased, more dyes were desorbed. RO16 desorbed was 0%, 0.14%, 29.24%, and 68.38% at pH 3, 5, 8, and 11, respectively. As solution pH rises, the adsorbent surface

net positive charge decreases. The electrostatic attraction of negatively charged dye particles to the more negatively charged SGMC surface indirectly aided desorption. This observation is consistent with research on reactive dye desorption using cationic polymer/bentonite by Li et al. [33].

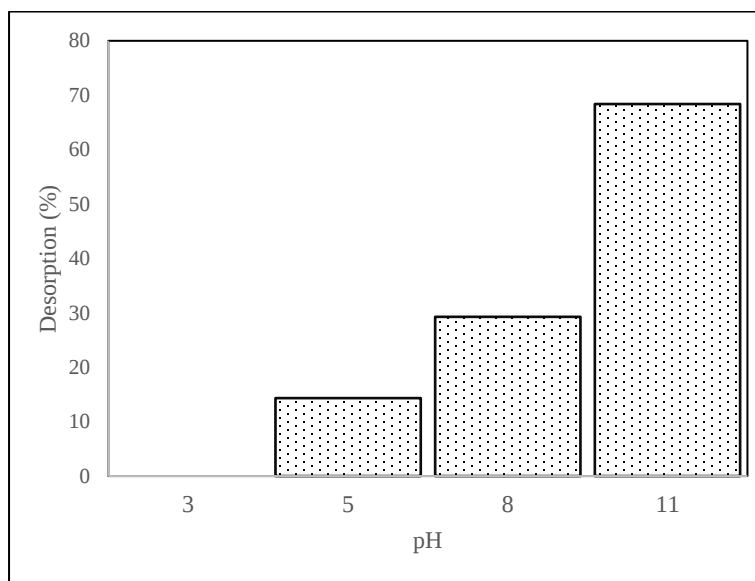


Figure 8. Desorption of RO16 from the dye-loaded SGMC

Conclusion

The results indicate that modifying the surface of grated coconut with a surfactant produced a promising reactive

dye adsorbent for aqueous solutions. The pHPZC studies revealed that the SGMC had a net zero surface charge at a solution pH of 5.5, whereas the surface area

was relatively small at $1.86 \text{ m}^2 \text{ g}^{-1}$. In the batch study, adsorption depended on the pH of the dye solution and the amount of adsorbent. The percentage of dye removed was greater in acidic conditions, with pH 3 showing the highest removal, whereas the adsorbent dosage of 10 g L^{-1} provided the highest adsorption capacity. With a maximum adsorption capacity of 9.72 mg g^{-1} , the Langmuir isotherm correlated best with the equilibrium adsorption data. The desorption of dye-loaded SMGC increased with increasing water pH and vice versa. Due to its abundance, ease of modification, and low cost, the application of SMGC proved to be an effective substitute treatment method for dye wastewater.

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