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Utilization of Rambutan Peel as a Potential Adsorbent for the Adsorption of Malachite Green, Procion Red, and Congo Red Dyes

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ABSTRACT

Bioadsorbent preparation from rambutan peel applied as adsorbent was characterized using FT-IR, SEM-EDS, BET and TG-DTA analysis. FTIR analysis showed the presence of specific cellulose compounds in the rambutan peel bioadsorbent, the rambutan peel bioadsorbent was amorphous, there were wavy and uneven pores in the morphology of the rambutan peel and had the highest elemental content of 74.3%, the surface area of the rambutan peel was 1.22 cm/g. The adsorption process was applied to malachite green, congo red, and procion red dyes with parameters such as pH, kinetics, isotherm and thermodynamics. Based on kinetic parameters, the adsorption process of malachite green, congo red, and procion red using rambutan peel tends to follow the pseudo second order kinetic model. The adsorption capacity achieved was 182.40 mg/g in procion red, 6.24 mg/g in congo red, and 11.73 mg/g in malachite green. The adsorption process takes place spontaneously which is indicated by a negative Gibs free energy value.

Keyword: Rambutan Peel, Malachite Green, Procion Red, Congo Red, Regeneration

INTRODUCTION

Rambutan peel is one of the natural ingredients that has the potential to be used as a bioadsorbent in the adsorption of dyes and metal ions, this is due to the presence of cellulose, hemi cellulose and phenolic compounds contained in it, to be used as a bioadsorbent, of course, rambutan peel must first be removed the water and some make the rambutan peel as activated charcoal (Castro & das Virgens, 2019). Rambutan peel contains cellulose which is quite high, which is 24%. The presence of these chemical compounds makes rambutan peel a potential raw material for the manufacture of biocharcoal which can then be used as an adsorbent in adsorption (Normah et al., 2021). Before the adsorption process is carried out, the material is first characterized with the aim of knowing the physical and chemical properties of the material.

The adsorption method used in the dye absorption process was sought for optimum conditions including time, temperature and concentration. This was done with the aim of looking at the performance of rambutan peel bioadsorbent in various environmental conditions and studying it using kinetic and thermodynamic parameters to determine whether the reaction proceeded chemically or physically. To see the effectiveness of the adsorbent to be used repeatedly in this study, desorption and regeneration processes were carried out on the rambutan skin bioadsorbent against malachite green, congo red, and procion red.

Malachite Green in Figure 1a is a group of cationic synthetic dyes having the molecular formula C₂₃N₂H₂₅Cl. The molecular weight of green malachite dye is 364.91 g/mol and has a melting point of 159°C (Zhao et al., 2018). This dye is completely soluble in water and alcohol, has a stable tendency to strong oxidizing agents, strong acids, sensitive to light and flammable. Azo dyes (Figure 1b) generally have an auxochrome hydroxyamine group and a substituted amino group, are known as Azo dyes because they have an Azo group (N=N) with a general structure of R-N=N-R(Youssef et al., 2016). Reactive dyes are azo and anthraquinone systems with relatively small molecular weights and low absorption of fiber (Benkhaya et al., 2018). Reactive groups are part of the dye that is easily released, with the release of this reactive group, the dye becomes easy to react with the fiber. This dye can be said to be a dye that belongs to the dichlorotriazine group which can dye cellulose fibers.

In research, rambutan peel is used as an adsorbent for dyes. The success of the synthesis of adsorbent materials was characterized using Fourier Transform Infra Red (FTIR), Scanning Electron Microscopy (SEM), BET surface area analysis and Thermogravimetry-Differential Thermal Analysis (TG-DTA). Rambutan peel was used as an adsorbent for malachite green, congo red, and procion red. The factors that determine the success of adsorption studied in this study were the effect of pH, the effect of adsorption time, the effect of concentration, adsorption temperature, desorption and regeneration of the adsorbent.

EXPERIMENT

Chemicals and Instrumentations

The materials used in this study were rambutan rind (*Nephelium lappaceum* L), sodium hydroxide (NaOH), hydrochloric acid (HCl), distilled water (H₂O), malachite green, congo red, and procion red. Material characterization was carried out using XRD device analysis (Rigaku Mini Flex 600), FT-IR spectrophotometer (Shimadzu Prestige-21), BET adsorption-desorption apparatus (ASAP 2020), SEM and UV-Vis spectrophotometer (Biobase BK)-UV1800 PC.

Synthesis of NiAl-LDH

NiAl-LDH was prepared by the coprecipitation method (Lesbani et al., 2020). The molar ratio used for the 3: 1 LDH synthesis from a mixed solution with Ni(NO₃)₂.6H₂O 3 M and Al(NO₃)₃.9H₂O 1 M is added to the beaker. The mixture was stirred for 30 minutes and then slowly dripped with 2M NaOH to pH 10. Then, stirred for 12 hours at a temperature of 60°C. After that, the samples were dried at 100°C in an oven for 12 hours.

Adsorbent Preparation

Samples of rambutan skin (*Nephelium lap-paceum* L) were cut into small pieces and then washed using clean water and then dried in the sun to remove the washing water, then oven dried at 100°C, the rambutan skin was then mashed using a mortar and sieved at a speed of 100 mesh.

Adsorption Kinetic Study

Rambutan peel bioadsorbent (*Nephelium lappaceum* L) as much as 0.05 g was added to a 100 mL erlenmeyer which had been filled with 25 mL of red procion dye solution with a concentration of 50 mg/L, Congo red solution with a



Figure 1. Structures of dyes: Malachite Green (a), Congo Red (b), and Procion Red (c)

concentration of 45 mg/L and malachite solution. green 10 mg/L whose pH value has been adjusted. The adsorption process was carried out by stirring using time variations of 5, 10, 20, 30, 50, 70, 90, 100, 120, 150, 180 and 200 minutes and then separated. The separation process was carried out by centrifugation and the filtrate obtained was measured using a UV-Vis spectrophotometer.

Adsorption Isotherm Study

A total of 0.05 g of rambutan peel (*Nephelium lappaceum* L) was added to a 100 mL erlenmeyer which had been filled with 25 mL of malachite green, congo red, and procion red dye solutions with various concentrations of 20, 40, 50, 60, 70, 80, and 90 mg/L which has been adjusted to the pH value. Then the adsorption process was carried out by stirring according to the optimum time in the previous study with variations in temperature 30, 40, 50 and 60°C. The separation process was carried out by centrifugation and then the absorbance value of the filtrate was measured using a UV-Vis spectrophotometer with a wavelength at the maximum absorbance from previous studies.

Desorpsi Study

Desorption was carried out by first adsorption using 50 ppm for red procion dye, red congo dye and green malachite then added as much as 0.05 g of rambutan skin sample (Nephelium lappaceum L). The separation process is carried out by centrifugation so that the adsorbent is then obtained and continued with the desorption process. The obtained filtrate was measured with a UV-Vis spectrophotometer. The desorption process was carried out using several desorption reagents, namely HCl, NaOH, ethanol, methanol, diethyl ether and hot water with a concentration of 1M each. Each 20 mL of solvent was added 0.05 g of rambutan peel (Nephelium lappaceum L) skin sample which had previously been used for the adsorption process. The mixture was then stirred for 1 hour and then filtered to separate the rambutan skin samples (Nephelium lappaceum L) with red procion and red congo dyes. The obtained filtrate was then measured its absorbance using a UV-Vis spectrophotometer.

Regeneration Study

A total of 0.05 g of adsorbent that had been desorbed and then re-adsorbed using 25 ml of

red procion dye solution with a concentration of 500 mg/L, Congo red 500 mg/L and green malachite 500 mg/L then stirred for 120 minutes. The obtained filtrate was measured absorbance using a UV-Vis spectrophotometer. The same process was carried out up to three times for the adsorbent regeneration process.

RESULTS AND DISCUSSION

The prepared rambutan peel was characterized using an FT-IR spectrophotometer to identify the functional groups contained in the rambutan peel (Figure 2). The FT-IR spectrum of rambutan peel showed a wide vibration at 3425.58 cm⁻¹ which indicated the presence of -OH from the alcohol and carboxylic acid groups. The presence of the C=O carbonyl group was indicated by the appearance of vibrations at 1705.07 cm⁻¹. The wave number at 1620.21 cm⁻¹ with a sharp absorption intensity indicates the presence of aliphatic C=C group vibrations which is strengthened by absorption at a wave number of 756.1 cm⁻¹ for aromatic C-H absorption. The wave number at 1442.75 cm⁻¹ indicates the presence of a symmetrical carboxylate group and the peak vibration at 1211.3 cm⁻¹ is a C-O ether group which as a whole can form lignin and cellulose bonds.

This research characterizes rambutan skin (*Nephelium lappaceum* L) using an FT-IR spectrophotometer which produces a vibration peak at 1716.61 cm⁻¹ which indicates the presence of a non-ionic carbonyl group (C=O) (Zhang et al., 2018). At the peak of 3419.33 cm⁻¹ there is a group (O-H) derived from the alcohol and carboxylic acid groups (Mohadi, Palapa, Taher, et al., 2021).



Figure 2. IR spectrum of Rambutan Peel

The vibration peak at 2926.03 cm⁻¹ indicates an aliphatic C-H group (Cheng et al., 2020). The wave number at 1618.48 cm⁻¹ is a –COO-carboxylate group, 1441.01 cm⁻¹ is a symmetrical carboxylate group and at the peak of 1216.67 cm⁻¹ is a C-O group cut from ether (lignin bonds with cellulose) (Duan et al., 2019; Castro & das Virgens, 2019). The results of the characterization of rambutan peel in this study have the same functional group characteristics in previous studies.

Characteristics of the successful preparation of rambutan peel as an adsorbent was supported by adsorption-desorption analysis of nitrogen which resulted in isotherm patterns, data on surface area, pore volume, and pore diameter. The results of rambutan peel characterization using BET isotherm analysis can be seen in Figure 3.

Based on the graph of the adsorption-desorption of N_2 gas on the rambutan peel above, it belongs to the category IV isotherm where this type tends to be mesoporous adsorbent characteristics which represent monolayer and multilayer adsorption and the presence of capillary condensation (Moller & Pich, 2017). The presence of capillary condensation indicates a loop hysteresis phenomenon. The type IV isotherm is characterized by no overlap between the adsorption and desorption graphs. The results of the BET characterization of rambutan peel showing data on surface area, pore volume and pore diameter which can be observed in Table 1.

Based on Table 1, it can be observed that the rambutan peel has a surface area of $1.220 \text{ m}^2/\text{g}$. Based on the comparison of the surface area data of 5 adsorbents, it can be seen that the rambutan peel bioadsorbent has a larger surface area than



Figure 3. Isoterm adsorption-desorption of Nitrogen on Rambutan Peel

the durian peel and papaya peel bioadsorbent but is smaller than the eggplant and taro leaf bioadsorbent. so that it has a larger surface area.

Rambutan peel was characterized using SEM-EDS analysis equipment which aims to determine the smallest structure of a material including texture, composition, and particle morphology. The results of the characterization of rambutan skin using SEM-EDS analysis are as presented in Figure 4.

Based on the results of SEM analysis with a magnification of 50 times as shown in Figure 4, it is known that the texture of the rambutan skin is uneven and has pores on its surface. The pores on the surface of the adsorbent play a role in the continuity of the adsorption process, where the dye particles will be absorbed into it and fill the voids in the adsorbent so that a reaction occurs between the cell walls of the adsorbent and the adsorbate (Normah et al., 2021). The results of the characterization of the SEM-EDS analysis of the rambutan peel which shows the data on the composition of the content contained in the rambutan peel can be observed in Table 2.

Qualitative and quantitative analysis of the chemical composition contained in a material is carried out using EDS contained in SEM equipment. The chemical composition of the rambutan peel bioadsorbent can be seen in Table 2.

Based on the results of SEM-EDS characterization of rambutan peel bioadsorbent in Table 2, the chemical composition of the most abundant chemical substances contained in rambutan peel bioadsorbent are elements C and O. This data is supported by (Monrroy et al., 2020) which states that the cellulose content in rambutan peel is relatively high, at 35%. Cellulose contains an active group -OH where this active group will play a role in the absorption of dye by the rambutan peel bioadsorbent. The dye adsorption method using rambutan peel bioadsorbent occurs due to the interaction of the active groups contained in the rambutan peel bioadsorbent and the active groups in the dye. This is reinforced by research data that has been carried out previously on characterization using an FT-IR spectrophotometer which shows a sharp vibration of the -OH group at a wave number of 3425.58 cm⁻¹ and this group is also one of the constituent groups of cellulose.

Thermogravimetric analysis aims to determine the decomposition process of rambutan peel. This analysis was carried out using a Thermal Analyzer instrument at a program temperature from

Adsorbent		Surface	Pore volume	Pore diameter	
		area (m²/g)	(cc/g)	(nm)	
Rambutar peel	۱	1.220	0.008	3.189	

Table 1. BET analysis of Rambutan Peel

 30° C to 900° C which was fed with N₂ gas so as to produce a thermogram pattern as presented in Figure 5.

Thermogravimetric analysis of rambutan skin showed that there were two main points of decomposition, namely at temperatures of 100°C and 430°C. The endothermic peak occurred at a temperature of 100°C indicating the loss of water molecules on the surface of the rambutan peel caused by heating but still not completely (Castro & das Virgens, 2019). The peak at 430°C indicates an exothermic process where the rambutan peel sample undergoes an oxidation process because it releases thermal energy and a horizontal line above 500°C indicates that the rambutan peel has undergone a complete decomposition process (Meili et al., 2019).

Based on Figure 6, the optimum condition was obtained at the 90 minutes which was marked by a constant state after that. This condition indicates that the adsorption process by the adsorbent on the adsorbate has reached the saturation point. The amount of dye adsorbed is directly proportional to the concentration of adsorbate where the greater the concentration of adsorbate used in the adsorption process, the greater the amount of dye that will be adsorbed. Based on the data obtained in Table 3 shows the adsorption of green malachite dye solution using rambutan peel bioadsorbent in the pseudo second order kinetic model has a calculated Q_p value that tends to approach the

Table 2. Chemical composition of Rambutan Peel

Adaarbaat	Element (%)						
Ausorbeni	0	Ν	S	Al	Р	Si	
Rambutan peel	94.1	3.4	0.5	0.4	0.3	1.2	

experimental Q_e value. The R^2 value of the pseudo second order kinetic model is also greater than the pseudo first order kinetic model with an average value of 0.99. These data concluded that the adsorption of green malachite dye is more suitable to use the pseudo second order kinetic model.

The data presented in Figure 6 shows that there is an increase in the concentration of the adsorbed dye from 0 to 90 minutes, this indicates that the adsorbent has adsorbed the adsorbate due to the collision between the adsorbent and the adsorbate. The optimum time was then obtained at 90 minutes which was marked by a constant condition, where this condition occurred because the adsorbent was completely filled with adsorbate so that the adsorbent had reached the saturation point (Taher et al., 2020). The concentration of adsorbate can also affect the adsorption process as presented in Figure 6 shows that the greater the concentration of adsorbate, the amount of adsorbed substance also increases with time.

The data from the research on the effect of the adsorption time of Congo red dye using the rambutan peel bioadsorbent that has been obtained are used to determine the reaction rate using pseudo first order and pseudo second order kinetic equations (Juleanti et al., 2021). Calculation of the magnitude of the kinetic parameters using pseudo first order and second order equations are presented in Table 3. Observational data that have been obtained from variations in contact time between the adsorbent and the



Figure 4. Morphology and composition of Rambutan Peel



Figure 5. TG-DTA profile of Rambutan Peel



Figure 6. Kinetic adsorption of MG, CR, and PR on Rambutan Peel

adsorbate are then used to calculate and determine the adsorption rate constant using pseudo first order and pseudo second order kinetic equations. The kinetic parameters using pseudo first order and pseudo second order are presented in Table 3. The data in Table 3 shows the linear regretion of the pseudo first order and pseudo second order kinetic equations. The correlation coefficient value in the first order pseudo kinetic model which has a lower R² value, and supported by k₂ data which has a smaller value than k₁. From the data obtained, it shows that the red procion adsorption process is more suitable to use the pseudo second order kinetic model.

The data on the effect of concentration and temperature on the adsorption of red procion dye using rambutan peel bioadsorbent which has been presented in Figure 7 shows that between temperature and adsorbate concentration is directly proportional where the greater the temperature and concentration of adsorbate, the greater the amount of dye or adsorbate that is adsorbed (Eltaweil et al., 2020). This is due to the interaction between the adsorbent and the adsorbate as the temperature and concentration of the adsorbate increase, the adsorption rate will increase so that the ability of the adsorbent to absorb the adsorbate can increase.

Based on the data in Figure 7, it can be stated that the increase in temperature and concentration in the Congo Red dye adsorption process using rambutan peel bioadsorbent can cause an increase in the concentration of the dye adsorbed on the adsorbate. This is because when there is an increase in temperature, the dye molecules will occupy the active site on the surface of the bioadsorbent so that the adsorbate will interact with the surface of the adsorbent (Mohadi et al., 2021).

The adsorption isotherm parameter was obtained using the Langmuir and Freundlich equations by processing the data on the effect of concentration and adsorption temperature on malachite green, congo red, and procion red dyes using rambutan peel bioadsorbent. Langmuir isotherm model illustrates that the adsorption process occurs in a monolayer, while the Freundlich adsorption isotherm explains that the adsorption process takes place in a multilayer manner (Siregar et al., 2021).

Based on the data presented in Table 4, it is shown that the Langmuir adsorption isotherm equation model is better used in explaining the adsorption process of red procion dye using rambutan peel bioadsorbent. This is shown from the linear regression coefficient value in the Langmuir adsorption isotherm equation which is closer to the value 1. The Langmuir adsorption isotherm equation shows that the adsorption process of red procion dye using rambutan peel takes place in a monolayer. The multilayer adsorption process occurs when the dye adsorption using rambutan peel bioadsorbent matches the Freundlich isotherm equation (Wijaya et al., 2021).

Meanwhile, in thermodynamic observations, the entropy value on the effect of temperature

Table 3. Pseudo first-order and second-order of adsorption MG, CR, and PR on Rambutan Peel

Durin	0.	PFO			PSO		
Dyes	Qe _{exp}	Qe _{calc}	k ₁	R ²	Qe _{calc}	k ₂	R^2
MG	1.112	0.567	0.011	0.353	1.22	0.04	0.993
CR	19.887	12.499	-0.02	0.845	22.123	0.002	0.996
PR	19.5	13.3335	0.022	0.838	21.052	0.002	0.993



Figure 7. Effect of initial concentration and temperature adsorption of MG (a), CR (b), and PR (c) on Rambutan Peel

and adsorption concentration of green malachite dye is inversely proportional, where the higher the temperature and concentration result in a decrease in the entropy value as shown in Table 5. This shows that at high concentrations the degree of regularity of the process occurs. adsorption will be more regular. Based on observations through the Gibbs free value, it can be seen that the adsorption takes place spontaneously which is indicated by a negative value in the data (ΔG) as shown in Table 5. Based on the data presented in Table 5, it can be seen that the adsorption of malachite green, congo red, and procion red dyes using the rambutan peel bioadsorbent as a whole proceeded spontaneously. This has been proven from the negative Gibbs free energy value caused by an increase in the kinetic energy of the molecules, which results in faster movement of dye molecules so that the adsorbate molecules occupy the active side of the adsorbent surface more spontaneously (Ebelgi et al., 2020).

The value of enthalpy on thermodynamic parameters indicates the type of reaction that takes

place (Mohadi et al., 2021). In the adsorption of malachite green, congo red, and procion red dyes using rambutan peel bioadsorbent, it was found that the reaction was endothermic, namely the adsorption process was accompanied by absorption of energy in the form of heat from the environment to the system. This is shown from the results of the calculation of the positive enthalpy value. The increase in adsorption capacity on the adsorption of malachite green, congo red, and procion red dyes was influenced by the increase in temperature during the adsorption process. In the adsorption process of malachite green, congo red, and procion red dyes, the results of the entropy value decreased as the concentration increased, indicating that the adsorption process of the three dyes as a whole had high regularity at large concentrations. adsorption. From the observational data in Table 5, it shows that increasing the concentration does not cause a significant change in the entropy value and at high concentrations it will cause the adsorption process to take place more regularly.

		Model isothermal adsorption						
Zat Warna	Temperature (°C)	Langmuir			Freundlich			
		Qm	KL	R²	n	KF	R²	
	30	6.242	0.057	0.986	6.242	5.963	0.949	
CP	40	1.013	0.082	0.649	2.666	1.128	0.841	
CK	50	1.183	0.079	0.821	0.626	6.76	0.84	
	60	2.295	0.116	0.966	0.164	5.494	0.927	
	30	29.806	0.073	0.713	0.781	2.042	0.913	
MC	40	3.184	0.16	0.776	4.255	1.38	0.87	
MG	50	11.737	0.589	0.992	0.558	6.31	0.998	
	60	1.088	0.242	0.897	0.89	15.849	0.726	
PR	30	145.37	0.497	0.906	1.629	199.526	0.977	
	40	182.4	781.001	0.995	1.679	9.884	0.998	
	50	180.98	1046.28	0.906	1.902	14.634	0.771	
	60	167.54	2510.02	0.999	2.423	30.69	0.997	

Table 4. The isotherm adsorption of MG, CR, and PR

The desorption process is carried out to determine the most effective medium for desorption of dyes so that the adsorbent can be regenerated. Figure 8 shows the results of desorption using several mediums based on polarity and acid-base properties. Based on the data exposed in Figure 8 it can be seen that NaOH has the highest percentage of MG, PR, and CR desorption. This is due to the presence of deprotonation in the alkaline solution so that the dye contained on the surface of the adsorbent can be separated from the surface of the adsorbent, resulting in a high absorbance value.

The regeneration process was carried out 3 times and then the absorbance was measured using a UV-Vis spectrophotometer. Figure 9 shows the adsorption percentage data from 3 times the regeneration process. Based on the data exposed in Figure 9, it shows that the regeneration process is carried out up to 3 repetitions where in the third regeneration, the adsorbed concentration has the smallest percentage value compared to the first and second repetitions. This is because when the addition of NaOH causes the mixture to be in an alkaline condition so that the pH condition

Table 5. The Thermodynamic adsorption of MG, CR,and PR

Dyes	T	ΔS	ΔH	ΔG
,	(K)	(J/mol·K)	(KJ/MOI)	(KJ/MOI)
	303			-11.06
MC	313	0.002	2 000	-13.386
IVIG	323	0.003	3.990	-15.712
	333			-18.038
	303		3.45	-6.135
CP	313	0.065		-6.781
CR	323	0.005		-7.428
	333			-8.074
PR	303			-0.491
	313	0.015	4 05 9	-0.641
	323	0.015	4.000	-0.792
	333			-0.942

exceeds its optimum state, the adsorption of red procion dye decreases.

Based on the data obtained from Figure 9 that the percentage of adsorption in the regeneration process of red congo dye by rambutan peel bioadsorbent decreases with each repetition. This is because in the desorption process using NaOH solvent, OH- ions are added to the surface of the rambutan peel bioadsorbent which causes less adsorbent to adsorb Congo red dye (Yang et al., 2020). The adsorption percentage was obtained for three repetitions in a row, namely 47.45%, 19.98% and 6.99%. Based on the data exposed in Figure 9, it shows that the regeneration process is carried out up to 3 cycle where in the third regeneration, the adsorbed concentration has the smallest percentage value compared to the first and second repetitions. This is because when the addition of NaOH causes the mixture to be in an alkaline condition so that the pH condition exceeds its optimum state, the adsorption of red procion dye decreases (Puchana-Rosero et al., 2016).

Figure 9 shows that regeneration in the first iteration has a higher adsorption percentage. This is because the surface of the adsorbent in the first regeneration has not yet experienced its saturation point so that absorption is still ongoing and has a large percentage value, while in the next regeneration stage, the surface of the adsorbent has begun to experience a saturated condition coupled with the addition of NaOH in the desorption process resulting in more surface the adsorbent is filled with OH- so that there is competition between the green malachite dye ions and OH- ions from NaOH (Daud et al., 2019). Based on the data in Figures 9 it can be concluded that the repetition of the regeneration process causes the percentage of the adsorbed substance to decrease. This is because the active groups contained in the rambutan skin structure have experienced instability in the regeneration process.



Figure 8. Desorption of MG (a), CR (b), and PR (c) after adsorption on Rambutan Peel



Figure 9. Adsorbent regeneration study of MG (a), CR (b), and PR (c)

CONCLUSIONS

The results of FT-IR characterization showed the presence of a specific functional group of cellulose compounds where this compound is one of the compounds contained in rambutan peel. The XRD results of rambutan peel showed that the bioadsorbent produced was amorphous. The adsorption ability of rambutan peel on malachite green, procion red and congo red showed significant results on the adsorption of procion red. the adsorption capacity of procion red reached the highest value of 182.40 mg/g.

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