

SURFACTANT MODIFICATION OF DURIAN PEELS FOR REMOVAL REMAZOL BRILLIANT ORANGE 3R (RBO3R) DYE FROM AQUEOUS SOLUTIONS: A COLUMN STUDIES

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ABSTRACT

The feasibility of durian peel as a non-conventional low-cost adsorbent for the removal of Remazol Brilliant Orange 3R (RBO3R) was investigated. In this study, durian peel had been chemically modified by cationic surfactant cetylpyridinium chloride monohydrate (CPC) for the removal of RBO3R in a continuous adsorption process on a fixed-bed column. According to the results, the SMDP was found to be an efficient media for the removal of dye from wastewater. Bulk density and pH all seem to play important roles in the adsorption of RBO3R. SEM micrograph showed raw durian peel (RDP) have elongated fiber line with small pores while surfactant modified durian peel (SMDP) showed crater-like-holes with large pore space that increase active binding site on SMDP surface and improve adsorption processes. For FTIR analysis, a successful impregnation of CPC onto the surface of durian peel was presented at peak of 2853 cm⁻¹ on SMDP showed C–H vibration of carbonyl group bands originated from CPC. At peak 3369 cm⁻¹ on SMDP indicate the O–H group in cellulose. All important functional group such as carbonyl and hydroxyl on durian peel surface improve its performance as adsorbent in binding and sequestering RBO3R dyes from aqueous solution. In column studies, the breakthrough time (tb) depend strongly on column parameters which was initial dye concentration, column bed height and flow rate. The breakthrough time (tb) and dye removal efficiency was proportional to column bed height and inversely proportional to initial dye concentration and flow rate. This study suggests the potential application of durian peel as adsorbents as an alternative to activated carbon can overcome biomass waste problem and sequester dye from colouring wastewater.

Keywords: Durian peel, Remazol Brilliant Orange 3R, Cationic Surfactant, Fixed Bed Column, cetylpyridinium chloride, Adsorption

1. INTRODUCTION

The most significant cottage textile industry in Malaysia is batik industry, especially in Kelantan and Terengganu state. Operation at a backyard or small cottage, this batik textile industry is a well-known industry that using a huge amount of water during and after processing and produces a tremendous amount of wastewater containing toxic dyes. Dyes are generally used in batik industry as a colouring substance in dyeing cotton fabrics. Types of dyes used in batik industry mostly are anionic reactive azo dye containing azo bonds (nitrogen-nitrogen double bonds -N=N-) that responsible for bright colours [1]The presence of dyes in the wastewater will cause severe environmental pollution, especially to the aquatic environment due to its highly water-soluble and recalcitrant in nature. The dyes also affect water transparency, visibility, aesthetics, and solubility of gases if present in water even in low concentration (less than 1 ppm) [2] [3]. The coloured effluents must undergo treatment before releasing to environments due to contain harmful substances that will affect pH, chemical oxygen demand (COD), dissolved oxygen concentration, and biological oxygen demand (BOD) of water [3]. There are several types of conventional treatment in removing various dyes from industry which are physical/physicochemical types (adsorption, ion exchange, filtration, and coagulation flocculation), biological types (aerobic and anaerobic degradation), and chemical/advanced types (ozonation, Fenton

reagent, photocatalytic) [4]. However, the cost of the conventional water treatment is considered high for small and medium batik industry. This constraint lead to most of the batik cottage to discharges the effluents directly into the drains without prior treatment and not comply to regulation [1]. So, it is important to develop non-conventional low-cost adsorbent as an alternative to activated carbon to overcome biomass waste problem and sequester dye from colored wastewater.

Water treatment by adsorption using low-cost adsorbent is a demanding area for water treatment (remove pollutants), waste management and environmental sustainability. One of the alternatives for high costs activated carbon is agricultural waste. Agricultural wastes are low cost, abundance and readily available resources, thus frequently used in the investigation to remove various types of pollutants from aqueous solutions. Agricultural waste contains cellulose, hemicellulose, lignin, lipids, simple carbohydrates, proteins, starch, extracts, hydrocarbons and water. Agricultural waste consists of types of functional groups such as carboxyl, carbonyl, amido, phenolic, alcohol, amino, and esters on the surface that involve in impregnation and adsorption processes. All the chemical properties contained in agricultural wastes determined whether basicity or acidity the agricultural waste is and can be a good potential as an adsorbent to treat various kinds of pollutants [5]. By converting into valuable material can prevent these wastes from creating disposal problem due to their limitation in application [6].

The main components in the dye molecules which are chromophores and auxochromes. Chromophores responsible for producing the color while auxochromes enhance the affinity of the dye toward the fibers [7]. There are three types of dyes which are cationic, anionic and nonionic dyes. Cationic dyes are basic dyes while the anionic dyes include direct, acid and reactive dyes. Dyes are generally used in batik industry as a colouring substance in dyeing cotton fabrics. Usually, 20-50% of dyes get wash away during processing and remain in the wastewater without any treatment. Cationic dyes are widely used in acrylic, wool, nylon and silk dyeing. humans. Adsorption of cationic dyes using agricultural solid wastes showed good adsorption capacities. Anionic dyes including a reactive group and interact with cotton, wool, etc., to form covalent bonds. Anionic reactive azo dyes have high stability towards light, heat, oxidizing agents, and brightness (Othman, 2010). Moreover, the release of reactive dyes into the environment is undesirable due to the hydrolysis of reactive groups in the water phase causes toxic, mutagenic, and carcinogenic to human beings [7].

Conventional technologies for dye contaminated from textile effluents by activated carbon using fixed-bed columns is the most efficient techniques due to flexibility and simplicity of design, lower initial cost, insensitivity to toxic pollutants and ease of operation but with drawbacks due to carbon limits in a large-scale application and high capital involved [9]. Thus, a cost-saving method of removing dye from wastewater by selecting an inexpensive alternative adsorbent derived from natural or waste materials which does not require any expensive pretreatment [9], [10] [11] using wheat straw to remove Congo Red while [12] using tea leaves to remove Reactive Blue 5 and Methyl Orange.

Recently, durian waste and other biomass are commonly used among researchers as potential adsorbents substitute to activated carbon due to lignocellulosic properties of biomass materials that can bind pollutants [13]. Durian (*Durio Zibethinus Murray*) is a tropical fruit and produces much waste especially during durian season. Therefore, utilize durian peel as a potential adsorbent to remove anionic reactive azo dyes is beneficial [14]. However, limitations using this biomass due to the hydrophilic character and negative charge of a functional group on surface unable to bind anionic pollutants. Therefore, some modifications important to improve its performance as adsorbents by using cationic surfactants. These cationic surfactants will transfer the cellulosic character of biomass from hydrophilic to hydrophobic. The dye chosen for this research was Remazol Brilliant Orange 3R (RBO3R) because the dye was well-known and used extensively in batik industry due to capability [7] of binding both synthetic and natural textiles fibres.

The utilization of Durian peel as a potential adsorbent by undergoing modification using cationic surfactant (CPC) to remove anionic reactive azo dyes from aqueous solutions was still new and limited [8]. This study is focusing on preparing the adsorbent from agricultural waste by modifying their surface to improve the effectiveness of the adsorbent. Therefore, in this paper, a study on the physicochemical change of cetylpyridinium chloride monohydrate (CPC) modified durian peel and its applicability for removing model anionic dye, RBO3R was presented. A continuous column experiment was applied to explore the effects of parameters such as the bed height of the adsorbent and inlet dye flow rate on the column breakthrough volume.

2. MATERIALS AND METHODS

Durian peel that was used as raw material throughout the experiment was collected and each locule of durian peel was peeled off to remove the green thorny outer husk and only the thin neck and thick bottom white inner husk has been taken. Then, the durian peel was washed, cut into tiny pieces (1-2 cm) long and then sun-dried for 8 hours and oven-dried again at 65°C for overnight. The dried durian peel was ground, store in an airtight container and labelled as raw durian peel (RDP) for the characterization and adsorption studies. Preparation surfactant modified durian peel was adopted from [15] with slight modification. Cationic surfactant cetylpyridinium chloride monohydrate (CPC, Bio Basic) (C₂₁H₃₈NCl; MW = 357.99). The surfactant modified adsorbent was prepared by soaking 50 g of Durian peel in 1 L of solutions at concentration of 2.5 mmol L⁻¹ CPC and mixed using propeller mixer at 500 rpm. The treated durian peel was separated, rinsed with distilled water several times, oven-dried at 65°C overnight, stored in an airtight plastic container and was labelled as surfactant modified durian peel (SMDP) for further experiment. Anionic reactive azo dyes for batik textiles dyeing, Remazol Brilliant Orange 3R (RBO3R), was sourced from local textile industries at Kelantan (Nordin Batik & Craft Sdn. Bhd). An anionic reactive azo dye stock solution was prepared by dissolving dye powder in distilled water. Dye concentration was measured using Spectrophotometer (HACH DR2500) at maximum absorbance wavelength (λ_{max}) of 485 nm and calibration curve was plotted. The chemical structure of RBO3R is shown in Figure 1. The characterization of adsorbents RDP and SMDP was involved physically and chemically. Physical characterization involved bulk density, pH and surface morphology using Scanning Electron Microscopy (SEM), Hitachi TM3030 plus Tabletop with voltage accelerates at 5kV and at a magnification of 500x and 1000x whereas chemical characterization for determination of functional groups using Fourier transform infrared (FTIR) (Thermoscientific/Nicolet 6700) spectrophotometer added by attenuated total reflectance (ATR) technique with absorbance spectra were recorded from 650 to 4000 cm⁻¹ under ambient condition.

2.1 General Adsorption Column Studies

Continuous adsorption process in a fixed bed column was performed in a 8.5 cm transparent cylindrical polypropylene column syringe with 1.5 cm inner diameter, packed with a known amount of surfactant modified durian peel (SMDP). A thin layer of plastic net was attached at the bottom of the column to prevent plug flow. The column was packed with a known amount of SMDP to certain bed. Before the influent dye was passed through the column, the column was flushed with distilled water in a down-flow direction to wet the adsorbate entirely and removed the air bubbles. Remazol Brilliant Orange 3R (RBO3R) solution was pumped at selected flow rates using Minipuls 2 Gilson peristaltic pumping upward through the column and percolated through the adsorbent in downflow mode. The column experiment was done at room temperature as shown in Figure 2. The effluent solution was collected at regular time intervals. The concentrations of effluent solutions before and after adsorption were determined using spectrophotometer (HACH DR2500) at maximum absorbance wavelength (λ_{max}) of 485 nm. The effects of experimental column parameters such as bed height, feed flow rate on the dye removal were investigated by varying the one parameter above while at the same time keeping the other parameters constant. For example, to study the effect of influent initial dye concentration, the initial dye concentration was varied at 25 mg/L to 150 mg/L while flow rate and bed height were kept constant at 3 mL/min and 2 cm respectively. To investigate the effect of column bed height, the column was set at three different heights from 1 cm (0.3 g) to 3 cm (0.9 g) corresponding to weight (g) of durian peel while flow rate and initial concentration was kept constant at 3 mL/min and 100 mg/L respectively. For the effect of flow rate of the influent, the influent flow rate was adjusted at 1 mL/min to 5 mL/min while the column bed height and initial concentration were kept constant at the height of 2 cm (0.6g) and 100 mg/L respectively. As a control experiment, raw durian peel (RDP) also been studied by filled in the column with RDP at 2 cm column bed height corresponding to 0.6 g weight of RDP while flow rate and initial dye concentration were kept constant at 3mL/min and 100 mg/L respectively. Dye removal was measured by using the equation accordingly:

$$\text{Dye removal (\%)} = \frac{C_o - C_t}{C_o} \times 100$$

Where C_o was the concentration of dye influent while C_t was the concentration of dye effluent at time (t) as suggested by [16].

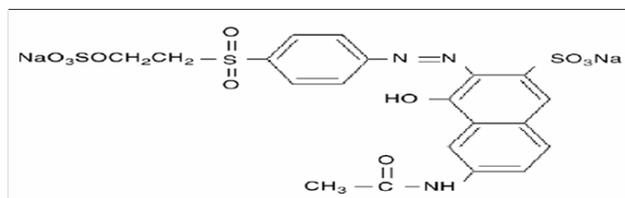


Figure 1: Chemical structure of RBO3R

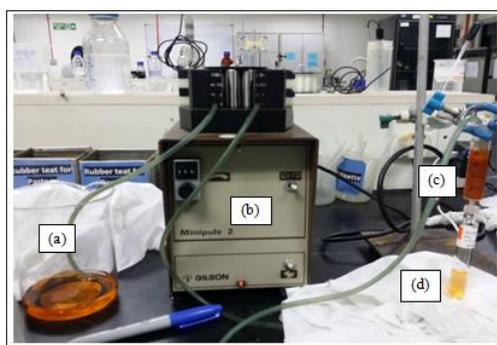


Figure 2: Column experiment RBO3R dye (b) peristaltic pump (c) column syringe (d) collection vial

3. RESULTS AND DISCUSSION

Physical characterization of pH for RDP and SMDP showed that SMDP more acidic than RDP with 4.71 and 5.19 respectively. Impregnation of CPC on the surface of SMDP contain sulfonic and carboxylic acid functional group which are strongest acids contribute to high acidity in SMDP. The carboxylic acid, phosphoric and sulfonic functional groups are the strongest acids while the others were weaker acids as suggested by [17]. This is an agreement with [18] where they found the inclusion of the acid functional group (carboxyl, phenolic) in the carbon structure during the preparation of activated carbon using acid processes causes the acidic property of activated carbon. For bulk density, SMDP show higher bulk density than RDP with 0.1912 g/mL and 0.1892 g/mL respectively. The results probably due to some of the contaminants was washed out during the treatment processes making SMDP more dense than RDP.

3.1 Surface morphology of durian Peel

Scanning Electron Microscope (SEM) has been used to characterize the surface morphology of adsorbent (SMDP) before and after surfactant modification using cationic surfactant (CPC) by using Hitachi TM3030 plus Tabletop with voltage accelerate at 5kV and at a magnification of 500x and 1000x. The difference in the porosity and surface structure was identified between RDP and SMDP. Based on Figure 3, surfactant modified durian peel (SMDP) presents more highly rough, uneven surface structure with crater-like-holes or cavities while raw durian peel showed elongated fibre line. This observation was similar with the study by [19] using durian peel to remove zinc stated that durian peel showed cavities of irregular surface and microstructures and availability of some pores on the surface of durian peel was observed especially after HCl modification. A study done by [7] to scavenge malachite green dye from aqueous solution using durian peel based activated carbon (DPAC) stated that the surface of DPAC was observed to be highly porous with cavities scattered irregularly. Other similar findings were observed by [5], on durian peel treated with sulfuric acid to remove Bisphenol stated that the raw durian peel has no pores. Then after treatment with sulfuric acid, more pore growth on the surface of durian peel responsible in adsorption processes, because the number of available binding sites increased.

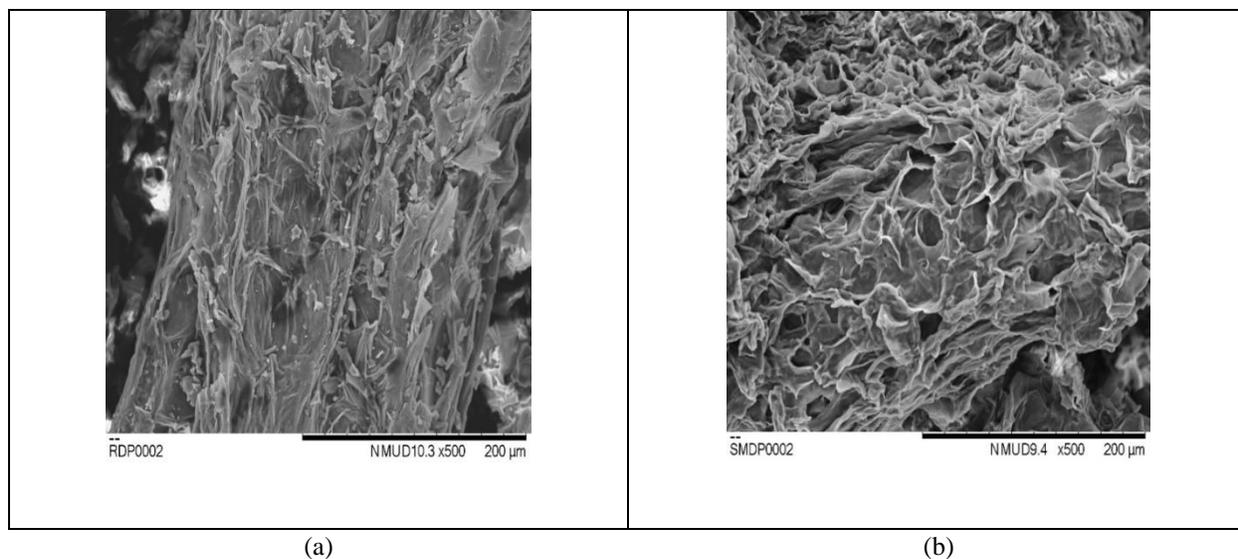


Figure 3: SEM micrograph of (a) RDP at 500x magnification (b) SMDP at 500x magnification

3.2 Surface groups of Durian Peel

Fourier transform infrared (FTIR) spectroscopy has been used to measure the surface structure of Durian peel and its functional group after chemically modification with CPC cationic surfactant. Based on Figure 4, the impregnation of (CPC) on durian peel (existed in SMDP spectra) existed at peak 2853 cm^{-1} for SMDP indicating of C–H vibration of carbonyl group bands of CPC although the peak was absent in raw durian peel (RDP). Moreover, existence of peak 3369 cm^{-1} represents O–H group in adsorbent cellulose may favour for CPC impregnated on the surface of durian peel. Similar findings were found by [20] on characterization on pre-treated durian peel with NaOH and isopropanol to remove methylene blue and crystal violet from aqueous solution showed the broad band near $3500\text{--}3200\text{ cm}^{-1}$ indicate the O–H stretching frequency of OH group that may favour for dye adsorption processes as suggested by [20].

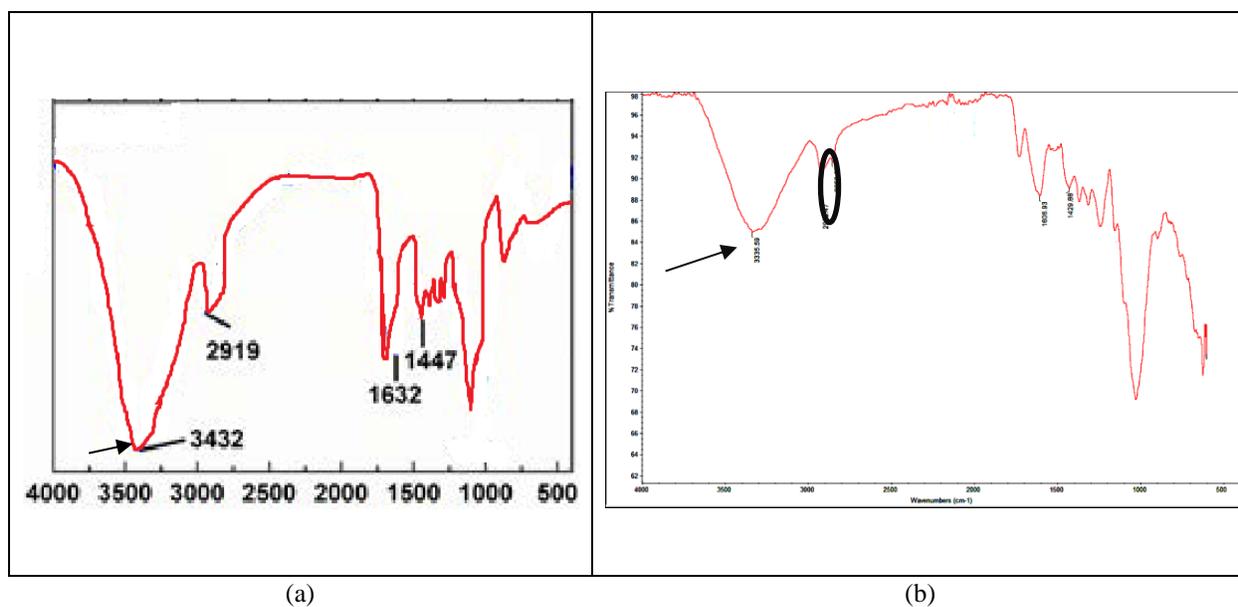


Figure 4: FTIR spectra of (a) RDP and (b) SMDP

3.3 Adsorption of Remazol Brilliant Orange 3R (RBO3R) Dyes in Fixed-Bed Column using RDP and SMDP (A Column Studies)

The effect of initial dye concentration of (Remazol Orange 3R) was studied by conducting the experiment at 25 mg/L, 100 mg/L and 150 mg/L in column that contain adsorbent of surfactant modified durian peel with constant flow rate (3mL/min) and bed height (2 cm). It can be seen that increase in influent initial concentration from 25 mg/L to 150 mg/L causes decrease in breakthrough time (t_b) from 58.05 min to 14.85 min. The dye removal increases from 79% to 99% when the initial concentration of dye solution decreases from 150 mg/L to 25 mg/L. This trend can be explained by higher concentration gradient causes adsorbent active site covered more with adsorbate and get saturated earlier [3], [21], [22]. Increased competition for the limited binding sites on the adsorbent was occurred among dye molecules, thus decreased breakthrough time [20]. Therefore, overall, we can conclude that diffusion is concentration dependent as suggested by [21]. The effect of bed height on adsorption was studied by varying the bed height of SMDP at 1 cm, 2 cm and 3 cm in syringe column at a constant flow rate of 3 mL/min and constant initial (RBO3R) at 100 mg/L. It can be seen that increase in bed height from 1 cm to 3 cm causes increased in breakthrough time (t_b) from 10.8 min to 67.5 min. The dye removal increases from 69% to 93% when the column bed height increase from 1 cm to 3 cm. It can be explained that as the bed height increased, dye solution had more time to contact with the adsorbent due to increase in adsorption binding sites and surface area thus slower the column saturation lead as suggested by [21]. Moreover, the residence time of adsorbate in adsorbents was increased and a lower concentration of dye in the effluent as suggested by [3], [21]. Therefore, a desirable bed height was the key factor in determining the success of the performance of fixed bed column. The effect of flow rate was studied by adjusted the influent flow rate at 1 mL/min to 5 mL/min while the column bed height and initial concentration were kept constant at a height of 2 cm (0.6 g) and 100 mg/L respectively. It can be seen that that increase in flow rate from 1 mL/min to 5mL/min causes decrease in breakthrough time (t_b) from 220 min to 16 min. The dye removal increases from 86% to 97% when the influent flow rate decreases from 5 mL/min to 1 mL/min. It can be explained that due to high turbulence inside the column causes weaker interaction and lesser intraparticle mass transfer between adsorbate molecule and adsorbent (SMDP) as suggested by [21]. Moreover, increased in the flow rate also causes breakthrough time (t_b) to reach saturation was decreased due to short residence and insufficient time inside the column and subsequent decreased the diffusion rate between the adsorbate into the pores of the adsorbents (active binding site) causes effluent leave earlier before achieved equilibrium as suggested by [3], [9], [22]. Effect of initial concentration, bed height and flow rate are shown in Table 1.

Table 1: Effect of initial concentration, bed height and flow rate to breakthrough time (t_b) and % dye removal efficiency

Initial concentration	t_b (min)	Dye removal (%)
25 mg/L	58.05	99.8375
100 mg/L	49.95	89.9085
150 mg/L	14.85	79.0700
Bed Height (cm)	t_b (min)	Dye removal (%)
1 cm	10.8	69.3411
2 cm	52.65	89.9085
3 cm	67.5	93.1681
Flow Rate (mL/min)	t_b (min)	Dye removal (%)
1	220	97.9305
3	52.65	89.9085
5	16	86.0983

4. CONCLUSION

In this study, surfactant modified Durian peel (SMDP) using cationic surfactant (CPC) were prepared, characterized by several physicochemical methods, and tested as an effective adsorbent for RBO3R dye removal from aqueous solutions. Surface morphology showed RDP have elongated fibre line with small pores while SMDP showed crater-like-holes with large pore space that increase active binding site on SMDP surface and improve adsorption processes. According to FTIR analysis, a successful impregnation of CPC onto the surface of durian peel was presented at peak 2853 cm^{-1} on surfactant modified durian peel (SMDP) showed C–H vibration of carbonyl group bands that was originated from CPC. At peak 3369 cm^{-1} on SMDP indicate the O–H group in cellulose. This functional group such as carbonyl and hydroxyl on durian peel surface improve its performance as an adsorbent in binding and sequestering RBO3R dyes from aqueous solution. In column studies, the breakthrough time (t_b), exhaustion time (t_{exh}) was proportional to column bed height and inversely proportional to initial dye concentration and flow rate. The experimental results were comparable with other literature of surfactant modification adsorbent. This study suggests the potential application of durian peel as adsorbents as an alternative to activated carbon can overcome biomass waste problem and sequester dye from colouring wastewater. Lastly, the highest breakthrough time for initial concentration of 25 mg/L, bed height at 3cm and flowrate of 1mL/min were 58.05 min, 67.5 min and 220 min, respectively.

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