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Research Article

Adsorption of Ciprofloxacyn by Plate-Like $Bi_4Ti_3O_{12}$

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ABSTRACT

Antibiotic compound waste has attracted significant attention because of its harmful effects on aquatic environments. One of the methods reported as a potential approach for antibiotic wastewater treatment is adsorption. Plate-like $Bi_4Ti_3O_{12}$ is a triple-layer Aurivillius compound reported to exhibit adsorption properties, although research remains limited. Therefore, we synthesized plate-like $Bi_4Ti_3O_{12}$ using the molten salt method. The adsorption test results showed that plate-like $Bi_4Ti_3O_{12}$ reduced ciprofloxacin concentration by ~51% within 120 minutes. It shows that plate-like $Bi_4Ti_3O_{12}$ has the excellent capability to adsorb the antibiotic compound ciprofloxacin.

Keywords: Ciprofloxacin, Adsorption, Plate-like $Bi_4Ti_3O_{12}$

1. Introduction

Antibiotic residues in aquatic environments have become a serious concern because it dangerous and can lead to the emergence of antibiotic-resistant bacteria [1]-[3]. Ciprofloxacin is a type of fluoroquinolone antibiotic and has been reported as one of the antibiotic residues frequently found in aquatic environments, thus requiring special attention for its handling [4]. Many techniques have been developed to reduce antibiotic pollution, such as photocatalyst, Flocculation, and adsorption [5]-[7]. One of the methods reported for treating antibiotic waste is the adsorption method. Several materials reported as adsorbents for ciprofloxacin include fly ash and active carbon [8][9].

$Bi_4Ti_3O_{12}$ is a member of the Aurivillius compound family that has been reported to possess several interesting properties, one of which is its potential use as a photocatalytic material [10]. Several researchers have already reported the adsorption properties of Aurivillius compounds. Bashofi and Prasetyo (2023) reported that the $Bi_4Ti_3O_{12}$ compound could adsorb rhodamine B dye by 16.37% for 30 minutes [11]. On the other hand, Saputra et al. (2024) also reported that the four-layer Aurivillius compound $CaBi_4Ti_4O_{15}$ was able to adsorb the antibiotic compound ciprofloxacin by 9.24% within 30 minutes [12]. Meanwhile, Hikmah and Prasetyo (2025) also reported that the plate-like Fe-doped $Bi_4Ti_4O_{15}$ compound ($Bi_4Ti_{2.9}Fe_{0.1}O_{12}$) was able to adsorb ciprofloxacin by ~55% for 120 minutes [13]. It indicated that plate-like Aurivillius compounds possess adsorbent properties; however, studies in this area are still limited, making it important to conduct further research on this property.

Several factors influence adsorption, like particle size and particle agglomeration [14][15]. One of the synthesis methods known to produce particles with uniform size and minimal agglomeration is molten salt synthesis [16]. Several researchers have reported the synthesis of plate-like $Bi_4Ti_3O_{12}$ compounds using the molten salt synthesis method. Prasetyo et al. (2022) synthesized $Bi_4Ti_3O_{12}$ compounds using the molten salt method and obtained plate-like particles with no agglomeration [17]. In addition, plate-like Aurivillius materials have been reported to exhibit good photocatalytic activity; however, studies on their adsorption properties are still limited [18]. Therefore, the study of the adsorption properties of plate-like Aurivillius materials synthesized via molten salt synthesis becomes important. In this study, $Bi_4Ti_3O_{12}$ was synthesized using the molten salt method and subsequently used as an adsorbent material for ciprofloxacin antibiotics.

2. Materials and Methods

2.1 *Materials*

The materials used in this research were TiO_2 (Sigma-Aldrich, 99.9%), Bi_2O_3 (Himedia, 99.9%), NaCl (Merck, 99.5% powder), KCl (Merck, 99.5% powder), $AgNO_3$ (Merck), acetone, distilled water, and ciprofloxacin.

2.2 *Synthesis*

In this study, the molar ratio of the product compound ($Bi_4Ti_3O_{12}$) to the NaCl/KCl salt mixture was 1:7. The required amounts of precursors and salts were calculated stoichiometrically, with the target compound ($Bi_4Ti_3O_{12}$) being 3 grams. The synthesis was done by grinding the precursor with a NaCl/KCl salt mixture using an agate mortar for 1 hour. The precursor and NaCl/KCl salt mixture were then calcined in stages at 750 and 820°C, respectively, for 6 hours. The residual NaCl/KCl salt was removed by washing it with hot distilled water, and the presence of residual salt could be checked using the $AgNO_3$ [13].

2.3 *Characterization*

The synthesized product was characterized using several instruments, namely: (a) *X*-ray diffraction (XRD) to determine the phases of the formed compounds, (b) Scanning electron microscopy (SEM) to observe particle morphology, and (c) ultraviolet-visible diffuse reflectance spectroscopy (UV-Vis DRS) to obtain the reflectance spectrum, which was then analyzed using the Kubelka-Munk equation to determine the band gap energy.

2.4 *Adsorption Test*

100 mL of ciprofloxacin solution (8 ppm) was mixed with the $Bi_4Ti_3O_{12}$ catalyst and stirred with a stirrer for 120 minutes. After the process, the mixture was filtered and measured using a UV-Vis spectrophotometer to calculate the absorbance value (concentration).

3. Result and Discussion

The diffractogram of the synthesized compound is shown in **Figure 1** and was compared with the standard data of $Bi_4Ti_3O_{12}$ from the Inorganic Crystal Structure Database (ICSD) No. 87808 (orthorhombic crystal structure with space group B2*ab*). The comparison results show that the diffractogram of the synthesized compound matches well, indicating that the synthesis of $Bi_4Ti_3O_{12}$ was successful. The characteristic peaks of $Bi_4Ti_3O_{12}$ were found at 2*θ* (°): 23.32, 30.1, 32.86, and 33.08. It showed that the synthesis conditions (type of salt, temperature, and calcination time) successfully facilitated the reaction between the precursors.

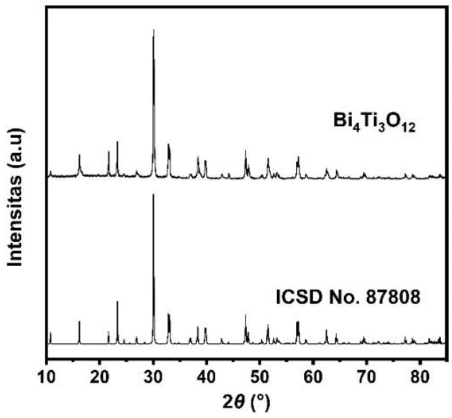

Figure 1. Diffractogram of product sample

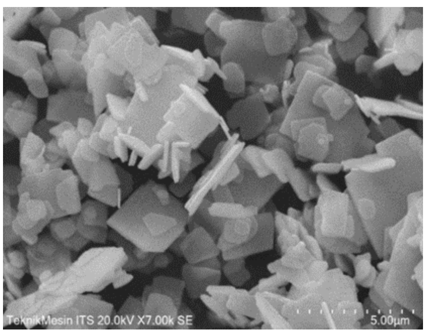

Figure 2. SEM Image of sample product

The micrograph of the $Bi_4Ti_3O_{12}$ compound is shown in **Figure 2**, and it can be observed that the product has a thin plate-like particle morphology with non-uniform sizes. The obtained morphology is consistent with the report by Prasetyo et al. (2022) and is a characteristic form of Aurivillius compound particle [17]. The particle growth mechanism in molten salt synthesis consists of two stages: (a) nucleation and crystal growth, and (b) particle growth. The particle size obtained is strongly influenced during the particle growth stage [19].

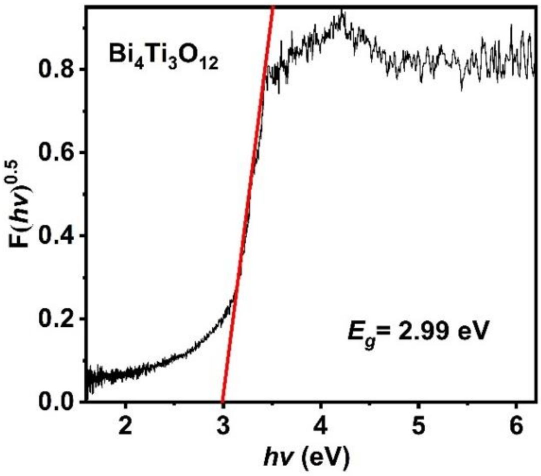

Figure 3. Plot tauc of product sample

The reflectance spectrum of the sample is shown in **Figure 3** and was further analyzed using the Kubelka-Munk equation to obtain the band gap energy. The calculation results show that the band gap energy of $Bi_4Ti_3O_{12}$ is 2.99 eV (414.9 nm). This band gap energy plays an important role when the material is tested for photocatalytic properties, as the process involves electronic transitions from the valence band in the Bi-6*s* + O-2*p* orbitals to the conduction band in the Ti-3*d* orbitals [11].

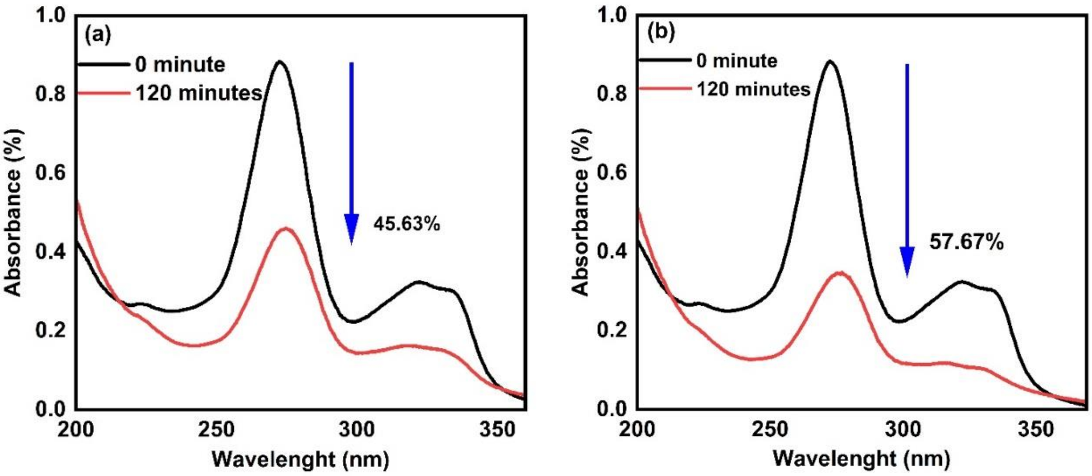

Figure 4. Adsorption test: (a) 1st and (b) 2nd test

The adsorption test of ciprofloxacin solution by $Bi_4Ti_3O_{12}$ was carried out for 120 minutes and repeated twice. **Figure 4** shows the UV-Vis spectra of the adsorption test, and it can be observed that there was a significant decrease in absorbance values, indicating a reduction in ciprofloxacin concentration of 45.63 and 57.67% within 120 minutes. It shows that the compound possesses adsorption capability. The adsorption test results also serve as an initial indication that plate-like Aurivillius compounds have good adsorption capability." Furthermore, studies on the adsorption capability of ciprofloxacin by $Bi_4Ti_3O_{12}$ have never been reported, making these results important for future investigations.The studies on the adsorption properties of Aurivillius compounds are still limited. However, by referring to the structural model of perovskite compounds, the interaction between the adsorbent ($Bi_4Ti_3O_{12}$) and the adsorbate (ciprofloxacin) may involve electrostatic interactions (physisorption) [20].

4. Conclusion

The plate-like $Bi_4Ti_3O_{12}$ has been successfully synthesized using the molten salt method. The adsorption test results showed that plate-like $Bi_4Ti_3O_{12}$ reduced the concentration of ciprofloxacin by ~51% within 120 minutes. It indicates that palte-like $Bi_4Ti_3O_{12}$ has very good adsorption capability for antibiotic ciprofloxacin compounds. However, studies on the adsorption properties of plate-like $Bi_4Ti_3O_{12}$ are still limited; therefore, further research on this property is required.

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