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Orange Peel Biochar As An Adsorbent For Removal Of Reactive Orange 16 (RO16)

**Maizatul Ashikin Binti Mustapha
J20A0469**

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degree of Bachelor of Applied Science (Bioindustrial
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UMK**

2023

DECLARATION

I declare that this thesis entitled “Orange Peel Biochar As An Adsorbent For Removal Of Reactive Orange 16 (RO16)” is the results of my own research except as cited in the references.

Signature : ASHIKIN

Student's Name : MAIZATUL ASHIKIN BINTI MUSTAPHA

Date : 22/2/2024

Verified by:

Signature : _____

Supervisor's Name : DR. ROSMAWANI BINTI MOHAMMAD

Stamp : _____

Date : _____

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Abstract Orange Peel Biochar As An Adsorbent For Removal Of Reactive Orange

16 (RO16)

ABSTRACT

This study aims to investigate the effectiveness of orange peel biochar as an adsorbent for the removal of Reactive Orange 16 (RO16) dye from an aqueous solution. The plentiful waste orange peels have the potential to be an inexpensive adsorbent for the removal of Reactive Orange 16 (RO16) dye. Adsorption is a popular method for removing dyes from aqueous solution because it is efficient, simple, and cost-effective. Numerous parameters, including the effect of carbonisation temperature, adsorbent dose, initial dye concentration, and contact time, have been investigated. The findings indicated that 700 °C for carbonisation, 1.2 g of adsorbent, 100 mg/L of initial dye concentration, and an eight-hour contact period were the ideal values. With orange peel biochar, the greatest percentage of Reactive Orange 16 dye removal was 97.48 %. The regression analysis of the adsorption results using the linear models revealed that a Langmuir model better approximated the adsorption.

Keywords: Adsorbent, Orange Peel Biochar, Reactive Orange 16, Adsorption

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Abstrak Arang Bio Kulit Oren Sebagai Penjerap Untuk Penyingkiran Reaktif

Oren 16 (RO16)

ABSTRAK

Kajian ini bertujuan untuk mengkaji keberkesanan arang bio kulit oren sebagai penjerap untuk penyingkiran pewarna Reaktif Oren 16 (RO16) daripada larutan cecair. Kulit oren sisa yang banyak mempunyai potensi untuk menjadi penjerap yang murah untuk menghilangkan pewarna Reaktif Oren 16 (RO16). Penjerapan ialah kaedah popular untuk mengeluarkan pewarna daripada larutan cecair kerana ia cepat, mudah dan menjimatkan kos. Banyak parameter, termasuk kesan suhu pengkarbonan, dos penjerap, kepekatan pewarna awal, dan masa sentuhan, telah disiasat. Penemuan menunjukkan bahawa 700 °C untuk suhu pengkarbonan, 1.2 g penjerap, 100 mg/L kepekatan awal pewarna, dan tempoh sentuhan lapan jam adalah nilai yang ideal. Dengan biochar kulit oren, peratusan terbesar penyingkiran pewarna Oren Reaktif 16 ialah 97.48%. Analisis regresi keputusan penjerapan menggunakan model linear mendedahkan bahawa model Langmuir lebih baik menghampiri penjerapan.

Kata kunci: Penjerap, Arang Bio Kulit Oren, Reaktif Oren 16, Penjerapan

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LIST OF ABBREVIATIONS

C	Carbon	14
O	Oxygen	14
H	Hydrogen	14
N	Nitrogen	14
S	Sulphur	14

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LIST OF SYMBOLS

$^{\circ}\text{C}$	Degree Celsius	7
θ	Theta	10
mL	Millilitre	14
mm	Millimetre	14
μm	Micrometre	14
μl	Microlitre	14
cm	Centimetre	14
g	Gram	15
L	Litre	18
C_e	Equilibrium dye concentration in the solution	18
C_o	Initial dye concentration in the solution	18
q_e	Quantity of dye uptake	18
q_{max}	Maximum adsorption capacity	18
K_L	Langmuir constant	18
K_f	Freundlich constant	19
%	Percent	24

CHAPTER 1

INTRODUCTION

1.1 Background of Study

The significance of dyes in various industries lies in their ability to provide a wide range of colours that can be created to meet specific requirements, thereby facilitating the production of different and attractive items. Furthermore, dyes can be manufactured to have desired attributes like stability, lightfastness, and washability, ensuring that the colour remains brilliant and durable over time (Li et al., 2022).

Dyes are used in a number of industries, including textiles, printing, cosmetics, food, and medicines. In these industries, the fundamental function of dyes is to provide colour to items, resulting in a more appealing look and aesthetic appeal (Konwar, 2020). Dyes are organic or inorganic (synthetic) chemicals that may be dissolved or dispersed in liquids, allowing them to be used in a variety of applications. In the textile industry, for instance, dyes are used to colour fabrics, yarns, and fibres, enabling manufacturers to create an extensive variety of colourful and interesting textile products. Dyes are used in inks in the printing industry to produce colourful prints in publications, packaging, and other printed products. Dyes are also used in the cosmetics industry to add colour to makeup goods such as lipsticks, eyeshadows, and nail polishes, allowing for the development of a diverse spectrum of cosmetic colours (Li et al., 2022).

The uses of dyes can have several kinds of consequences for water, including potential environmental and ecological consequences. When dyes are dumped into wastewater streams without being properly treated, they can contaminate aquatic bodies and create water pollution. Many dyes are poisonous or carcinogenic, and their release into bodies of water can kill aquatic life, alter natural ecological balance, and deteriorate water quality (Castillo et al., 2023).

One of the methods that can be used to remove the dyes in the wastewater is adsorption. Adsorption is a mass transfer process in which one or more substances

(adsorbate) from a gaseous or liquid stream are transferred selectively to the surface of a porous material (adsorbent). This means the adsorption is a process of adsorbate attach on the surface of adsorbent. In this study, the adsorbent used is orange peel biochar and the adsorbate is Reactive Orange 16 (RO16) dye. The utilisation of orange peel biochar presents a range of potential applications. Biochar made from orange peels can be used as an adsorbent for the removal of dyes and also removal of heavy metals from wastewater (Liu et al., 2022).

1.2 Problem Statement

Orange peel biochar has a wide surface area and a very porous structure. There is enough of surface area in this arrangement for the adsorption of dye molecules. Specifically, orange peel biochar may have a distinct pore structure that improves its adsorption ability (Yang et al., 2020). Meanwhile, the raw orange peel is less efficient for removal of dye because of lower porosity and a comparatively small surface area. Synthetic dyes such as Reactive Orange 16 dye typically have more colour intensity because containing intricate chemical structures with several functional groups, including azo groups and also have more purity than natural dyes. This property allows for more accurate measurement and analysis of dye adsorption, as even minor variations in dye concentration are easier to detect with synthetic dyes.

1.3 Objectives

The objectives of this study were:

- 1) To investigate the potential of orange peel biochar as an adsorbent based on the percentage removal of Reactive Orange 16.
- 2) To characterize the chemical and physical properties of orange peel biochar as an adsorbent using Fourier-transform infrared spectroscopy (FTIR) and Brunauer Emmett Teller (BET).
- 3) To determine the optimum parameters and an adsorption isotherm model for removal of Reactive Orange 16 using orange peel biochar as adsorbent.

1.4 Scope of Study

In this study, orange peel biochar was used as an adsorbent to remove the Reactive Orange 16 dye. The orange peel was obtained from Kuala Klawang, Negeri Sembilan. The orange peel was ground to become a powder. Moreover, there were several parameters were studied to determine the potential of orange peel biochar as adsorbent for removing the Reactive Orange 16 such as the initial concentration of dye, the contact time, the amount of adsorbent dose, and effect of carbonisation temperature. The characterisation of orange peel biochar was conducted by using Fourier-transform infrared spectroscopy (FTIR) and Brunauer Emmett Teller (BET). The adsorption isotherm models used were Langmuir and Freundlich model.

1.5 Significances of Study

The removal of synthetic dyes from contaminated water is significant for several reasons, including protecting human health because the synthetic dyes can pose a significant risk to human health if ingested through contaminated water or food. Removing these dyes from water sources can reduce the risk of exposure and prevent health problems. Furthermore, it for environmental protection. The technique used was adsorption (Zhang et al., 2022). The use of orange peel biochar as an adsorbent makes it a cost-effective alternative compared to other adsorbents. Orange peel biochar has a high surface area, which provides a large surface area for adsorption to occur. This makes it a highly efficient adsorbent, as it can effectively capture a wide range of contaminants from water.

CHAPTER 2

LITERATURE REVIEW

2.1 Agricultural Waste Act As An Adsorbent

Agricultural waste is a significant source of solid waste for the environment, and carbonization can transform it into a more stable form of carbon, like biochar. In order to create biochar, organic material must be heated without oxygen. The economic value of agricultural waste in the manufacture of biochar can be significant, depending on the market price of biochar and the cost of production (Li et al., 2022).

Agricultural waste can be used to remove pollutants from wastewater by acting as an adsorbent. Because of its cheap operating costs and high flexibility, the use of agricultural waste as an adsorbent is regarded as one of the most successful solutions for pollutant treatment (Dai et al., 2018). Using a low-cost adsorbent can have social, economic, and environmental benefits. Agricultural waste features loose and porous structures, as well as functional groups such as the carboxyl and hydroxyl groups, which make it a good biological adsorption material. The use of agricultural waste as an adsorbent adheres to the concepts of green chemistry and green environmental practices.

2.2 Orange Peel Act As An Adsorbent



Figure 2.2: Orange peels.

(Source: Mueller, 2017)

Orange peel is an agricultural waste product that is produced in large quantities worldwide. It is a rich source of flavonoids, carotenoids, pectin, and other organic compounds, which have been found to exhibit excellent adsorption properties. Researchers have been concentrating their efforts on discovering adsorbents for the treatment of wastewater that are both inexpensive and kind to the environment. This is because of the growing worry for environmental contamination brought on by a variety of pollutants. Orange peel has been reported to have good potential as an adsorbent for the removal of various pollutants from wastewater (Li et al., 2022).

It has been discovered that orange peel contains functional groups that are responsible for adsorption. These groups include pectin, hydroxyl, carbonyl, carboxyl, and amine groups. Physical alteration increased the surface area available for adsorption and binding of pollutants, whereas chemical treatments increased the number of carboxylic groups, which enhanced both adsorption and binding of contaminants (Michael-Igolima et al., 2023). Table 2.2 showed the orange peel has been used as adsorbent for removal of dyes.

Table 2.2: Orange peel as adsorbent.

Adsorbent	Adsorbate	Adsorption Capacity Mg/g	Reference
Orange Peel	Acid Violet	19.88	(Alver et al., 2020)
Orange Peel	Direct Red 80	21.05	(Sahu et al., 2020)
Orange Peel	Congo Red	22.44	(Kyi et al., 2020)
Orange Peel	Methylene Blue	96.00	(Aswathy et al., 2016)

2.3 Carbonisation

Biochar is a thermochemical substance produced by converting biomass at high temperatures in the absence of oxygen (Brickler et al., 2021). Carbonization involves heating organic material to temperatures ranging from 350 to 700 °C. The organic material is turned into biochar during this process, which is a stable form of carbon that can remain in the soil for hundreds or even thousands of years. The product produced in carbonization is biochar only. Meanwhile, the product produced in pyrolysis are bio-oil, gas, and char (Wang et al., 2019). Those is a difference between the carbonization and pyrolysis process. A carbonization of agriculture waste, particularly food waste, is a potential option for converting trash that would otherwise be disposed of in the natural environment into value-added products that may be used to improve resource efficiency, particularly water efficiency.

2.4 Reactive Orange 16 (RO16) Dye

The term "dye" refers to substances that, when applied in solution from either an aqueous or an organic solvent, can give a substrate colour. Substrates include things like textiles, plastics, and polymers. Fundamentally, chemical concepts are involved in the applications of dyes to materials. Dyes are categorised according to their use and chemical components, and they are made up of a group of atoms called chromophores that give the dye its colour. Various functional groups, including azo, anthraquinone,

methine, nitro, aril-methane, carbonyl, and others, are the foundation of these chromophore-containing centres. In addition, auxochromes are substituents that donate electrons from the chromophores to produce or amplify their colour (Brickler et al., 2021).

A type of dye used in the textile industry is Reactive Orange 16. It is an azo dye, which is notorious for being toxic and bad for both the environment and human health. Reactive Orange 16 dye is water-soluble and hard to separate (Torres et al., 2021). Furthermore, it can readily adsorb onto surfaces and create stable complexes since it has a high affinity for surfaces (Obulapuram et al., 2021). In this research, Reactive Orange 16 will be selected as an adsorbate. The chemical formula is $C_{20}H_{17}N_3Na_2O_{11}S_3$.



Figure 2.4: Reactive Orange 16 dye.

(Source: IndiaMart, 2023)

2.5 Adsorption

Adsorption is a common approach for removing dyes from industrial effluents. A molecule is transferred from a fluid bulk to a solid surface, generating an interface layer between two distinct phases. It is a method by which dye molecules in a liquid phase become saturated and bound to the surface of an adsorbent, a solid substance. Natural or artificial substances with a strong affinity for the dye molecules can be used as the adsorbent. The dye molecules adhere to the adsorbent surface due to the attractive

forces between them and their surface, such as van der Waals forces, electrostatic interactions, or chemical bonds (Kong & Zhang, 2022).

2.6 Adsorption Isotherm

The adsorption isotherm is important for understanding how the adsorbent will interact with the adsorbate and for estimating adsorption capacity. Adsorption data is typically described using Langmuir and Freundlich isotherms (Aziz et al., 2018). The Langmuir isotherm model is based on the assumption of a homogenous surface and assumes monolayer adsorption. The Freundlich isotherm model is an empirical equation that assumes a heterogeneous surface and describes multilayer adsorption.

2.6.1 Langmuir

The Langmuir equation describes the adsorption of a solution onto a surface using an isotherm model. Therefore, the Langmuir equation assumes that a solute adsorbs onto a surface via a monolayer and that the adsorption is reversible and homogeneous. Besides, the Langmuir equation can be used to calculate the maximum adsorption capacity of a surface as well as the strength of the adsorbate-surface interaction. However, it is crucial to highlight that the Langmuir equation has certain limitations, including the assumption of a homogenous surface and the inability to account for multilayer adsorption (Zhang et al., 2022). The following equation gives the equation: $\theta = Kc / (1 + Kc)$, where θ is the fraction of the adsorbate-covered surface, c is the adsorbate concentration in the bulk solution, and K is the Langmuir constant, which is proportional to the strength of the adsorbate-surface contact.

2.6.2 Freundlich

The Freundlich equation is an empirical equation that represents the relationship between a solute's concentration in solution and the amount of solute adsorbed onto a surface. The Freundlich equation is frequently used to describe solute adsorption on heterogeneous surfaces where adsorption is not limited to a monolayer. The following equation gives the equation: $q = Kc^{(1/n)}$, where q is the quantity of solute adsorbed per

unit mass of adsorbent, c represents the solute concentration in the bulk solution, K represents the Freundlich constant, and n represents the Freundlich exponent.

2.7 Factor Affecting

There are few factors are affecting the adsorption.

2.7.1 Effect Initial Concentration of Dye

The more concentrated the dye solution is at the beginning, the more it can adsorb. At first, the adsorbent may not reach its full potential for adsorption. When there is more dye present, the adsorbent can hold onto more dye molecules until it cannot hold anymore. This is called the saturation point. When there is a higher initial concentration, more dye can be adsorbed until the system reaches a point of equilibrium (Alorabi et al., 2020).

2.7.2 Effect of Contact Time

The amount of time that the contact occurs between substances has a direct impact on how quickly adsorption takes place. When the substance attracts particles and the particles themselves initially touch, the process of particles sticking to the substance often happens quickly. Over time, the rate at which adsorption occurs decreases. This suggests that the concentration of the adsorbate in the solution is going down. Once the adsorption equilibrium is reached, the rate of adsorption gradually decreases and eventually becomes zero. When the contact period is extended, more adsorption can occur until a balance is achieved (Li et al., 2022).

2.7.3 Effect Amount of Adsorbent Dose

The amount of adsorbent used directly affects how much the system can adsorb. As increase the amount of adsorbent used, its ability to adsorb also increases. The reason for this is that when there is a greater amount of adsorbent, there are more places

for the dye molecules to attach themselves. When the adsorbent reaches its limit, it cannot adsorb any more dye molecules. Using more adsorbent can enhance its capacity to adsorb (Li et al., 2022).

2.7.4 Effect of Carbonisation Temperature

Higher carbonisation temperatures increase the adsorption capacity of biochar and increase its surface area and pore volume. Lower carbonisation temperatures, on the other hand, lead to a reduction in surface area and pore volume, which lowers adsorption capacity (Jadhav & Thorat, 2022). The temperature affects an adsorbent's ability to absorb Reactive Orange 16 dye by weakening the adsorptive interactions between the active sites on the molecule.

2.8 Characterize of Adsorbent

The chemical and physical properties of orange peel biochar were characterized by using two techniques which are Fourier-transform infrared spectroscopy (FTIR) and Brunauer Emmett Teller (BET).

2.8.1 Fourier-transform infrared spectroscopy (FTIR)

The FTIR technique measures the absorption of infrared radiation by a sample, which can reveal information about the sample's functional groups and chemical bonds.

2.8.2 Brunauer Emmett Teller (BET).

The Brunauer Emmett Teller (BET) is often used to determine the surface area of porous materials.

CHAPTER 3

MATERIALS AND METHODS

3.1 Materials

The material used was orange peels obtained from Kuala Klawang, Negeri Sembilan. The orange peel has undergone a carbonisation process to produce biochar. The orange peel biochar acts as an adsorbent. Meanwhile, the Reactive Orange 16 acts as an adsorbate.

3.1.1 Apparatus and Equipment

The apparatus used were volumetric flasks (50 mL, 100 mL, and 500 mL), conical flasks (250 mL), beakers (100 mL, 200 mL, and 500 mL), measuring cylinder (10 mL and 50 mL), filter papers (Smith 102 – 125 mm), filter funnels (75 mm), cuvette spectrophotometer, spatula, glass rod, dropper, micro pipet (50 μ l and 1000 μ l), tips, aluminium foil, and zip lock (4 cm x 6 cm and 6 cm x 9 cm). The equipment used were oven, blender, sieve (500 μ m), furnace, crucible, weighing balance, UV Spectrophotometer, FTIR, and BET.

3.1.2 Chemicals and Reagent

The chemical used was Reactive Orange 16 ($C_{20}H_{17}N_3Na_2O_{11}S_3$).

3.2 Methods

3.2.1 Preparation of Adsorbent (Orange Peel Biochar)

Orange peel waste was cleaned to remove the dirt and cut into small sizes. Orange peel was dried under sunlight for three days, to remove the moisture content of orange peel. The dry orange peel was ground using grinder or blender. The powder was sieved using a 500 μm sieve. The orange peel powder was weighed for 30 g. The orange peel had run carbonisation (slow pyrolysis) process using a furnace at temperatures 400 °C and 700 °C for 2 hours. The orange peel biochar was put in a sealed plastic bag or zip lock bag.

3.2.2 Preparation of Adsorbate (RO16 Dye Stock Solution)

0.5 g of dye was dissolved in 500 mL of distilled water using a volumetric flask to obtain 1000 mg/L concentration.

3.2.3 Preparation of Calibration Curve

The Reactive Orange 16 stock solution was diluted with distilled water to obtain 0.5, 2.0, 4.0, 6.0, 8.0, 10.0, 12.0, and 14.0 mg/L concentration. The distilled water was used as a blank. The several concentrations of Reactive Orange 16 were measured using a UV spectrophotometer at a wavelength of 494 nm.

3.2.4 Effect of Carbonisation Temperature

30 g of orange peel powder was placed in a closed crucible for each temperature. The carbonisation temperatures studied were 400 °C and 700 °C for 2 hours using a furnace. 0.5 g of orange peel biochar, 50 mL of 50 mg/L, 5 hours, and under room temperature were fixed. The glass rod was used to mix the orange peel biochar and Reactive Orange 16. Next, the mixture of orange peel biochar (adsorbent) and Reactive Orange 16 (adsorbate) was filtered using filter paper. Note: This process was repeated 3

times. Then, the filtrate was analyzed using a UV spectrophotometer at a wavelength of 494 nm.

3.2.5 Effect Amount of Adsorbent Dose

The amounts of adsorbent dose used were 0.2 g, 0.5 g, 0.8 g, 1.0 g, 1.2 g, and 1.5 g. The experiment was conducted using 50 mL of Reactive Orange 16 dye at 50 mg/L, contact time at 5 hours and under room temperature. The glass rod was used to mix the orange peel biochar and Reactive Orange 16. Next, the mixture of orange peel biochar and Reactive Orange 16 was filtered using filter paper. Note: This process was repeated 3 times. Then, the filtrate was analyzed using a UV spectrophotometer at a wavelength of 494 nm.

3.2.6 Effect of Initial Dye Concentration

The initial dye concentrations were studied at 50 mg/L, 100 mg/L, 150 mg/L, 200 mg/L, and 250 mg/L. The experiment was conducted using 50 mL of Reactive Orange 16 dye at different concentrations, adsorbent dosage at 1.2 g, contact time at 5 hours and under room temperature. The glass rod was used to mix the orange peel biochar and Reactive Orange 16. Next, the mixture of orange peel biochar and Reactive Orange 16 was filtered using filter paper. Note: This process was repeated 3 times. Then, the filtrate was analyzed using a UV spectrophotometer at a wavelength of 494 nm.

3.2.7 Effect of Contact Time

The contact times studied were 1 hour, 3 hours, 5 hours, 8 hours, and 24 hours. The experiment was conducted using 50 mL of Reactive Orange 16 dye at 100 mg/L, adsorbent dosage at 1.2 g, and under room temperature. The glass rod was used to mix the orange peel biochar and Reactive Orange 16. Next, the mixture of orange peel biochar and Reactive Orange 16 was filtered using filter paper. Note: This process was repeated 3 times. Then, the filtrate was analyzed using a UV spectrophotometer at a wavelength of 494 nm.

3.3 Characterization of Orange Peel Biochar

3.3.1 Characterization Using FTIR

0.1 g of orange peel biochar (powder) before and after adsorption was studied using FTIR to identify the presence of functional group. The wavelength used was 500 cm^{-1} to 4000 cm^{-1} .

3.3.2 Characterization Using BET

1.0 g of orange peel biochar (powder) before and after adsorption was studied using BET to determine the surface area and pore size distribution. BET was performed by an automated gas sorption instrument (autosorb iQ) to determine the surface area of orange peel biochar.

3.4 Data Analysis

3.4.1 Adsorption Capacity

Adsorption capacity was calculated using equation 3.1,

$$q_e = \frac{(C_0 - C_e) V}{m}$$

Equation 3.1

Where:

C_e = Equilibrium dye concentration in the solution (mg/L)

C_0 = Initial dye concentration in the solution (mg/L)

V = Volume of solution (L)

m = Mass of adsorbent (g)

3.4.2 Langmuir Adsorption Model

Langmuir adsorption model was calculated using equation 3.2,

$$q_e = \frac{q_{\max} K_L C_e}{1 + K_L C_e} \quad \text{Equation 3.2}$$

Where:

q_e = Quantity of dye uptake (mg/g)

q_{\max} = Maximum adsorption capacity (mg/g)

K_L = Langmuir constant

C_e = Equilibrium dye concentration

3.4.3 Linearised Langmuir Isotherm

Linearised Langmuir isotherm was calculated using equation 3.3,

$$\frac{C_e}{q_e} = \frac{1}{q_{\max} K_L} + \frac{1}{q_{\max}} C_e \quad \text{Equation 3.3}$$

Where:

q_e = Amounts of dye adsorbed at equilibrium (mg g⁻¹)

C_e = Amounts of unadsorbed dye concentration in aqueous solution (mg L⁻¹)

q_{\max} = The monolayer biosorption capacity of the adsorbent (mg g⁻¹)

K_L = Langmuir constant (Lm g⁻¹)

3.4.4 Freundlich Adsorption Model

Freundlich adsorption model was calculated using equation 3.4,

$$q_e = K_f C_e^{\frac{1}{n}} \quad \text{Equation 3.4}$$

Where:

K_f = Freundlich constant (mg/g)

n = Adsorption intensity

C_e = Equilibrium dye concentration (mg/L)

q_e = Quantity of dye uptake (mg/g)

3.4.5 Linearised Freundlich Isotherm

Linearised Freundlich isotherm was calculated using equation 3.5,

$$\log Q_e = \frac{1}{n} \log C_e + \log K_f \quad \text{Equation 3.5}$$

Where:

K_f = Adsorption capacity (mg/g)

$1/n$ = Linearity of adsorption

RESULT AND DISCUSSION

4.1 Characterization of Adsorbent

In this study, reactive orange 16 (RO16) was removed using orange peel biochar as an adsorbent. The adsorbent was characterized using Fourier Transform Infrared Spectroscopy (FTIR) and Brunauer-Emmett-Teller (BET).

4.1.1 Fourier Transform Infrared Spectroscopy (FTIR)

The FTIR spectrum on orange peel biochar before adsorption and orange peel biochar after adsorption was shown in Figure 4.1. Based on the figure, there was no functional group present in the orange peel biochar. It is because of functional groups may break down due to the high temperatures used in the carbonisation process, which would further reduce their presence in the biochar that is produced (Thomas et al., 2021). However, according to study orange peel biochar has the following functional groups: C-H aliphatic, C=C, and C-H aromatic. When orange peel was subjected to FTIR quality examination, the following spectra were found, 1490 cm^{-1} with strong intensity (C-H aromatic), 1645 cm^{-1} with medium intensity (C=C), and 2860 cm^{-1} with medium intensity (C-H aliphatic) (Cantika et al., 2023). In addition, according to Patino (2021), point out that the orange peel's Cr (III) adsorption process involves a number of functional groups such as carboxylic groups and esters, as well as the CH and CH₂ groups of aliphatic molecules, cellulose, hemicellulose, and pectin. The qualities and possible applications of orange peel biochar can be influenced by these functional groups, which also contribute to its chemical structure.

However, its usefulness is not always limited by the absence of functional groups because other characteristics like porosity and surface area might increase how successful it is when used to remove dyes (Hu et al., 2023).

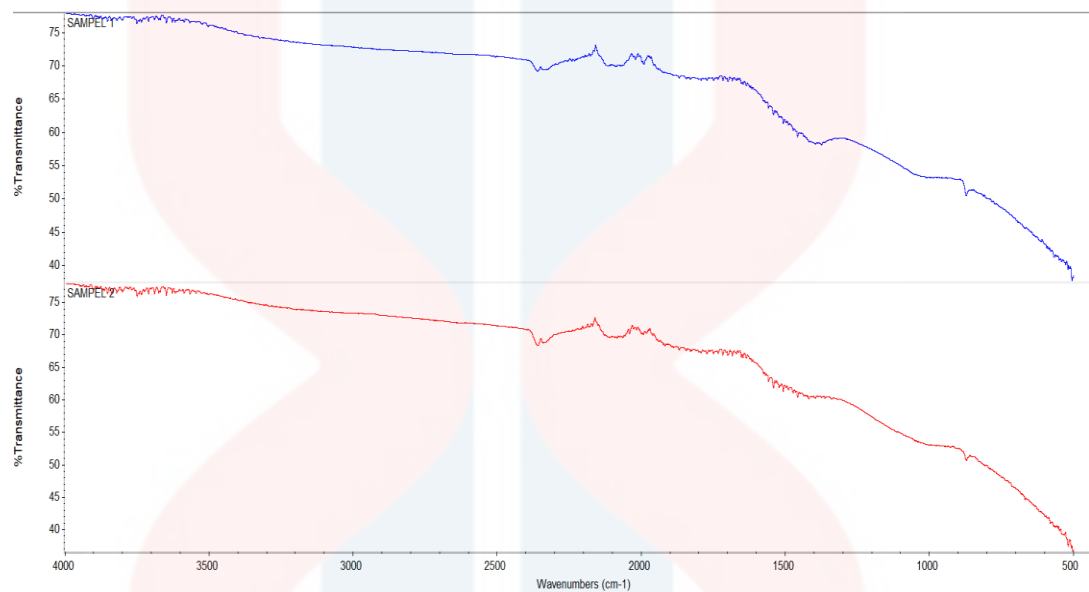


Figure 4.1: FTIR on orange peel biochar before adsorption (blue) and orange peel biochar after adsorption (red).

4.1.2 Brunauer-Emmett-Teller (BET)

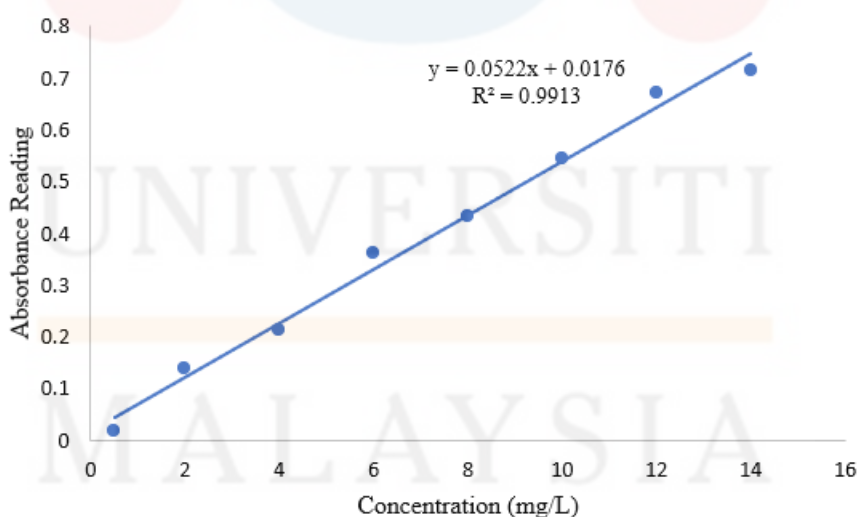
Based on the Table 4.1, the surface area of orange peel biochar before adsorption was $77.811 \text{ m}^2/\text{g}$ and the surface area was decreased to $65.893 \text{ m}^2/\text{g}$ after adsorption process occurred. The surface area decreased because of the molecules of the Reactive Orange 16 fill up the pores, taking up space and form a layer on the surface of orange peel biochar. Therefore, lowering the volume that can hold more molecules of Reactive Orange 16. Hence, through obstructing pore access, this layer can effectively decrease surface area of pore. According to Zango et al. (2020) having a larger surface area means there are more active sites available for adsorption to take place. The areas of a material's surface known as "active sites" are where interactions between the adsorbate molecules and the adsorbent occur. Thus, the total adsorption capacity increases with a bigger surface area because there are more opportunities for these interactions to happen at the same time and that is advantageous for the removal of dye.

Table 4.1: Surface area of orange peel biochar before and after adsorption.

Orange peel biochar	Surface area (m ² /g)
Before adsorption	77.811
After adsorption	65.893

4.2 Calibration Curve

The calibration curve of Reactive Orange 16 at 494 nm is shown in Figure 4.2. The calibration curve's coefficient value of 0.9913, which represents a good result, was obtained. A benchmark for evaluating the acceptability of research that show an exact correlation of 1 or close to 1 is the correlation coefficient (R^2). A high correlation is shown by a value near 1. It aids in determining how linear the calibration curve is. The final dye concentration after adsorption was calculated using the liner calibration equation ($y = 0.0522x + 0.0176$).

**Figure 4.2:** Linear calibration curve of RO16 at 494 nm.

4.3 Effect of Carbonisation Temperature

Figure 4.3 illustrates how the carbonisation temperature affects the RO16 dye removal process. The orange peel was heated in the furnace for two hours at two different temperatures 400 °C and 700 °C for the purpose of this study. The highest percentage of removal, 90.06 %, was obtained at the optimal carbonisation temperature of 700 °C. Zhao et al. (2022) claimed the enrichment of the micropore volume following the emission of volatile materials, the carbonisation temperature affected the pore structure and surface area of biochar. Increasing carbonisation temperatures led to an increase in the surface area and porosity of the orange peel biochar, which in turn produced the furan and arene groups (Deshan et al., 2021). In addition, the higher carbonisation temperatures also result in low polarity, high aromatic structures, and a high carbon content. When removing Reactive Orange 16 dye, it improves the effectiveness of the adsorption capacity gave the highest percentage removal of dye.

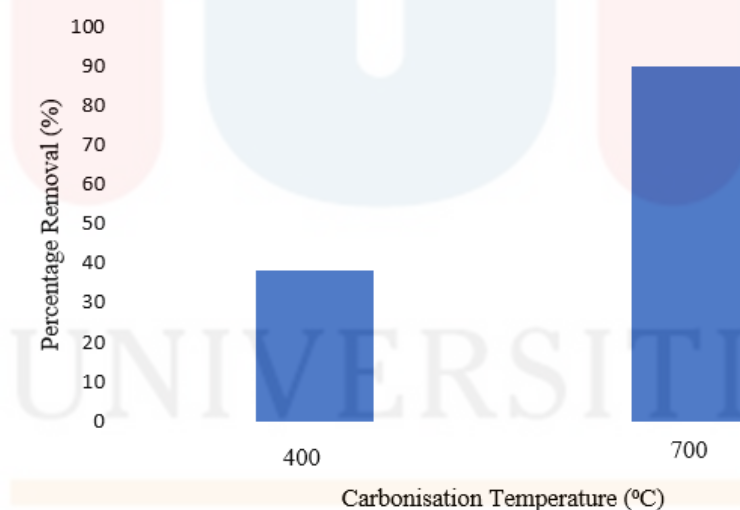


Figure 4.3: Effect of carbonisation temperature (°C) on the removal of Reactive Orange 16 dye.

4.4 Effect of Adsorbent Dosage

Figure 4.4 demonstrated how the amount of adsorbent affected the Reactive Orange 16 dye's elimination. Various dosages of orange peel biochar, including 0.2 g, 0.5 g, 0.8 g, 1.0 g, 1.2 g, and 1.5 g, were studied. Based on the figure, the proportion of Reactive Orange 16 dye removed increased slightly from 71.48 % to 90.90 % at 0.2 g to

1.5 g of adsorbent. It depends on the surface site vacancy and the fixed mass of orange peel biochar to adsorb a specific amount of Reactive Orange 16 molecule. Thus, a large dosage of adsorbent aids in raising the clearance percentage while staying constant beyond the optimal point. Accordingly, when the amount of adsorbent increases, more dye molecules can be removed from the solution (Gajipara et al., 2023). The limiting of dye concentration at high dosages is the reason for this. The ideal value in this investigation is achieved with a 1.2 g adsorbent dosage, 50 mg/L dye concentration, and 50 mL of solution volume. However, because it turned colourless and initially appeared at this dosage, 1.2 g of present was determined to be the ideal amount.

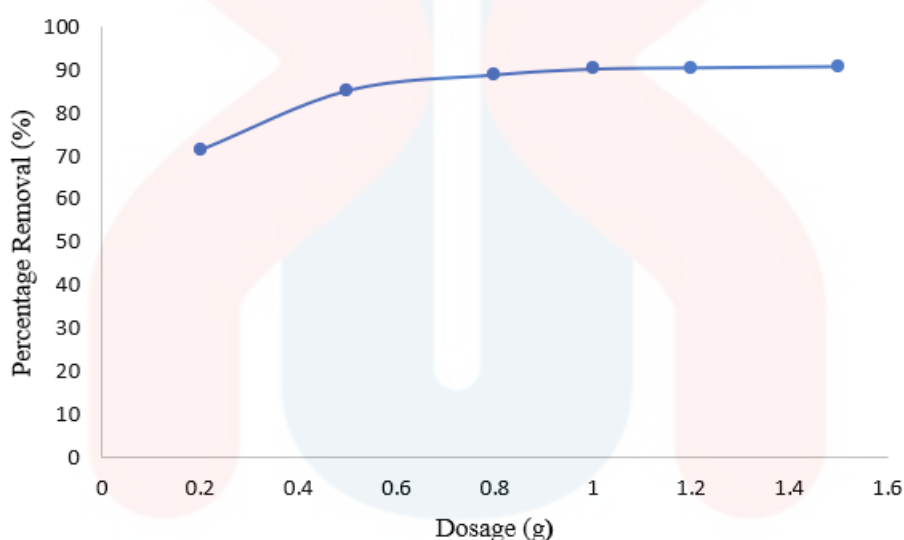


Figure 4.4: Effect of adsorbent dosage (g).

4.5 Effect of Initial Dye Concentration

Based on Figure 4.5, strong driving forces between the liquid and the adsorbent caused the initial adsorption to occur quickly. The loading surface will then start to affect slowly. Higher initial concentrations resulted in more competition between Reactive Orange 16 molecules for available adsorption sites on the orange peel biochar (Gajipara et al., 2023). Therefore, this enhanced competition has the potential to alter adsorption rates and overall process efficiency. The graph indicates that the percentage removal for 50 mg/L and 100 mg/L were high at 91.67 % and 91.64 %, respectively, and that the removal rate slows down above 100 mg/L concentration. The ideal initial dye concentration for this investigation was determined to be 100 mg/L. Hence, 100

mg/L of initial dye concentration, can be adsorbed using 1.2 g of the ideal adsorbent dosage.

Furthermore, saturated adsorption during the equilibrium phase also contributes to an increase in the final dye concentration. Since the adsorbent cannot adsorb the dye molecules, they are free. According to Gajipara et al. (2023), the external boundary layer's thickness is indicated by the slope's constant value between 50 and 100 mg/L. Nevertheless, the slope starts to decline at 150 mg/L and continues until 250 mg/L because the external boundary layer offers less resistance to mass transfer. When treating high initial dye concentrations, such as 100 mg/L, 1.2 g of adsorbent dose is most efficient.

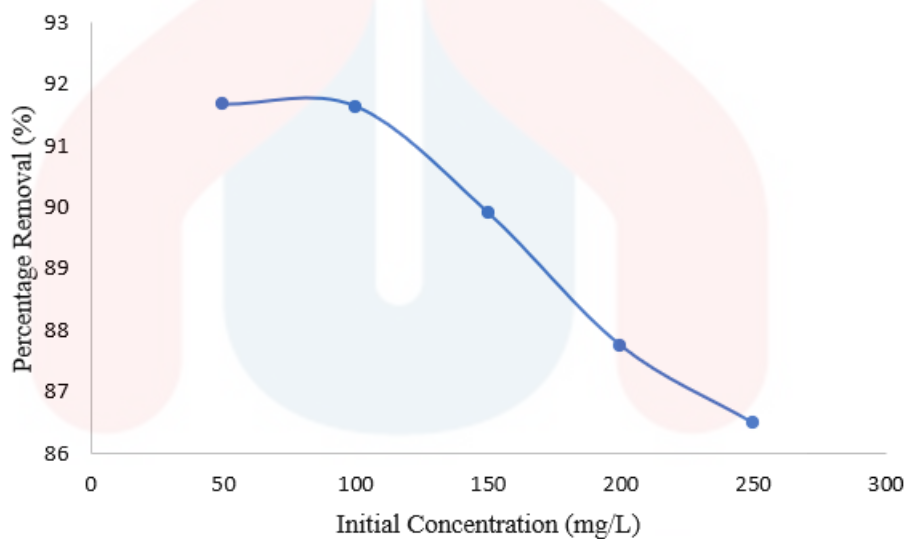


Figure 4.5: Effect of initial dye concentration (mg/L).

4.6 Effect of Contact Time

Figure 4.6 displayed the effect of contact time for Reactive Orange 16 removal. In this study, several interaction times, including 1 hour, 3 hours, 5 hours, 8 hours, and 24 hours, were examined. The best contact duration in this study was determined to be 8 hours due to the higher percentage of Reactive Orange 16 dye removal is 97.29 %. As the duration of contact increases, the percentage elimination increases every hour to 24 hours and stays constant. Because sufficient contact time is required for effective mass transfer of Reactive Orange 16 molecules from the solution to the adsorption sites on

the orange peel biochar (Elwardany et al., 2023). Therefore, short contact periods may impede mass transfer, resulting in reduced adsorption capability. This is influenced by attractive forces that exist between the dye molecule and the adsorbent, such as ionic bond, van der Waals forces and electrostatic attracting forces. Diffusion began at the orange peel biochar's outside and proceeded via its pores into the intraparticle matrix until it reached an equilibrium phase. At 8 hours after adsorption, solutions lose their hue. Nonetheless, 8 hours can adequately treat dye in the minimum amount of time.

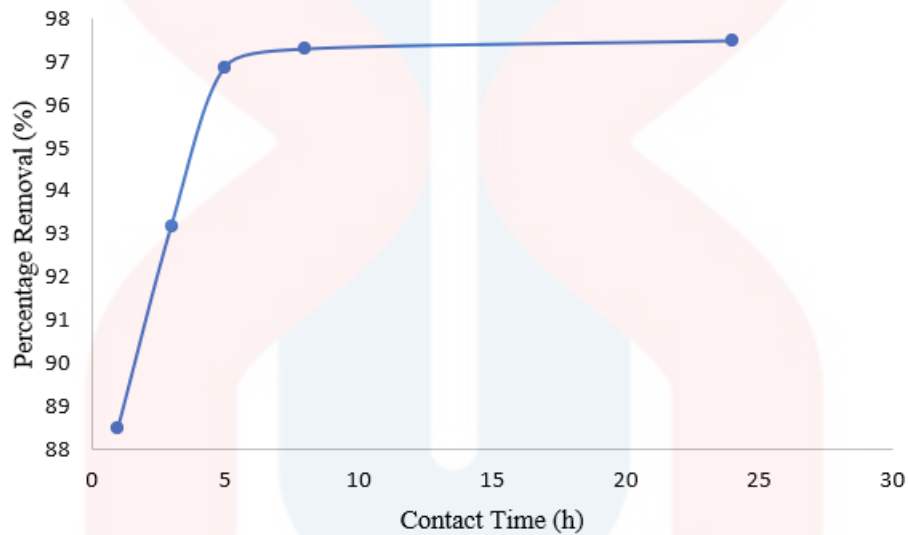


Figure 4.6: Effect of contact time (h).

4.7 Adsorption Isotherm

Based on data relating to initial dye concentration and contact time, a graph of Langmuir and Freundlich adsorption was created.

4.7.1 Langmuir Isotherm and Freundlich Isotherm

In Table 4.2, the parameters for the Freundlich and Langmuir isotherms for Reactive Orange 16 were displayed. The correlation coefficient (R^2) between initial dye concentration (Figure 4.7) and contact time (Figure 4.8) is 0.980 and 0.999, respectively, according to the Langmuir isotherm. The contact time parameter's correlation coefficient (R^2) displays the most optimal value when it approaches 1. The

greatest amount of dye that orange peel biochar can absorb in this investigation is 17.361 mg/g. Meanwhile, according to the Freundlich adsorption, the correlation coefficient (R^2) for initial concentration (Figure 4.9) and contact time (Figure 4.10) is 0.983 and 0.971. The contact time value that corresponded to the adsorption capacity was consistently 4.318 mg/g.

It suggests that because it is closest to 1, the Langmuir isotherm is most appropriate for the adsorption model employed in this investigation. The Freundlich isotherm's correlation coefficient (R^2) in the contact time parameter was not very close to 1. It shows that the adsorption model employed in this work is not compatible with the Freundlich isotherm. Therefore, the adsorption capacity varies depending on the type of agricultural waste. However, compared to other adsorbents, the orange peel biochar adsorption capability was deemed satisfactory for these investigations.

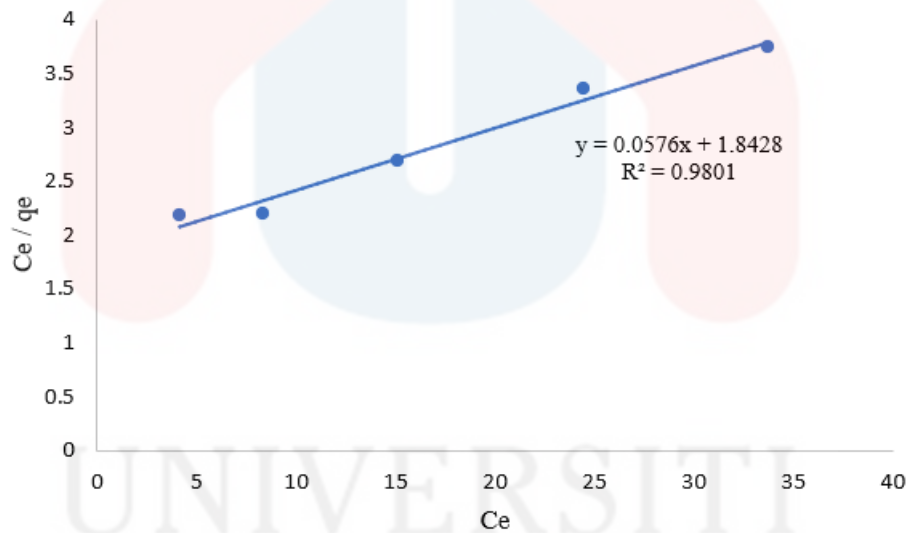


Figure 4.7: Langmuir isotherm for the effect of initial dye concentration.

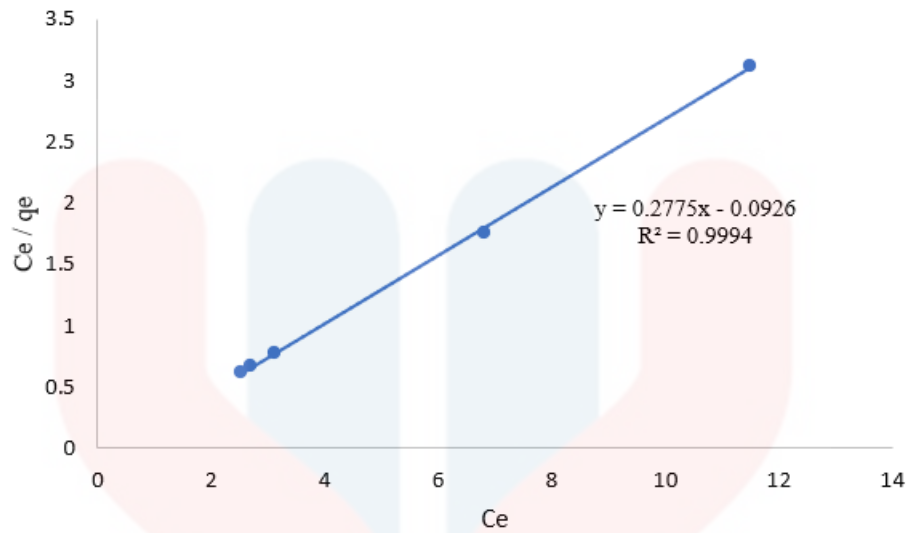


Figure 4.8: Langmuir isotherm for the effect of contact time.

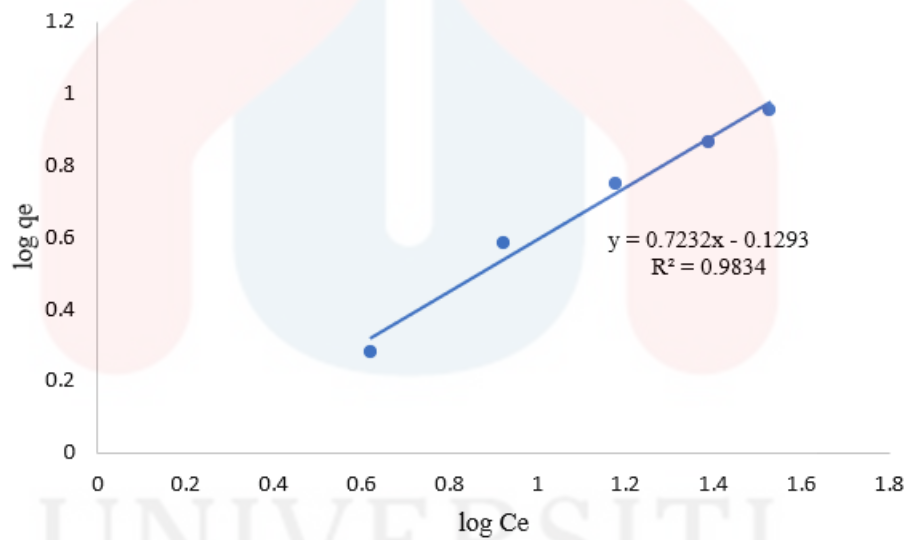


Figure 4.9: Freundlich isotherm for the effect of initial dye concentration.

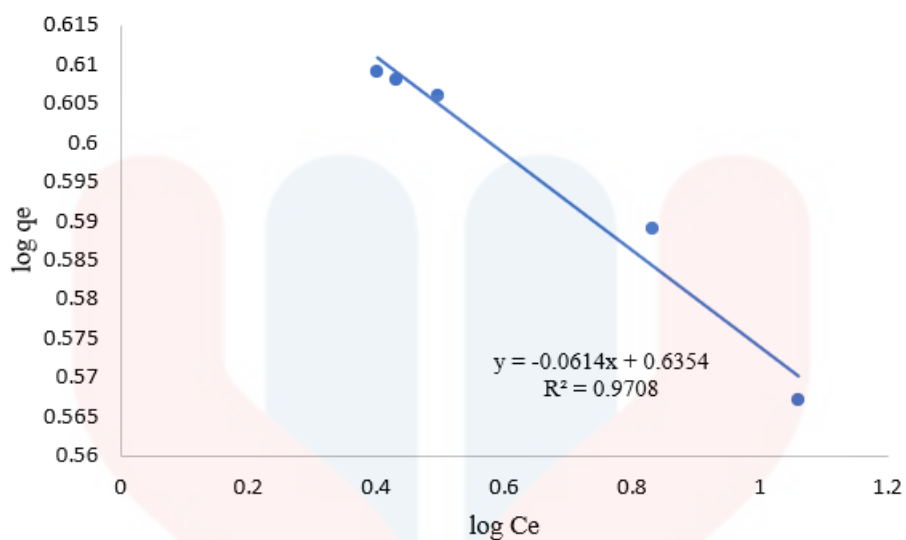


Figure 4.10: Freundlich isotherm for the effect of contact time.

Table 4.2: Parameters of Langmuir (a) and Freundlich (b) isotherm for Reactive Orange 16

Adsorption Isotherm	Parameters	q_{\max} (mg/g)	K_L (L/mg)	R^2
Langmuir	Initial concentration	17.361	0.031	0.980
Langmuir	Contact time	3.604	2.997	0.999
Adsorption Isotherm	Parameters	N	K_L (L/mg)	R^2
Freundlich	Initial concentration	1.383	0.743	0.983
Freundlich	Contact time	-16.287	4.318	0.971

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

In conclusion, the removal of Reactive Orange 16 (RO16) dye has been successful using orange peel biochar. The orange peel biochar has shown a significant adsorption capacity, indicating its promise as an economical and environmentally friendly substitute for treating wastewater. Applying the carbonisation principle in this study facilitates the production of high porosity, a greater surface area, and a rich carbon content (Yang et al., 2020). Because of its porous structure and large surface area, biochar is often good at adsorbing substances through a variety of mechanisms, such as electrostatic interactions.

In this research, various parameters were used to determine the ideal adsorption. 700 °C was determined to be the ideal carbonisation temperature because of the high percentage of Reactive Orange 16 dye elimination. Following that, 1.2 g of orange peel biochar works well as an adsorbent dose to remove 100 mg/L of Reactive Orange 16 dye, with a contact time of eight hours. The larger surface area was proven by BET analysis. Thus, the equilibrium data in this research indicated the monolayer adsorption between orange peel biochar as an adsorbent and Reactive Orange 16 as an adsorbate, and it was best fitted to the Langmuir isotherm model.

5.2 Recommendation

Recommendations are crucial for advancing new information in all circumstances, including experiments. Here are a few suggestions for this research. Firstly, make sure all apparatus and equipment have been dried. Moisture or water content can affect the accuracy of measurement of the chemical substances.

Secondly, studying the FTIR analysis of raw orange peel powder to make the comparison of functional group between the raw orange peel powder and orange peel biochar. In addition, studying the FTIR analysis of orange peel biochar at 400 °C and 700 °C will allow you to compare the carbon content first. Thirdly, adding a few analysis investigations, including Thermogravimetric Analysis (TGA), Response Surface Methodology (RSM), X-Ray Fluorescence (XRF), X-Ray Diffraction (XRD), and Scanning Electron Microscopy (SEM).

TGA is useful in determining the temperature ranges at which various biochar constituents volatilize or break down for understanding the thermal properties and stability of biochar (Hassaan et al, 2023). Meanwhile, RSM can be used to optimise the biochar synthesis process by taking factors such as temperature, residence duration, and feedstock type. Moreover, XRF is a technique that measures the distinctive X-ray fluorescence released when the sample is exposed to X-ray radiation. It is used to analyse the elements of biochar. Furthermore, XRD aids in determining which mineral phases are found in biochar. It helps determine the crystalline structure of the ash components in biochar and comprehending how the pyrolysis conditions and feedstock affect the final mineral composition (Alorabi et al., 2020). Finally, pore size distribution, surface roughness, and the existence of any coatings or contaminants are all visible when the surface structure of biochar particles is examined using SEM (Abdo et al., 2023).

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APPENDIX A



Figure A1: Orange peel.



Figure A2: Reactive Orange 16 stock.



Figure A3: Orange peel biochar.



Figure A4: Calibration curve.

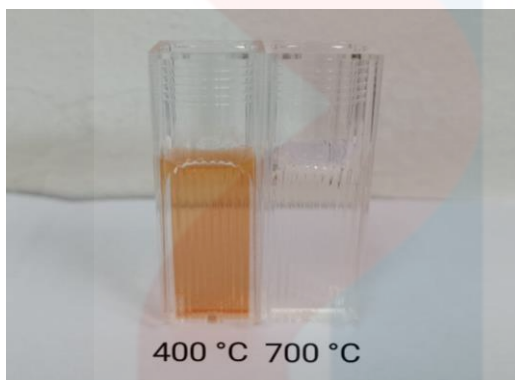


Figure A5: Effect of carbonisation temperature.

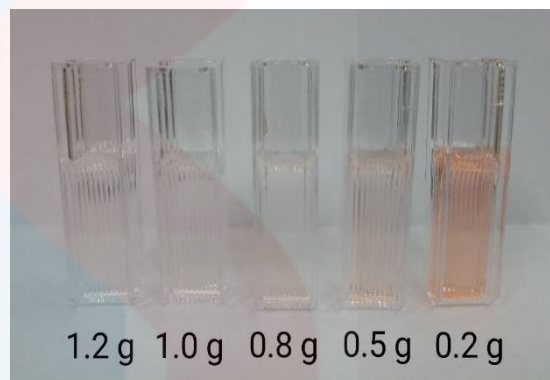


Figure A6: Effect of adsorbent dosage.

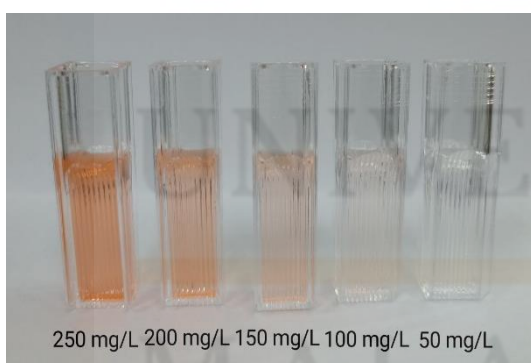


Figure A7: Effect of initial concentration.

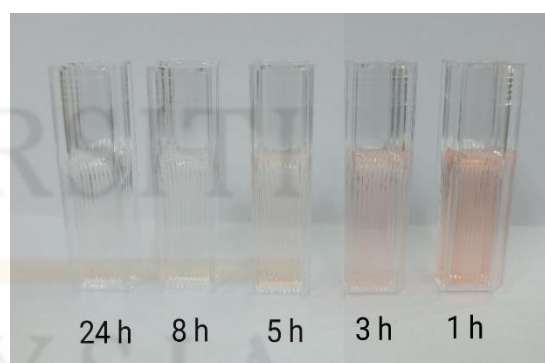


Figure A8: Effect of contact time.

MBET summary	
Slope =	45.513 1/g
Intercept =	-7.564e-01 1/g
Correlation coefficient, r =	0.998357
C constant=	-59.166
Surface Area =	77.811 m ² /g

Figure A9: Surface area of orange peel biochar before adsorption.

MBET summary	
Slope =	54.124 1/g
Intercept =	-1.273e+00 1/g
Correlation coefficient, r =	0.998096
C constant=	-41.528
Surface Area =	65.893 m ² /g

Figure A10: Surface area of orange peel biochar after adsorption.

APPENDIX B

Table B1: Effect of carbonisation temperature (°C).

Carbonization Temperature (°C)	Average Reading (Abs)	Final Concentration (mg/L)	Removal Percentage (%)	Adsorption Capacity (mg/g)
400	1.627	30.831	38.338	1.917
700	0.277	4.969	90.062	4.503

Table B2: Effect of adsorbent dosage (g).

Adsorbent Dosage (g)	Average Reading (Abs)	Final Concentration (mg/L)	Removal Percentage (%)	Adsorption Capacity (mg/g)
0.2	0.762	14.261	71.478	8.935
0.5	0.401	7.345	85.320	4.266
0.8	0.306	5.525	88.950	2.780
1.0	0.271	4.854	90.330	2.257
1.2	0.263	4.701	90.598	1.887
1.5	0.255	4.548	90.904	1.515

Table B3: Effect of initial concentration (mg/L).

Initial Concentration (mg/L)	Average Reading (Abs)	Final Concentration (mg/L)	Removal Percentage (%)	Adsorption Capacity (mg/g)
50	0.235	4.165	91.670	1.910
100	0.454	8.360	91.640	3.818
150	0.808	15.142	89.905	5.619
200	1.297	24.510	87.745	7.312
250	1.781	33.782	86.487	9.009

Table B4: Effect of contact time (h).

Contact Time (h)	Average Reading (Abs)	Final Concentration (mg/L)	Removal Percentage (%)	Adsorption Capacity (mg/g)
1	0.618	11.502	88.498	3.687
3	0.373	6.808	93.192	3.883
5	0.181	3.130	96.870	4.036
8	0.159	2.709	97.291	4.054
24	0.149	2.517	97.483	4.062

APPENDIX C

Table C1: Adsorption isotherm for initial concentration.

Initial Concentration (mg/L)	C_e	q_e	C_e / q_e	$\log C_e$	$\log q_e$
50	4.165	1.910	2.181	0.620	0.281
100	8.360	3.818	2.190	0.922	0.582
150	15.142	5.619	2.695	1.180	0.750
200	24.510	7.312	3.352	1.389	0.864
250	33.782	9.009	3.750	1.529	0.955

Table C2: Adsorption isotherm for contact time.

Contact Time (h)	C_e	q_e	C_e / q_e	$\log C_e$	$\log q_e$
1	11.502	3.687	3.120	1.061	0.567
3	6.808	3.883	1.753	0.833	0.589
5	3.130	4.036	0.776	0.496	0.606
8	2.709	4.054	0.668	0.433	0.608
24	2.517	4.062	0.620	0.401	0.609

Table C3: Parameters of Langmuir and Freundlich isotherm for Reactive Orange 16 (R016).

Adsorption Isotherm	Parameters	q_{\max} (mg/g)	K_L (L/mg)	R^2
Langmuir	Initial concentration	17.361	0.031	0.980
Langmuir	Contact time	3.604	2.997	0.999
Adsorption Isotherm	Parameters	N	K_L (L/mg)	R^2
Freundlich	Initial concentration	1.383	0.743	0.983
Freundlich	Contact time	-16.287	4.318	0.971