

ADSORPTION of METHYL ORANGE DYE USING POWDERED ACACIA (*Crassiacarpa*) BARK AS AN ADSORBENT

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ABSTRAK

Serbuk kulit kayu *Acacia crassiacarpa* adalah biomassa lignoselulosa yang kaya akan selulosa, diteliti sebagai biosorben untuk menghilangkan Metil Oranye (MO) dari larutan berair. Studi ini bertujuan untuk menentukan kondisi adsorpsi optimum, termasuk dosis adsorben, pH, waktu kontak, kecepatan pengadukan, konsentrasi adsorbat, dan suhu. Eksperimen adsorpsi dilakukan menggunakan sistem batch, dan adsorben dikarakterisasi menggunakan analisis FTIR, SEM-EDS, dan BET. Kondisi optimum diperoleh pada dosis 0,75 g, pH 2, waktu kontak 80 menit, kecepatan pengadukan 200 rpm, konsentrasi MO 20 ppm, dan suhu 30 °C, mencapai efisiensi adsorpsi sebesar 82,34% dan kapasitas sebesar 5,6492 mg/g. SEM-EDS menunjukkan morfologi berpori yang didominasi oleh unsur karbon dan oksigen. Analisis BET menunjukkan luas permukaan sebesar 0,460 m²/g, sedangkan FTIR mengkonfirmasi keberadaan gugus fungsional hidroksil, karbonil, dan aromatik yang terlibat dalam adsorpsi. Hasil ini membuktikan bahwa akasia dapat menjadi adsorben.

Kata kunci: adsorpsi, akasia *Crassiacarpa*, *Methyl Orange*, zat warna.

ABSTRACT

Acacia crassiacarpa bark powder, a lignocellulosic biomass rich in cellulose, was investigated as a biosorbent for the removal of *Methyl Orange* (MO) from aqueous solutions. This study aimed to determine the optimum adsorption conditions, including adsorbent dosage, pH, contact time, stirring speed, adsorbate concentration, and temperature. Adsorption experiments were conducted using a batch system, and the adsorbent was characterized using FTIR, SEM-EDS, and BET analyses. The optimum conditions were obtained at a dosage of 0.75 g, pH 2, contact time of 80 min, stirring speed of 200 rpm, MO concentration of 20 ppm, and temperature of 30 °C, achieving an adsorption efficiency of 82.34% and a capacity of 5.6492 mg/g. SEM-EDS showed a porous morphology dominated by carbon and oxygen elements. BET analysis showed a surface area of 0.460 m²/g, while FTIR confirmed the presence of hydroxyl, carbonyl, and aromatic functional groups involved in the adsorption. These results prove that acacia can be an adsorbent.

Keywords: adsorption, acasia *Crassiacarpa*, *Methyl Orange*, dyes.

INTRODUCTION

The bark of *Acacia crassicarpa* contains 55.48% cellulose and 24.13% lignin, which differs from *Acacia mangium* that contains 51.46% cellulose and 27.66% lignin. The percentage indicates that *Acacia crassicarpa* has a higher cellulose content compared to other *Acacia* species. *Acacia* bark, an abundant by-product of the wood industry in Indonesia, is mostly utilized as compost in the plantation industry (Wina et al., 2010). According to the Central Bureau of Statistics (2018), the production of roundwood from *Acacia* plantations in Riau Province has increased annually since 2016, with industrial timber estate (ITE) roundwood production of 16,991,099 tons in 2016, rising to 19,922,579 tons in 2017, and further increasing to 19,965,510 tons in 2018. According to the Central Bureau of Statistics (BPS) in 2020, the area of *Acacia* industrial plantation forests (HTI) in Riau Province covered 2,335,450 ha, with a roundwood production of 31,509,228 m³ (Puspita et al., 2022). The average wood production of *Acacia crassicarpa* reaches 27 m³/ha/year. Research conducted by Nguyen et al., (2021) successfully utilized *Acacia crassicarpa* bark for the adsorption of *Methyl Orange* and Methylene Blue dyes with adsorption capacities of 10.36 mg/g and 15.34 mg/g, respectively. Based on its chemical composition and abundance, *Acacia crassicarpa* bark has strong potential to be developed as an adsorbent in adsorption processes.

Methyl Orange is a synthetic dye widely used in the textile industry. It is synthesized from sulfanilic acid and N,N-dimethylaniline (Nurlaili et al., 2017). The permissible limit of *Methyl Orange* in aquatic environments ranges between 5–10 mg/L, and therefore, various treatment methods are required to reduce its concentration in wastewater, one of which is adsorption. *Methyl Orange* is toxic, carcinogenic and mutagenic characteristics. Adsorption is a mass transfer process that occurs on the pore surface of adsorbent particles. It takes place due to surface energy and the attractive forces at the interface (Pungut et al., 2021). Adsorption is considered the most advantageous method due to its simple process, high adsorption efficiency and capacity, selectivity, and low operational cost.

Adsorbent dosage affects the adsorption process because each active site can bind only one adsorbate molecule. When all active sites become saturated, no further increase in adsorption occurs (Veronika et al., 2023). Changes in acidity (pH) can affect the chemical and surface properties of the adsorbent, the solubility of the adsorbate, as well as ion competition during the adsorption process (Ngapa & Ika, 2021). Higher adsorbate concentration leads to greater adsorption because more molecules accumulate on the adsorbent surface. Contact time plays a crucial role in the adsorption process; the longer the contact time, the greater the

adsorption capacity (Ikhsany et al., 2024). Stirring speed affects adsorption by improving mixing between the adsorbent and adsorbate, which increases adsorption efficiency. Temperature affects adsorption because higher temperatures increase particle movement, which can cause adsorbate to detach from the adsorbent (Ikhsany et al., 2024). The significant differences observed may be attributed to the different types of adsorbent used. In this study, unactivated bark was employed to evaluate the intrinsic adsorption capacity of the bark. The research also aims to serve as a comparison with previous studies.

Although previous studies have reported higher adsorption capacities using activated *Acacia crassicaarpa* bark (Nguyen et al., 2021), such treatments involve additional chemical or thermal processes that increase cost and environmental burden. Therefore, evaluating the adsorption performance of untreated (non-activated) bark is important to assess its intrinsic adsorption capability as a low-cost and sustainable biosorbent. The significant differences observed may be attributed to the different types of adsorbent used. In this study, unactivated bark was employed to evaluate the intrinsic adsorption capacity of the bark. The research also aims to serve as a comparison with previous studies.

METHOD

Tools and Materials

The instruments used in this study included a chopper, grinder, 100 and 200 mesh sieves (CBN test sieve analysis), oven (Stericell), analytical balance (Kern ABJ-NM/ABS-N), spray bottle, stirring rod, spatula, pH meter (Hanna), hotplate (Corning PC-420D), magnetic stirrer (IKA C-MAG MS10), centrifuge (IEC Centra CL2), Scanning Electron Microscopy–Energy Dispersive X-Ray Spectroscopy (SEM-EDS, JEOL JBM-6510LA), Brunauer–Emmett–Teller (BET, Quantachrome Quadrasorb-Evo), Fourier Transform Infrared Spectroscopy (FTIR, IR Prestige-21, Shimadzu), UV-Vis spectrophotometer (Agilent Cary 60 UV-Visible Spectrophotometer), and other standard laboratory glassware. The materials used in this research were *Methyl Orange* powder ($C_{14}H_{14}N_3NaO_3S$), *Acacia crassicaarpa* bark samples, sodium chloride (NaCl), sodium hydroxide (NaOH), hydrochloric acid (HCl), ethanol (C_2H_6O), demineralized water (H_2O), distilled water (H_2O), and aluminum foil.

Procedure

Adsorbent preparation

Acacia crassicaarpa bark samples were obtained from an industrial plantation forest (HTI) in Riau. The bark was washed with distilled water and then dried under sunlight for ± 7 days. The dried *Acacia crassicaarpa* bark was ground using a mortar and pestle, and then sieved using

a 100-mesh sieve and retained on a 200-mesh sieve, resulting in a particle size of $100 \text{ mesh} < x < 200 \text{ mesh}$ ($x = \text{particle size}$). The powdered *Acacia crassicarpa* bark obtained was subsequently oven-dried at $105 \text{ }^\circ\text{C}$ for 24 hours.

Adsorbent Characterization

Acacia crassicarpa bark powder was characterized before and after adsorption using several methods. FTIR was used to identify the functional groups in the material, SEM-EDS examined the surface morphology and elemental composition, and BET analysis measured the specific surface area and pore size of the bark powder prior to adsorption. These analyses help understand the adsorption properties of the material.

Optimization of adsorbent

A 50 mL solution of *Methyl Orange* with a concentration of 20 ppm was prepared at pH 2 in a 100 mL beaker. *Acacia crassicarpa* bark powder with varying dosages of 0,10; 0,25; 0,50; 0,75; 1,00; and 1,25 g was added to each solution. The mixtures were stirred at 150 rpm for 60 minutes at room temperature. The mixtures were centrifuged at 3000 rpm for 15 minutes. The supernatant was collected, and its absorbance was measured using a UV-Vis spectrophotometer at the optimum wavelength. The adsorption efficiency of *Methyl Orange* in the solution and the equilibrium adsorption capacity were calculated using the following equations 1 and equations 2:

Adsorption Capacity:

$$Q_e = \frac{(C_0 - C_e) \times V}{m} \quad (1)$$

Adsorption Efficiency:

$$Q_e = \frac{C_0 - C_e}{C_0} \times 100\% \quad (2)$$

Notes:

q_e = adsorption capacity per unit mass of adsorbent (mg/g)

C_0 = initial concentration of solution (mg/L)

C_e = equilibrium concentration of solution (mg/L) V = volume of solution (mL)

m = mass of adsorbent used (g) Anggriani et al. (2021).

Optimization of Solution pH

A 50 mL solution of *Methyl Orange* with a concentration of 20 ppm was prepared in a 100 mL beaker, and the pH of the solution was adjusted to 2, 4, 6, 8, and 10 using 0,1 M NaOH and 0,1 M HCl solutions. The optimum adsorbent dosage of 0,75 g of *Acacia crassicarpa* bark powder was added to each solution. The mixtures were stirred at 150 rpm for 60 minutes at room temperature, then centrifuged at 3000 rpm for 15 minutes. The supernatant was collected,

and its absorbance was measured with a UV-Vis spectrophotometer. Adsorption efficiency and equilibrium capacity of *Methyl Orange* were then calculated using **Equations (1) and (2)**.

Optimization of Contact Time

A 20 ppm *Methyl Orange* solution was prepared in a volume of 50 mL at pH 2 in a 100 mL beaker. *Acacia crassicaarpa* bark powder with a dosage of 0,75 g was added to each solution. The mixtures were stirred using a magnetic stirrer at 150 rpm with varying contact times of 20, 40, 60, 80, and 100 minutes. The suspensions were then centrifuged at 3000 rpm for 15 minutes. The supernatant was collected, and the absorbance was measured using a UV-Vis spectrophotometer at the optimum wavelength.

Optimization of Stirring Speed

A 20 ppm *Methyl Orange* solution was prepared in a volume of 50 mL at pH 2 in a 100 mL beaker. *Acacia crassicaarpa* bark powder with a dosage of 0,75 g was added to each solution. The mixtures were stirred using a magnetic stirrer for 80 minutes at different stirring speeds of 100, 150, 200, and 250 rpm. The suspensions were then centrifuged at 3000 rpm for 15 minutes. The supernatant was collected, and the absorbance was measured using a UV-Vis spectrophotometer at the optimum wavelength.

Optimization of Adsorbate Concentration

Methyl Orange solutions with concentrations of 10, 20, 30, 40, and 50 ppm were each prepared in a volume of 50 mL at pH 2 in 100 mL beakers. *Acacia crassicaarpa* bark powder with an optimum dosage of 0,75 g was added to each solution. The mixtures were stirred using a magnetic stirrer for 80 minutes at a stirring speed of 200 rpm. The suspensions were then centrifuged at 3000 rpm for 15 minutes. The supernatant was collected, and the absorbance was measured using a UV-Vis spectrophotometer at the optimum wavelength and the adsorption capacity at equilibrium were calculated using **Equations 1 and 2**.

Optimization of Temperature

A 20 ppm *Methyl Orange* solution was prepared in a volume of 50 mL at pH 2 in a 100 mL beaker. *Acacia crassicaarpa* bark powder with a dosage of 0,75 g was added to each solution. The mixtures were stirred using a magnetic stirrer for 80 minutes at a stirring speed of 200 rpm with varying temperatures of 30, 40, and 50 °C (Dharmambal & Mani, 2015). The suspensions were then centrifuged at 3000 rpm for 15 minutes. The supernatant was collected, and the absorbance was measured using a UV-Vis spectrophotometer at the optimum wavelength.

Data Analysis

The obtained data were presented in the form of tables, graphs, and figures. All experiments were conducted in triplicate, and the results are presented as mean \pm standard deviation. Data from each adsorption parameter were statistically analyzed using a one-way ANOVA test followed by Duncan's multiple range test.

RESULTS AND DISCUSSION

SEM-EDS Characterization

The surface and elemental composition of *Acacia crassicaarpa* bark powder were analyzed using SEM at 5000 \times magnification. Before adsorption, the surface had open pores and was rough, while after adsorption, the pores were filled with dye., as illustrated in Figure 1.

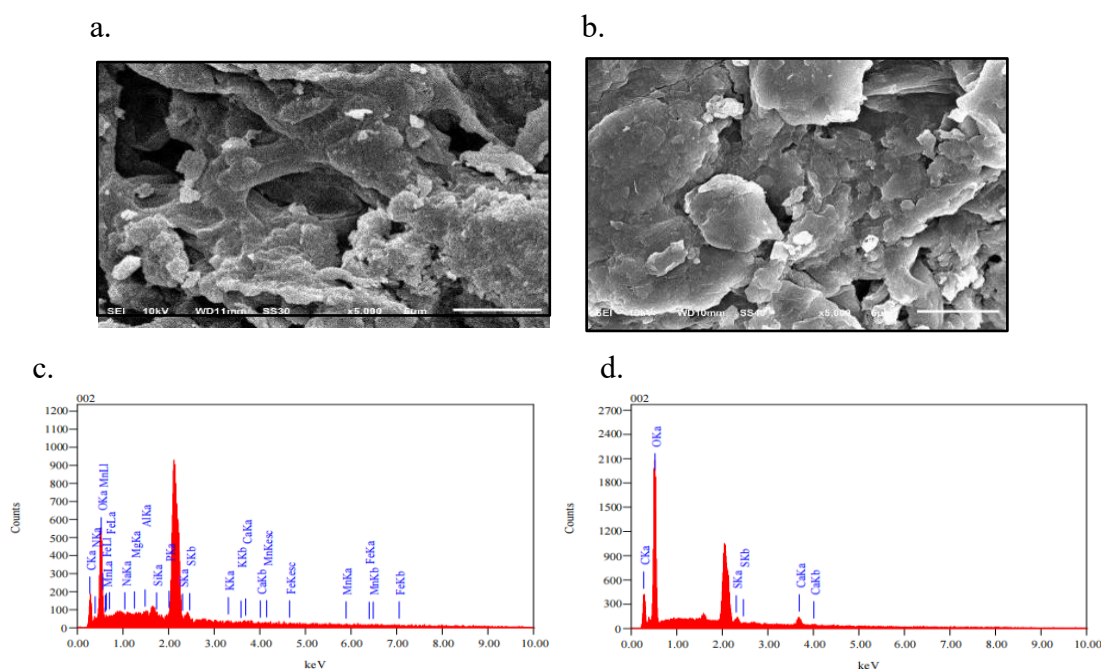


Figure 1. Shows the surface morphology of *Acacia crassicaarpa* bark powder at a magnification of 5000 \times **(a)** before adsorption, **(b)** after adsorption, **(c)** EDS spectrum before adsorption and **(d)** EDS spectrum after adsorption

Before adsorption, the bark powder had a rough, porous, and uneven surface, which helps in dye uptake. Since it was not physically or chemically activated, many pores were irregular or blocked by impurities. Lignocellulosic plant fibers show this rough texture (Yang et al., 2024). Figure 1 (b) shows the surface after adsorption with *Methyl Orange*, where the characteristic rough morphology was replaced by a smoother and more homogeneous surface.

This indicates the successful adsorption of *Methyl Orange* onto the *Acacia crassicaarpa* bark powder adsorbent. Figure 1 (c) shows the composition element of *Acacia crassicaarpa*.

Characterization using the Brunauer–Emmett–Teller (BET) method

The surface area, pore volume, and pore diameter of *Acacia crassicaarpa* bark powder adsorbent were analyzed using a Surface Area Analyzer (SAA), which provides information on pore characteristics and surface area based on the BJH (Barrett–Joyner–Halenda) and DFT (Density Functional Theory) methods. The results of the surface area, pore volume, and pore diameter analyses of the adsorbent are presented in Table 1.

Table 1. Results of surface area, pore volume, and pore diameter analysis of *Acacia crassicaarpa* bark powder adsorbent

Surface Area (m ² /g)	Mesopore Diameter (Å)	Total Pore Volume (cc/g)	Average Pore Diameter (Å)
0,460	<ul style="list-style-type: none"> • 32,707 (metode BJH adsorption) • 31,561 (metode BJH desorption) • 33,152-45,426 (metode DFT) 	4,361 x 10 ⁻³	379,008

BET analysis showed that *Acacia crassicaarpa* bark powder has a surface area of 0,460 m²/g, a pore volume of 4,361 × 10⁻³ cc/g, and an average pore diameter of 379,008 Å. The adsorbent has a low surface area and underdeveloped pores since it was not activated. BJH analysis gave mesopore diameters of 32,707 Å (adsorption) and 31,561 Å (desorption), while DFT showed pore sizes of 33,152–45,426 Å, indicating the presence of mesopores. The pore size distribution (32–45 Å) indicates the presence of mesopores according to IUPAC classification (20–500 Å). No significant micropore structure was observed.

Characterization using Fourier Transform Infrared (FTIR) Spectroscopy

The functional groups of *Acacia crassicaarpa* bark powder before and after adsorption were analyzed using Fourier Transform Infrared (FTIR) spectroscopy. The results before and after adsorption showed shifts in the wavenumbers, indicating the presence of N–H, O–H, C–H, C=O, C=C, and C–O functional groups, as shown in Figure 2.

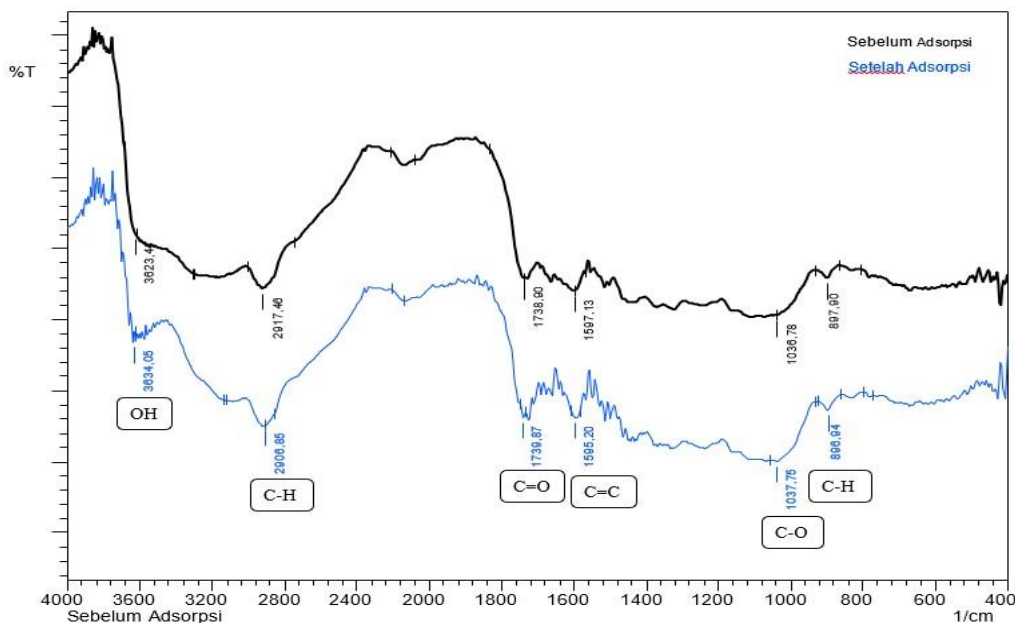


Figure 2. FTIR spectra of *Acacia crassicaarpa* bark powder before and after adsorption

Based on Figure 2, the FTIR spectra before and after adsorption indicate the presence of several functional groups. N–H amine vibrations ($3623\text{--}3634\text{ cm}^{-1}$) come from the bark and *Methyl Orange*. O–H stretching ($3301\text{--}3127\text{ cm}^{-1}$) indicates alcohol, phenol, and carboxylic acid groups, confirming cellulose presence. C–H stretching ($2917\text{--}2906\text{ cm}^{-1}$) comes from CH_2 in cellulose, C=O stretching ($1738\text{--}1739\text{ cm}^{-1}$) from hemicellulose, and C=C stretching ($1597\text{--}1595\text{ cm}^{-1}$) appears after adsorption. C–O stretching ($1036\text{--}1037\text{ cm}^{-1}$) is from ester, alcohol, ether, carboxylic acid, and phenolic groups, while C–H bending ($782\text{--}781\text{ cm}^{-1}$) is also observed. Shifts in the O–H and C=O stretching bands after adsorption suggest involvement of hydrogen bonding and electrostatic interactions in the adsorption mechanism (Aditama & Ardhyana, 2017).

Optimization of Adsorbent Dosage

The effect of adsorbent dose on the adsorption of *Methyl Orange* dye was obtained an adsorption efficiency of 65,56% and an adsorption capacity of 0,8759 mg/g. These results are presented in Figure 3.

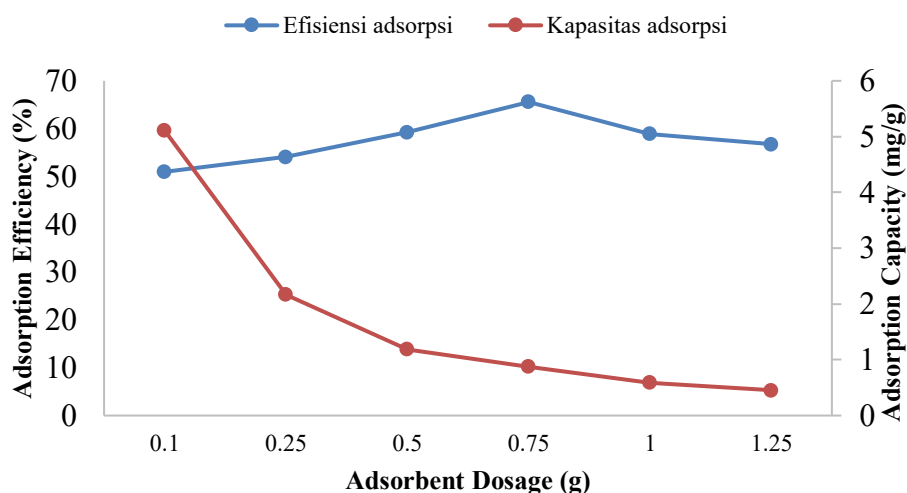


Figure 3. Graph of the effect of adsorbent dosage variation on the adsorption efficiency and adsorption capacity of Methyl Orange.

Optimization of Solution pH

The experimental results for different solution pH values of 2, 4, 6, 8, and 10 on the adsorption of *Methyl Orange* were obtained using an adsorbent dosage of 0,75 g, a contact time of 60 minutes, a stirring speed of 150 rpm, an initial adsorbate concentration of 20 ppm, and room temperature. The optimum pH was found to be 2, achieving an adsorption efficiency of 65,31% and an adsorption capacity of 0,8703 mg/g. These results are presented in Figure 4.

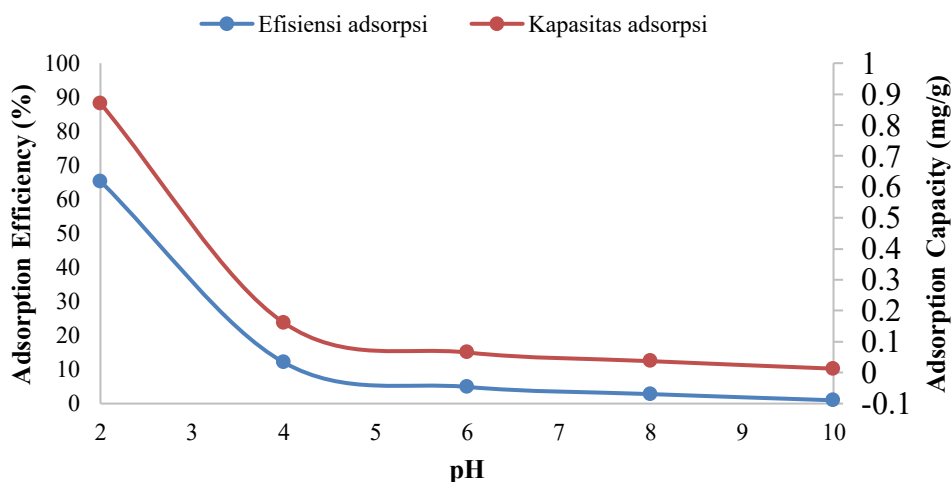


Figure 4. Graph of the effect of pH variation on the adsorption efficiency and adsorption capacity of Methyl Orange.

The adsorption efficiency of *Acacia crassicaarpa* bark powder toward *Methyl Orange* under different pH conditions showed the highest efficiency at pH 2, with an adsorption efficiency of 65,31% and an adsorption capacity of 0,8703 mg/g. The adsorption of *Acacia*

crassicarpa bark powder occurs mainly through electrostatic attraction. In acidic conditions, the adsorbent surface becomes positively charged due to protonation. This positive surface then attracts the negatively charged SO_3^- groups of *Methyl Orange*, allowing the dye to attach to the adsorbent.

Methyl Orange has a pKa of 3,5. At pH below 3,5, the amine group is protonated, so the dye exists in a neutral or cationic form. This protonation balances the negative charge of the sulfonate group under acidic conditions. At pH 4, adsorption efficiency was 12,11%, much lower than at pH 2. This is because *Methyl Orange* (pKa 3,5) is mostly anionic at pH above 3,5, causing electrostatic repulsion with the negatively charged adsorbent surface and reducing adsorption (Ghosh et al., 2020).

Optimization of Contact Time

The effect of contact time variation on the adsorption of *Methyl Orange* reached its optimum at 80 minutes, with an adsorption efficiency of 70.47% and an adsorption capacity of 4.6629 mg/g, as shown in Figure 5.

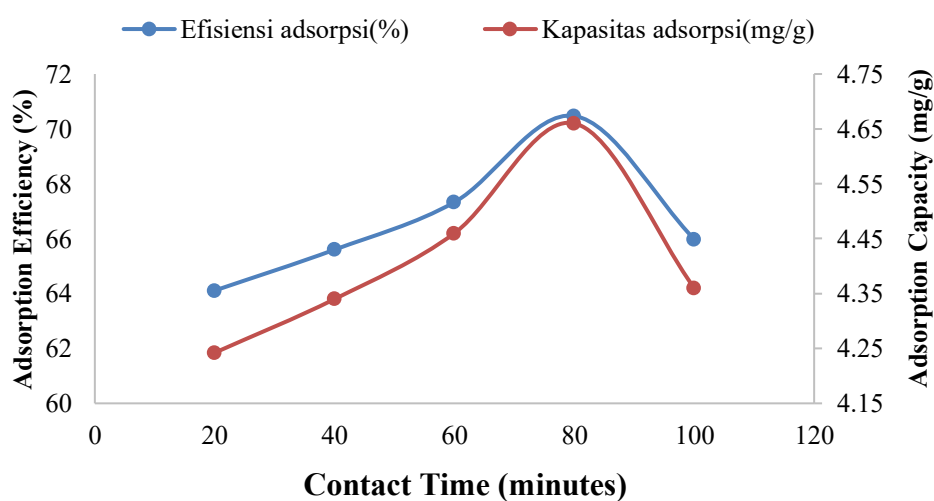


Figure 5. Effect of contact time variation on adsorption efficiency and adsorption capacity of *Methyl Orange*.

The effect of contact time on *Methyl Orange* adsorption was tested at 20, 40, 60, 80, and 100 minutes with 20 ppm dye, pH 2, 0,75 g adsorbent, and 150 rpm stirring. Adsorption increased at 20–60 minutes as the dye gradually filled the empty pores of the adsorbent. At 100 minutes, adsorption decreased to 65,96% (4,36492 mg/g) because most active sites were already occupied. The optimum contact time was 80 minutes, giving the highest adsorption efficiency. Adsorbate molecules attach to active sites and fill the adsorbent pores. Once

equilibrium is reached and the adsorbent is saturated, some molecules may be released, and longer contact time does not increase adsorption (Ikhsany et al., 2024).

Stirring Speed Optimization

The optimum stirring speed for *Methyl Orange* adsorption was 200 rpm, with 74.18% efficiency and 5,07663 mg/g capacity, as shown in Figure 6.

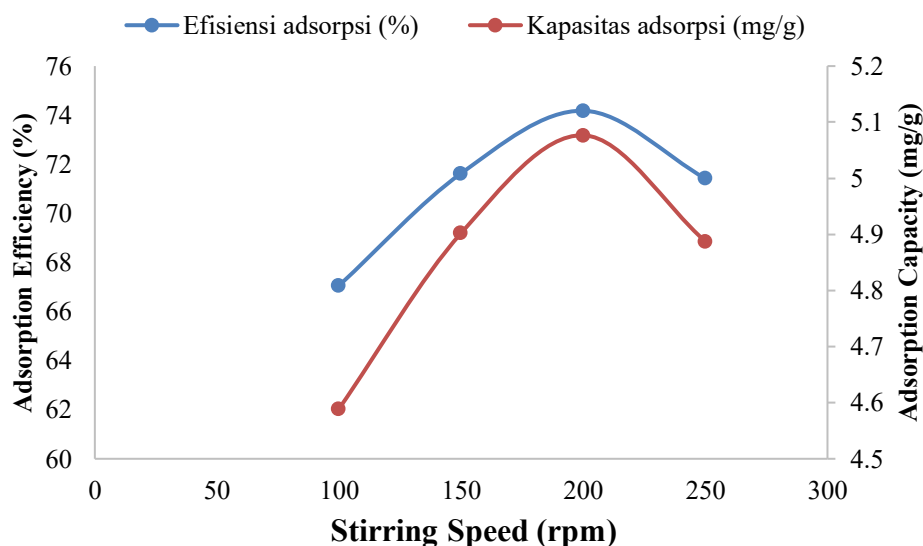


Figure 6. Effect of stirring speed variation on adsorption efficiency and adsorption capacity of *Methyl Orange*.

The effect of stirring speed on *Methyl Orange* adsorption was tested at 100, 150, 200, and 250 rpm with 20 ppm dye, pH 2, 0,75 g adsorbent, and 80 minutes contact time. At 100 rpm, adsorption was low because slow stirring reduced collisions between the dye and bark powder, limiting interaction and adsorption. Adsorption improved at 100–200 rpm due to better mixing, but dropped at 250 rpm because excessive stirring caused some dye to detach

Optimization of Adsorbate Concentration

The experimental data were obtained from variations in *Methyl Orange* concentrations of 10, 20, 30, 40, and 50 ppm using an adsorbent dosage of 0,75 g, at pH 2, a contact time of 80 minutes, a stirring speed of 250 rpm, and room temperature conditions. The results indicated that the optimum concentration was 20 ppm, yielding an adsorption efficiency of 80,58% and an adsorption capacity of 1,0763 mg/g. These findings are presented in Figure 8.

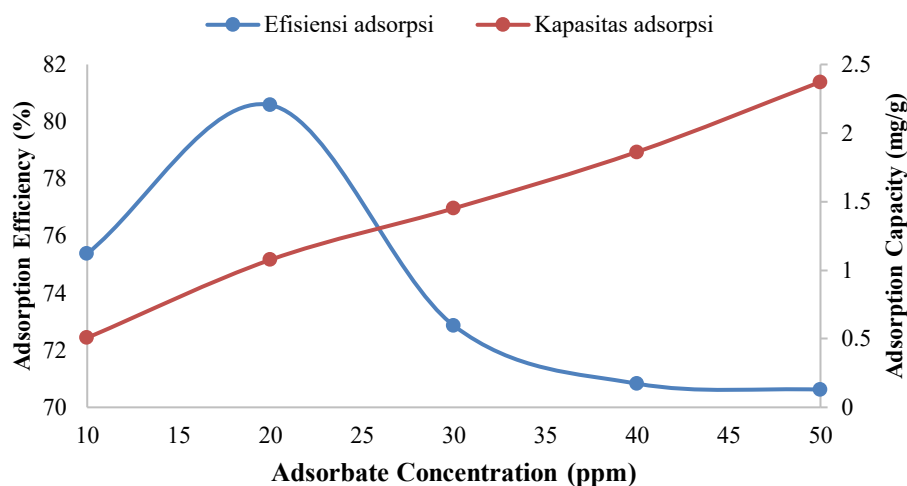


Figure 8. Graph of the effect of adsorbate concentration variation on the adsorption efficiency and adsorption capacity of Methyl Orange

Adsorption increased from 10 to 20 ppm as more dye molecules were available to bind to the adsorbent. At 30–50 ppm, adsorption decreased because the adsorbent quickly became saturated due to its small surface area. Adsorption efficiency (%) and adsorption capacity (mg/g) represent different aspects of the adsorption process. Efficiency indicates the percentage of adsorbate removed from the solution, while capacity reflects the amount of adsorbate adsorbed per unit mass of adsorbent. At higher adsorbate concentrations, the adsorption capacity (mg/g) increased due to the greater availability of dye molecules interacting with the adsorbent surface. However, the adsorption efficiency (%) decreased because the proportion of dye removed relative to the total initial concentration became smaller. This distinction highlights the importance of differentiating between adsorption capacity and efficiency when evaluating adsorbent performance.

Optimization of Temperature

Temperature variations of 30, 40, and 50 °C were employed to determine the optimum adsorption temperature using *Acacia crassicarpa* bark powder at the optimum dosage of 0,750 g and pH 2, with a *Methyl Orange* concentration of 20 ppm. The optimum temperature was found to be 30 °C, yielding an adsorption efficiency of 82,34% and an adsorption capacity of 5,6490 mg/g. These results are presented in Figure 9.

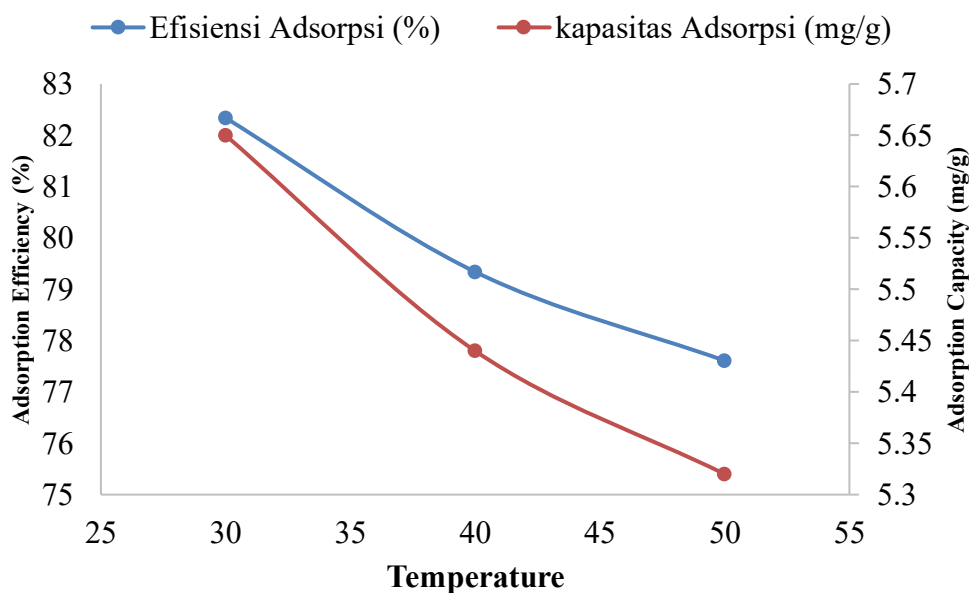


Figure 10. Graph of the relationship between temperature variation and the adsorption efficiency and adsorption capacity of Methyl Orange

The best adsorption performance was achieved at 30 °C, with an adsorption efficiency of 82,34% and an adsorption capacity of 5,6492 mg/g. Increasing the temperature up to 50 °C resulted in a decrease in *Methyl Orange* removal, there by reducing both adsorption efficiency and capacity in line with the temperature rise.

Adsorption decreased at higher temperatures because faster particle movement caused some dye to detach, showing the process is exothermic (Charismayani et al., 2017). Higher temperatures increase molecular motion, which can weaken interactions between the adsorbate and adsorbent, leading to desorption.

The adsorption capacity obtained in this study (~5.6 mg/g) is lower than that reported for activated *Acacia crassicarpa* bark (10–15 mg/g) in previous studies. This difference is attributed to the absence of activation treatment in this study, resulting in a significantly lower surface area (0.460 m²/g). Activation processes typically enhance pore development and increase surface area, thereby improving adsorption capacity. Nevertheless, the use of non-activated bark offers advantages in cost-effectiveness and environmental sustainability.

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CONCLUSION

The *Acacia crassicarpa* bark powder exhibited a porous and rough surface, mainly composed of carbon and oxygen. BET analysis indicated a mesoporous structure, while FTIR confirmed functional groups such as N–H, –OH, C–H, C=O, C=C, and C–O. The optimum adsorption was achieved at 0.75 g adsorbent, pH 2, 80 min contact time, 200 rpm stirring, 20 ppm adsorbate, and 30 °C, resulting in 82,34% efficiency and 5,6492 mg/g capacity. Despite its low surface area due to the absence of activation treatment, functional groups such as hydroxyl and carbonyl contributed to dye adsorption through electrostatic and hydrogen bonding interactions. Although the adsorption capacity was lower than that of activated materials, the use of untreated bark offers a cost-effective and environmentally friendly alternative.

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