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STUDY OF THE INFLUENCE OF VARIATIONS IN CATALYST MASS ON THE DEGRADATION OF METHYL ORANGE DYES BY ZnO/KJ-CTAB UNDER UV-LED ILLUMINATION

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ABSTRAK

Berkembangnya industri tekstil menyebabkan penggunaan zat warna semakin meningkat. Salah satu zat warna yang digunakan adalah metil jingga. Tujuan dari penelitian ini untuk mempelajari pengaruh massa katalis ZnO/Kulit Jeruk(KJ)-CTAB, mempelajari degradasi metil jingga (MJ), dan mempelajari isoterm adsorpsi. Penelitian ini menggunakan ZnO dan ZnO/KJ-CTAB yang dikarakterisasi dengan FTIR (*Fourier Transform Infra-Red)*, XRD (*X-Ray Diffractometer)*, BET (*Brunauer-Emmett-Teller surface area*), SEM (*Spectroscopy Scanning Elektron Microscopy)*, dan DR-UV *(Diffuse Reflectance-UV)*. Berdasarkan hasil penelitian, ZnO dan ZnO/KJ-CTAB termasuk struktur mesopori. Berdasarkan pengaruh vasiasi massa katalis yang dilakukan 2 perlakuan mode gelap dan mode terang, diketahui bahwa proses degradasi metil jingga dengan waktu optimum 30 menit dan konsentrasi 8 mg/L yang terbaik adalah ZnO/KJ-CTAB MT pada massa katalis 2 mg/ L dengan diperoleh penyisihan 93,85%, sedangkan untuk ZnO mode terang memperoleh removal 49%. Adsorpsi metil jingga oleh ZnO dan ZnO/KJ-CTAB mengikuti isoterm adsorpsi Langmuir, artinya proses degradasi pada metil jingga oleh ZnO dan ZnO/KJ-CTAB muncul pada situs permukaan yang homogen, sedangkan tidak ada interaksi antara molekul adsorbat dan molekul yang berdekatan.

ABSTRACT

The development of the textile industry caused the use of dyes to increase. One of the dyes used is methyl orange. This research aimed to study the effect of ZnO/Orange peel (KJ)-CTAB catalyst mass, the degradation of MJ absorption, and the adsorption isotherm. This study used ZnO and ZnO/KJ-CTAB, which were characterized by FTIR (Fourier Transform Infra-Red), XRD (X-Ray Diffractometer), BET (Brunauer-Emmett-Teller surface area), SEM (Spectroscopy Scanning Electron Microscopy), and DR-UV (Diffuse Reflectance-UV). Based on the research results, ZnO and ZnO/KJ-CTAB belong to mesoporous structures. Based on the effect of mass variation of the catalyst carried out by 2 dark mode and light mode treatments, it is known that the best degradation process for methyl orange with an optimum time of 30 minutes and a concentration of 8 mg/L is ZnO/KJ-CTAB MT at a catalyst mass of 2 mg/L—removal of 93.85%, while for ZnO light mode obtained 49% removal. The adsorption of methyl orange by ZnO and ZnO/KJ-CTAB follows the Langmuir adsorption isotherm, meaning that the adsorption process on methyl orange by ZnO and ZnO/KJ-CTAB appears on a homogeneous surface site. In contrast, there is no interaction between the adsorbate molecule and adjacent molecules.

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INTRODUCTION

Textile production requires chemicals and dyes. Along with the increase in the textile industry, the use of dyes has also increased by more than 700,000 tons, which are dissolved in waste. MJ (Methyl Orange) is a toxic dye that can disrupt the life of aquatic ecosystem organisms (Xin et al., 2015), and the presence of dangerous ingredients that can cause serious threats to the physicochemical properties of fresh water (Subbaiah & Kim, 2016).

Photocatalysis is an alternative method of waste processing because it is able to degrade surrounding compounds using light. This process is easy, reusable, and environmentally friendly. Photocatalysis is used to reduce dyes, which are used as catalyst activation. To speed up the process of destroying pollutants, toxic compounds can be overcome using photocatalyst materials (Baruah et al., 2013).

One of the semiconductor materials used in the photocatalysis process is ZnO because it has a band gap energy distance from the visible light region. Compared with TiO_2, ZnO has advantages, including high photocatalytic properties and surface reactivity. Apart from that, ZnO is able to work as a photocatalyst in the ultraviolet (UV) light range ($\lambda < 387$ nm, this is because ZnO has an Energy Gap (Eg) value ranging from 3.2 to 3.37 eV (Hakim & Haris, 2016). However, research needs to be carried out to increase the effectiveness of ZnO as a photocatalyst (Ghorbani et al., 2015). Adding a matrix or dopant can increase the photocatalytic activity. One of the natural materials that can be used as a dopant is orange peel (KJ). The addition of CTAB (Cetyltrimethylammonium bromide) is used as a medium for forming mesoporous structures and active compounds that can increase particle stability (Purbaningtias et al., 2019).

Based on the results of the presentation above, it is necessary to carry out research to analyze the degradation of MJ dye by ZnO/KJ CTAB. Modification of ZnO with KJ/CTAB is rarely studied, so this research is expected to contribute to efforts to degrade MJ by ZnO/KJ-CTAB

METHODS

Materials

The materials used in this research were orange peel, aqua DM (Bratachem), Zn acetate (Emsure ACS Supelco), NaOH (Merck), acetone (Merck), Whattman filter paper (42 Aschless diameter 90 mm, Ge Healthcare Life Sciences) aluminum foil, CTAB (Merck), MJ (Merck).

Tools

The tools used in this research were a set of laboratory glassware (Merck), magnetic stirrer (Merck), blender (Maspion), sieve (Standard Test Sieve 60 Mesh), mortal, Buchner vacuum filter, analytical balance (Us Solid Electronic Precision Balance), centrifuge (800 electric centrifuge), oven (Memmert), furnace (Thermolyne), IR spectrophotometer (Nicolet Avatar 360 IR), X-Ray Diffractometer (Bruker D2 Phaser), UV-Vis Spectrophotometer (Shimadzu UV – 2450), Spectroscopy Scanning Electron Microscopy (Phenom Desktop ProXL), and Brunauer-Emmett-Teller surface area (BELSORP-mini. BEL. Japan Inc).

Preparation

Orange Peel Preparation

50 grams of orange peel is washed thoroughly and dried in the sun for 2 days. The dried orange peel is crushed using a blender until smooth. After that, the finished orange peel powder is sieved using a 60-mesh sieve.

KJ-CTAB Modification

Five grams of orange peel powder were soaked in 100 mL of 1% CTAB solution (w/v) for 24 hours, and the mixed solution was filtered using a Buchner vacuum. The precipitate obtained was then dried in an oven at 75°C for 8 hours. The resulting dry sediment was crushed

until smooth and sieved using a 60-mesh sieve. The photodegradation results were tested using a UV-Vis Spectrophotometer instrument to determine the remaining.

Synthesis of ZnO/KJ-CTAB Using the Wet Impregnation Method

Orange peel powder-CTAB was added to a 0.1 M Zn acetate solution with a mass ratio of orange peel-CTAB to Zn solution of 1:25, then stirred using a stirrer for 1 hour. The solution was added with 0.1 M NaOH until the pH of the solution became 12 at a temperature of 60°C and stirred for 1 hour. After that, the solution obtained was left at room temperature for 24 hours. After being left to rest, the mixed sample was separated using a centrifuge at a speed of 3000 ppm for 30 minutes. After that, it was filtered using a Buchner, and the solid was washed with 100 mL acetone. The solid was dried using an oven at a temperature of 75°C for 6 hours. The dry solid obtained was crushed until smooth and then calcined for 4 hours at a temperature of 450°C. The synthesized material was characterized using FTIR, XRD, DR-UV, SEM, and BET.

Degradation of Methyl Orange by ZnO/KJ-CTAB

Degradation of methyl orange with ZnO/KJ-CTAB was done using the batch method, the light method (MT), and the dark method (MG). First, ZnO/KJ-CTAB was weighed with a mass variation of 0.002, 0.004, 0.006, 0.008, 0.001, 0.012, 0.014, 0.016 grams then added MJ with a concentration of 8 ppm for 25 mL. The solution mixture was put into the UV reactor while stirring with a magnetic stirrer for 30 minutes. After that, the solution was separated using a centrifuge at a speed of 3000 rpm for 10 minutes. Then, the absorbance of the filtrate obtained was measured using a UV-VIS spectrophotometer at a wavelength of 465 nm.

RESULT AND DISCUSSION

FTIR analysis

To analyze the functional groups in ZnO with ZnO/KJ-CTAB, characterization using an FTIR instrument was carried out. FTIR analysis utilizes a sample molecule's adsorption of infrared (IR) waves. This radiation energy can only excite a sample molecule, so only covalent bond vibrations occur between functional groups and atoms in the sample (Laxmi, 2012). Figure 1 shows the spectra result.

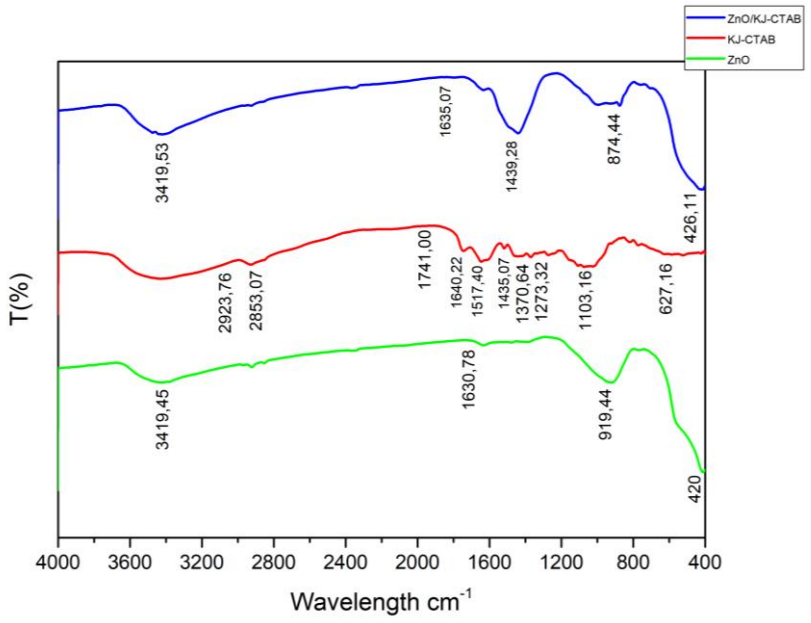

Figure 1. FT-IR spectra of ZnO, KJ-CTAB and ZnO/KJ-CTAB

FTIR analysis of ZnO, KJ-CTAB, and ZnO/KJ-CTAB was carried out in the wave number range 4000-400cm^{-1}. As shown in Figure 1, in the ZnO spectra, there is an

absorption band in the 500-400cm^{-1} area, indicating bond tension between Zn-O (Rosanti, et al., 2022a). The KJ-CTAB spectra show that orange peel has been successfully modified with CTAB. The typical absorption of CTAB is seen in peaks 2923cm^{-1} and 2853cm^{-1}, which indicate the stretching vibration of C-CH_2 in the CTAB methylene chain. The strong and wide absorption at a wave number of around 3425cm^{-1} indicates the stretching of the O-H hydrogen bond from the hydroxy group (Duarte et al., 2017). Peaks at wave numbers around 1741cm^{-1} and 1640cm^{-1} indicate the presence of C=O and C=C bond strain absorption (Zapata et al., 2009). Absorption at waves 1435cm^{-1} and 1370cm^{-1} is the absorption characteristic of bending methylene and methyl groups in lignocellulose in orange peel (Zapata et al., 2009). Based on the FTIR spectra in Figure 1 (ZnO/KJ-CTAB), it can be seen that the ZnO/KJ-CTAB synthesis process has been successfully carried out. Based on the FTIR spectra, the synthesized ZnO/KJ-CTAB composite is clean of orange peel and CTAB residues, as indicated by the absence of absorption peaks from orange peel and CTAB in the ZnO/KJCTAB composite.

XRD analysis

Characterization using an XRD instrument aims to strengthen the identification results and determine the ZnO/KJ-CTAB phase that is formed by looking at the 2θ angle that appears.

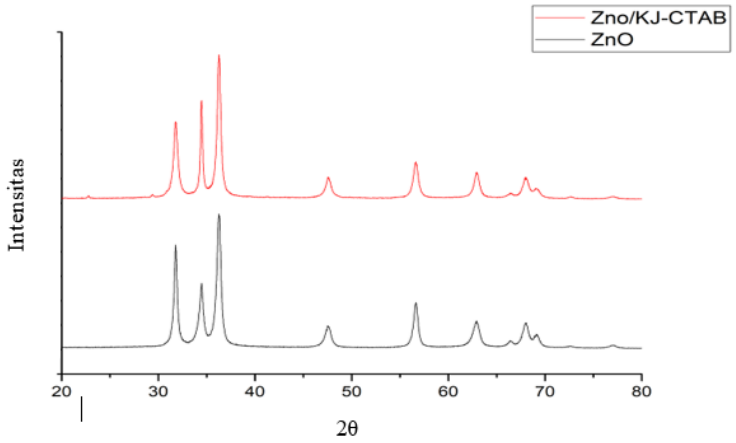

Figure 2. Diffractograms of ZnO and ZnO/KJ-CTAB

Based on the XRD results in Figure 2 show that there is no difference in the results between the diffraction patterns of ZnO and ZnO/KJ-CTAB. The following XRD test results for ZnO and ZnO/KJ-CTAB can be seen in Table 1.

Table 1. XRD test results of ZnO and ZnO/KJ-CTAB

Material	2θ	d(Å)	Crystal Form	Crystal Structure	%Crystallinity
ZnO	31,798	2,811	Heksagonal	*Wurtzite*	98
	34,475	2,601			
	36,281	2,475			
ZnO/KJ-CTAB	31,786	2,812	Heksagonal	*Wurtzite*	96
	34,461	2,600			
	36,296	2,473			

Based on Figure 2 and Table 1, the synthesis results contain a relatively large 2θ spectral peak in the 36˚ region. This peak appears in both ZnO and ZnO/KJ-CTAB with a diffraction field of d101 (2.4Å). Other peaks that have relatively greater intensity are at peaks 34˚ and 31˚ with diffraction fields d100 (2.8 Å) and d002 (2.6 Å). Form a hexagonal structure

with a wurtzite shape and obtain the angular position of the diffraction peak in the region 2θ = 31.75˚, 34.40˚; and 36.22˚ with a value of 2θ = 36-69˚ showing a typical ZnO peak. At this position, the characteristic ZnO diffraction pattern in the *hkl* (101) plane has the highest intensity (Rosanti, et al., 2022a). This is also proven by comparing JCPDS data with XRD data, which shows that in ZnO and ZnO/KJ-CTAB, a wurtzite crystal structure with a hexagonal shape is formed.

Based on Figure 2, it is known that adding KJ/CTAB to ZnO does not damage the structure of ZnO. However, it can cause crystallinity to decrease. The smaller crystallinity causes the crystal structure to be irregular, which can increase photocatalytic activity.

DR-UV analysis

Characterization uses DR-UV to determine the absorbance and band gap width of ZnO and ZnO/KJ-CTAB. The synthesis results show the wavelength with visible light range to the absorbance value

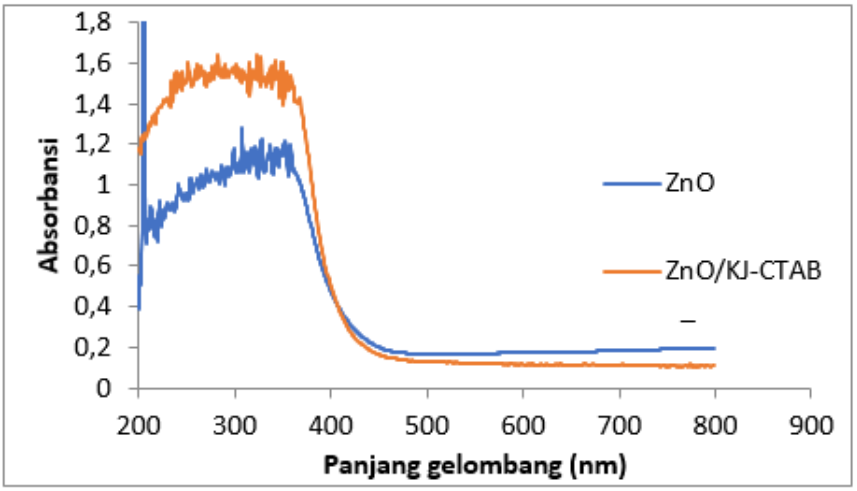

Figure 3. DR-UV spectra of ZnO and ZnO/KJ-CTAB

Based on Figure 3, it shows that the addition of KJ-CTAB to ZnO causes the absorption area to shift towards a larger wavelength. This is because the addition of ZnO/KJ-CTAB causes the energy band gap to narrow so that the absorption shifts towards the UV. Gap energy is needed for excitation and moves to the conduction band. If the band gap energy becomes smaller, the energy required for photocatalysis to excite electrons from the valence band to the conduction band will also become smaller. The energy gap value obtained from the synthesis of ZnO is 3.25 eV and produces a wavelength that shifts to visible light of 381 nm, while for ZnO/KJ-CTAB, the value obtained is 3.20 eV and produces a wavelength that shifts to visible light of 387 nm. The energy gap value of ZnO/KJ-CTAB is smaller than ZnO. The smaller the energy required for electron excitation, the smaller the band gap (Rosanti et al., 2020; Rosanti, et al., 2022a).

BET analysis

BET characterization was used to determine the pore size and surface area of ZnO and ZnO/KJ-CTAB. The characterization results using BET are shown in Table 2.

Table 2. Results of BET analysis of ZnO and ZnO/KJ-CTAB

Material	Surface Area (m2/g)	Pore volume (cm3/g)	Pore Size (nm)
ZnO	29,780	0,059	3,899
ZnO/KJ-CTAB	36,710	0,083	4,634

Based on Table 2, ZnO's surface area, pore volume, and pore size are smaller than ZnO/KJ-CTAB. Based on pore size, porous materials can be grouped into three classes, namely microporous materials (pore size <2 nm), mesoporous (pore size 2-50 nm), and macroporous (pore size >50 nm). ZnO and ZnO/KJ-CTAB can be classified as mesoporous pore size-types because they have pore sizes of more than 2 nm, namely 3.899 nm and 4.634 nm. CTAB surfactant is able to induce the formation of mesopores, which can be used for polymers and other large compounds to produce more particle dispersion. The decrease in diffraction intensity in ZnO with ZnO/KJ-CTAB is also related to the increase in surface area of ZnO/KJ-CTAB particles, which causes a decrease in crystallinity, thereby increasing the photocatalytic properties.

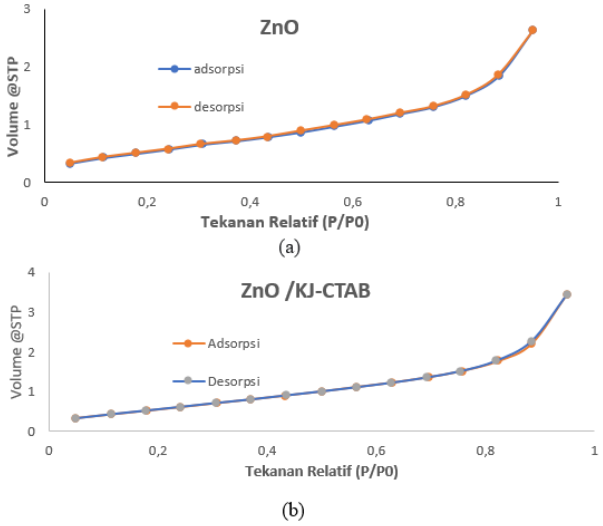

Figure 4. Adsorption-desorption graph of a) ZnO b) ZnO/KJ-CTAB

Figure 4 shows that ZnO and ZnO/KJ-CTAB are type III isothermal curves, which means they experience multilayer adsorption. The catalyst quantity is higher when the relative pressure increases. At the same pressure, there is a difference in the amount of adsorbed and desorbed gas, resulting in a hysteresis loop. formed. This indicates that the synthesized ZnO and ZnO/KJ-CTAB materials have meso pore sizes (Wellia et al., 2019)

SEM analysis

Scanning Electron Microscope (SEM) was carried out to determine the surface morphology of ZnO and ZnO/KJ-CTAB. The test was carried out at 10,000x magnification.

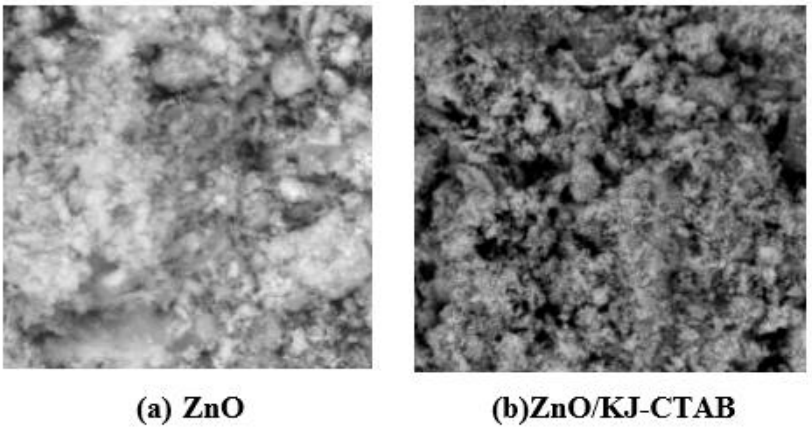

Figure 5. SEM results (a) ZnO (b) ZnO/KJ-CTAB

Based on the SEM test shown in Figure 5, there are clear morphological differences in the surface of the adsorbent. As seen in Figure 5. (a), The morphology of the ZnO looks lumpy

and not hollow, which shows that the morphology of the ZnO particles is round and sticks together with each other. According to Hossein et al. (2015), the morphology of ZnO is stuck together and has a spherical shape, whereas in Figure 5. (b) the ZnO/KJ-CTAB particles appear smaller, homogeneous, and separated due to the presence of CTAB surfactant as a mesoporous template. This is in accordance with the IR results, which state that no uptake from CTAB was stated. ZnO/KJ-CTAB has a larger pore size and has a mesoporous structure, so the surface area is more specific. This is proven by the results of the BET analysis, which shows that the surface area of ZnO/KJ-CTAB is greater than ZnO. These surface characteristics will result in a higher adsorption capacity (Feng & Guo, 2012).

Adsorption Isotherm

The adsorption isotherm shows the relationship between the concentration of solute absorbed per unit weight of the adsorbent and the concentration of the adsorbate in a certain amount at a temperature under equilibrium conditions (Subbaiah & Kim, 2016).

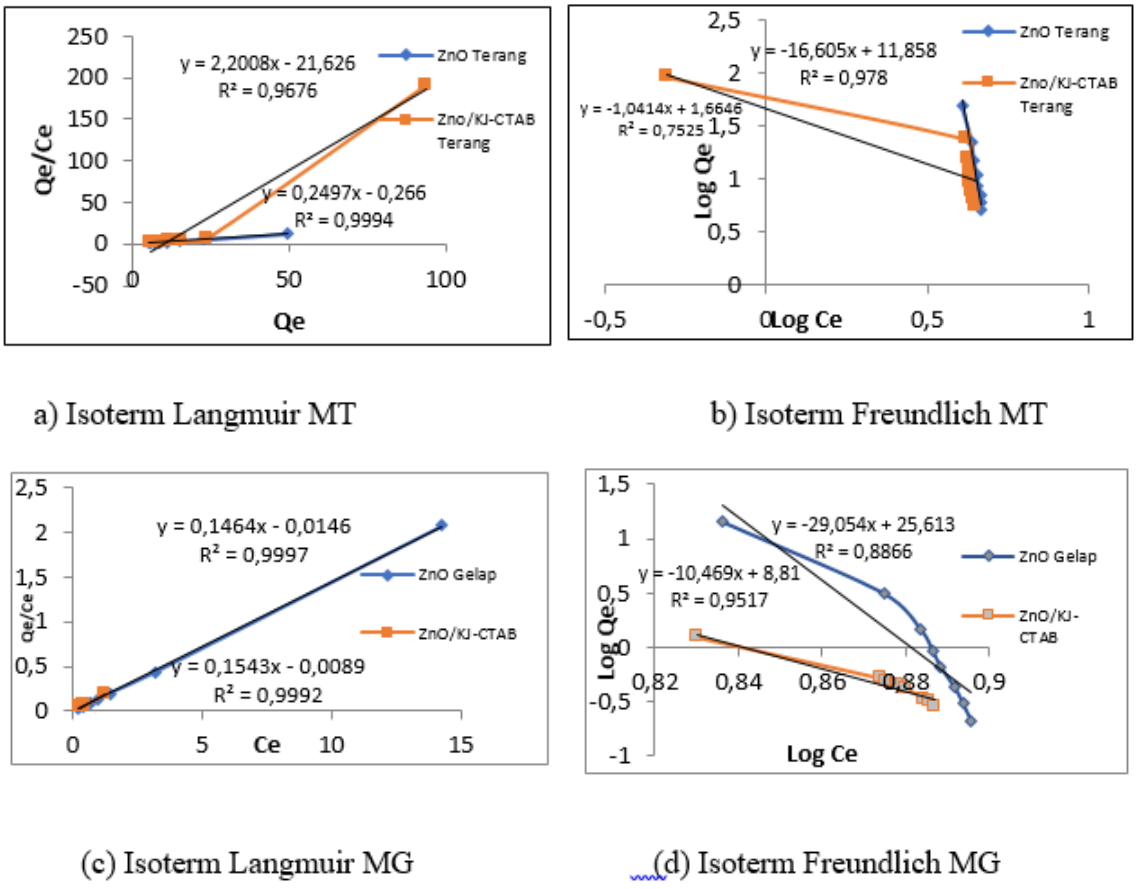

Figure 6. Graph of MT and MG degradation isotherms

Based on Figure 6 above, it can be concluded that the appropriate adsorption isotherm model for ZnO and ZnO/KJ-CTAB in MJ adsorption is the Langmuir isotherm. This is indicated by the R^2 value of ZnO and ZnO/KJ-CTAB MT and MG, which is close to one. The Langmuir isotherm model is suitable for orange peel adsorbent, which is capable of forming a monolayer of adsorbate on the outer surface of the adsorbent and no further adsorption (Munagapati & Kim, 2016).

Table 3. Equations and Correlation Coefficient Values for Adsorption Isotherm Modeling

Sampel	Isoterm Langmuir			Isoterm Freundlich		
	R_L	Qmaksimal	R^2	K_F	n	R^2
ZnO+MJ (MT)	-1,000	1,06	0,9994	7,21	0,06	0,978
ZnO/KJ-CTAB+MJ (MT)	-0.060	9,82	0,9676	46,19	0,960	0,7525

Sampel	Isoterm Langmuir			Isoterm Freundlich		
	R_L	Qmaksimal	R^2	K_F	n	R^2
ZnO+MJ (MG)	-5,8	0,09	0,9997	4,10 $X10^{25}$	0,03	0,8866
ZnO/KJ-CTAB+MJ (MG)	-4,34	0,057	0,9992	64 $X\ 10^7$	0,09	0,9517

Methyl Orange photodegradation test with variations in catalyst mass

The photodegradation activity of MJ by varying catalyst mass can be determined by calculating the removal efficiency using equation (1) (Rosanti, et al., 2022a; Rosanti, et al., 2022b):

$$Removal\ Efficiency\ (\%) = \frac{C_0 - C_t}{C_0} x100\% \quad (1)$$

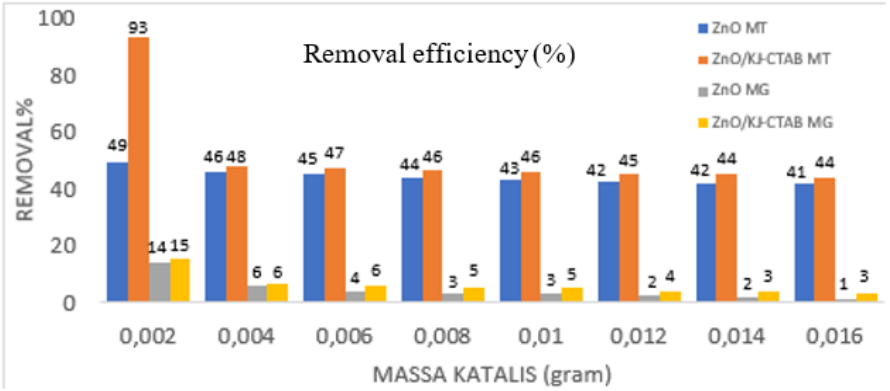

Figure 7. Removal efficiency (% removal) of ZnO and ZnO/ KJ-CTAB

Based on Figure 7. at a mass of 0.002 grams, shows that the difference between ZnO/KJ-CTAB MT is (93%) compared to ZnO (49%) MT, while in MG, the efficiency obtained is (15.46%) for ZnO/KJ-CTAB and for ZnO (14.2%). Based on the results above, ZnO/KJ-CTAB tends to be more effective in absorbing MJ. This is in accordance with the results of XRD, BET, and DR-UV characterization that ZnO/KJ-CTAB has a large surface area, which leads to a mesoporous structure, and The band gap value is smaller than ZnO so that it can increase photocatalytic activity (Rosanti et al., 2022a; Rosanti et al., 2022b).

The removal efficiency decreases with increasing catalyst mass at a mass of 0.004 grams—0.016 grams. This is due to the interaction of photocatalytic material particles, such as aggregation, which decreases the photocatalytic material's total surface area and increases the diffusion path's length (Rosanti, 2014; Atsabiti, 2019).

CONCLUSION

The optimum catalyst mass for MJ removal for ZnO and ZnO KJ-CTAB was 0.002 g with an optimum time of 30 minutes, and the concentration used was 8 mg/L with a removal efficiency for ZnO/KJ-CTAB of 93.85%, while for ZnO it was 49%. The adsorption isotherm modeling used for good MJ adsorption for ZnO and ZnO/KJ-CTAB is the Langmuir isotherm by obtaining linear regression R^2 of 0.9994 and 0.9960, respectively.

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