

# Methylene Blue Adsorption using Magnetite-Modified Tea Waste Adsorbent

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## Received

28 April 2025

## Received in revised form

9 June 2025

## Accepted

13 June 2025

## Published online

30 June 2025

## DOI

<https://doi.org/10.56425/jr1w4h95>



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## Abstract

Methylene blue (MB) is a hazardous waste for living things and the environment. In this study, MB adsorption was carried out using tea waste as an adsorbent. To enhance the adsorption process, activation with HCl and modification with magnetite ( $\text{Fe}_3\text{O}_4$ ) were carried out. The purpose of this study was to determine the effectiveness of using tea waste adsorbent activated and modified with  $\text{Fe}_3\text{O}_4$  on methylene blue waste. Tea waste was activated with HCl and modified with  $\text{Fe}_3\text{O}_4$  using the coprecipitation method. Two variations of testing were conducted, namely, the variation of pH and contact time. The adsorbent was characterized using Fourier transform infrared and scanning electron microscopy. The results showed that the optimum pH at pH 10 with an adsorption power of 98.4% and a contact time of 90 minutes with an adsorption power of 98.6%, when the adsorbent was applied directly to the methylene blue waste produced an adsorption power of 99%, and an adsorption isotherm model that was suitable using Freundlich isotherm with an  $R^2$  value of 0.9704.

**Keywords:** adsorbents, methylene blue, magnetite, tea waste

## 1. Introduction

Many industries are generating a lot of waste, which increases various environmental problems. The environmental problems occur because the industrial waste disposal process is carried out without waste treatment [1]. The industrial waste consists of dyes and heavy metals. Methylene blue is one of the wastes used in industry. Methylene blue waste is not only produced by industry, but also in laboratories, one of which is in the research laboratory at the State University of Jakarta. Methylene blue is a carcinogenic waste that can cause environmental pollution and harm human health [2]. Methylene blue waste discharged into the water will cause water pollution because water is an essential element for the survival of living beings, so it can damage aquatic biota

and endanger human health when the water is consumed [4]. So, it is necessary to process methylene blue waste.

Various types of processing methods have been widely developed through physical, chemical, and biological approaches [5]. One of the treatment methods that can be done is the adsorption method. Adsorption is a process of bonding two surfaces between a substance in the form of a liquid or gas to a solid [6]. The adsorption method is an effective, easy-to-do, practical, and environmentally friendly method [7]. In the adsorption method, an adsorbent is needed to adsorb methylene blue waste. Adsorbent is a solid substance that functions to remove pollutants from waste [8]. The commonly used adsorbent is activated carbon, because activated carbon has a high carbon content, a porous structure, and a large surface area [9]. But the use of activated carbon requires an

expensive cost [10]. To overcome this weakness, adsorbents were developed using natural materials or biomass.

Biomass adsorbents come from agricultural, forestry, and fiber waste. Using biomass as an adsorbent is beneficial because it is environmentally friendly, easy to find, and can reduce environmental pollution [11]. One of the sources that can be used is tea waste. Tea waste has lignocellulose deposits that contain hydroxyl and carboxyl groups that have the potential to adsorb waste [12]. The adsorption capacity can be increased by activating using strong acid or strong base activators like KOH, ZnCl<sub>2</sub>, H<sub>3</sub>PO<sub>4</sub>, and H<sub>2</sub>SO<sub>4</sub> [13]. One of the activators that can be used is HCl.

The activator can remove impurities that clog the pores, so that the pores are more open and the adsorption capacity becomes larger [14]. In addition to activation, making modifications using magnetite (Fe<sub>3</sub>O<sub>4</sub>) can also increase adsorption capacity, due to the large surface area and the presence of new functional groups [15]. Modification with Fe<sub>3</sub>O<sub>4</sub> can also facilitate the process of separating adsorbents from waste after adsorption because of their magnetic properties that can adhere to magnetic fields [16]. The use of Fe<sub>3</sub>O<sub>4</sub> material as an adsorbent modification has also been proven before when used in activated carbon and corn cobs [17][18].

In previous studies, the adsorption process of methylene blue using modified tea waste adsorbent with Fe<sub>3</sub>O<sub>4</sub> material has been carried out. The results of the study showed that the modification carried out was effective in adsorbing methylene blue [19]. Meanwhile, to enhance the adsorption capacity, a chemical activation process of tea wastes using HCl will be carried out before modification using Fe<sub>3</sub>O<sub>4</sub>. The research problems discussed in this article are whether the activation process carried out provides adsorption results that remain effective on methylene blue and how the results of direct application of the adsorbent to methylene blue waste.

## 2. Materials and Methods

### 2.1 Materials

The materials used in this research were tea waste as an adsorbent component, iron(III) sulfate heptahydrate (FeSO<sub>4</sub>·7H<sub>2</sub>O), iron(III) chloride hexahydrate (FeCl<sub>3</sub>·6H<sub>2</sub>O), ammonia (NH<sub>3</sub>), methylene blue (C<sub>16</sub>H<sub>18</sub>ClN<sub>3</sub>S) and methylene blue waste from UNJ chemistry laboratory, aquades, hydrochloric acid (HCl), and sodium hydroxide (NaOH).

### 2.2 Methods

#### 2.2.1 Adsorbent preparation

Tea waste was washed several times with distilled water, then boiled in distilled water at 100°C for 1 hour to remove any remaining color. After boiling, it was washed again with distilled water until the filtrate was colorless. The tea waste was then dried in an oven at 110°C for 4 hours. Once dried, the tea waste was ground and sieved using a 100-mesh sieve. The resulting adsorbent was then soaked in a 0.1 M HCl solution for 24 hours, using a ratio of 25 grams of tea waste to 200 mL of 0.1 M HCl. After 24 hours, the tea waste was filtered and washed multiple times with distilled water until a neutral pH was reached. Finally, the tea waste was dried again in an oven at 110°C for 4 hours.

#### 2.2.2 Synthesis and modification of Fe<sub>3</sub>O<sub>4</sub> with tea waste

Fe<sub>3</sub>O<sub>4</sub> synthesis was carried out using the coprecipitation method. The process involved mixing FeSO<sub>4</sub>·7H<sub>2</sub>O and FeCl<sub>3</sub>·6H<sub>2</sub>O in a molar ratio of 1:2 in 100 mL of distilled water, followed by stirring for 30 minutes at 80°C. Next, 10 mL of 25% NH<sub>3</sub> solution was added dropwise until a black precipitate formed. Once the precipitate had formed, 5 g of tea waste was added, and the mixture was stirred for another 30 minutes. The sediment was filtered and washed until a neutral pH was reached, then dried in an oven at 100°C for 3 hours. The adsorbent was characterized using Fourier transform infrared (FTIR, Thermo Scientific Nicolet iS-10 with iD5 ATR Detector) spectroscopy and scanning electron microscopy (SEM, Zeiss EVO MA 10).

#### 2.2.3 Standard calibration curve of methylene blue

The calibration curve was prepared using concentrations of 10, 20, 30, 40, and 50 ppm. Adsorption measurements were taken in the wavelength range of 600–700 nm to determine the maximum wavelength with the highest absorbance using a UV-Vis spectrophotometer (Shimadzu UV1280). A calibration curve plotting concentration against absorbance was then constructed to facilitate the determination of sample concentrations.

#### 2.2.4 Adsorption condition optimization

The optimization process was conducted based on pH and contact time parameters. The experiment used 50 mL of 40 ppm methylene blue (MB), 0.05 g of adsorbent, stirred at 500 rpm, and maintained at room temperature. The pH values tested were 2, 4, 6, 8, 10, 12, and 14, with each sample shaken for 60 minutes. For the contact time parameter, experiments were conducted at the optimum pH for durations of 30, 60, 90, and 120 minutes. After

treatment, the mixture was filtered, and the filtrate was analyzed using a UV-Vis spectrophotometer.

### 2.2.5 Methylene blue waste adsorption

The methylene blue waste used was taken from the UNJ Chemistry Laboratory. The initial concentration of waste was measured using a spectrophotometer UV-VIS. Then 50 mL of waste was adsorbed at pH and optimum contact time using 0.05 g of adsorbent, at a speed of 500 rpm, and at room temperature. Measured the final concentration of waste obtained.

### 2.2.6 Methylene blue waste adsorption

The adsorption isothermal process is carried out to describe the adsorption process between adsorbents and adsorbates. This isotherm modeling is carried out based on two models, namely the Langmuir isotherm and the Freundlich isotherm. Langmuir's isotherm describes the process of monolayer adsorption, where each active site can only absorb one waste molecule. The equation of Langmuir's isotherm [20].

$$\frac{1}{Q_e} = \frac{1}{K \cdot Q_m} \times \frac{1}{C_e} + \frac{1}{Q_m} \quad (1)$$

Where  $C_e$  is the concentration of adsorbates in equilibrium,  $Q_e$  is the amount of adsorbate absorbed in equilibrium,  $K$  is the Langmuir constant, and  $Q_m$  is the adsorption capacity [21].

The Freundlich isotherm model describes the multilayer adsorption process, where each active site of the adsorbent has different adsorption capabilities so that it can form a multilayer in its interaction between adsorbent and adsorbate. Here is Freundlich's isotherm equation [20].

$$Q_e = K \cdot C_e^n \quad (2)$$

$$\text{Log } Q_e = \frac{1}{n} \cdot \text{Log } C_e + \text{Log } K \quad (3)$$

Where  $Q_e$  is the number of adsorbates that are absorbed,  $C_e$  is the equilibrium concentration,  $K$  and  $n$  are constants [22].

## 3. Results and Discussion

The adsorbent preparation process begins with washing and boiling tea wastes to clean tea wastes from impurities such as dust and remove tannins that have the potential to interfere with the adsorption process [23]. Then the tea wastes are dried and crushed into a size of 100 mesh. The loss of tannin content in tea wastes is characterized by a change in the color of the tea waste's filtrate from brown to clear. This process aims to avoid any interaction when tea wastes are modified with  $\text{Fe}_3\text{O}_4$ . Tannin compounds can react with  $\text{Fe}^{3+}$  ions to form

complex compounds [24]. Then, an activation process is carried out using 0.1M HCl; this process is carried out to enlarge the adsorbent pores so that the surface area of the adsorbent becomes larger and can absorb more waste [14]. After the adsorbent activation process, it is washed to neutralize the pH, then dried.

$\text{Fe}_3\text{O}_4$  was used to increase the adsorption capacity because the material has a large surface area and has a potential group, namely Fe-O, which can adsorb methylene blue waste [25]. Also,  $\text{Fe}_3\text{O}_4$  is a ferromagnetic material that can facilitate the separation process between methylene blue waste and adsorbents after adsorption by bringing the adsorption container closer to the external magnetic field [26]. The powder adsorbent is characterized by a black colour, as shown in Fig. 1.



Figure 1. Powder tea waste –  $\text{Fe}_3\text{O}_4$  adsorbent.

### 3.1 Structural analysis

FTIR characterization was performed on tea waste adsorbents before and after modification with magnetite material. Based on the FTIR spectrum in Fig. 2, in the adsorbent of tea waste before modification, a wide spectrum was obtained at the absorption of the wave number of  $3337.88 \text{ cm}^{-1}$  suspected to be the -OH group, at the absorption of the wave number of  $1615.08 \text{ cm}^{-1}$  it is suspected that there is a C=C bond, at the absorption of the wave number of  $2917.57 \text{ cm}^{-1}$  and  $2850.66 \text{ cm}^{-1}$  it is suspected that there is an aliphatic C-H bond, at the absorption of the wave number of  $1030.32 \text{ cm}^{-1}$  there is a suspected -C-C- bond. Meanwhile, in the modified tea waste adsorbent, the same number of waves was absorbed, but there was a new absorption at  $550.71 \text{ cm}^{-1}$ , which is suspected to be Fe-O absorption [27]. The existence of a new peak proves that  $\text{Fe}_3\text{O}_4$  compounds

have been successfully synthesized and modified with tea waste.

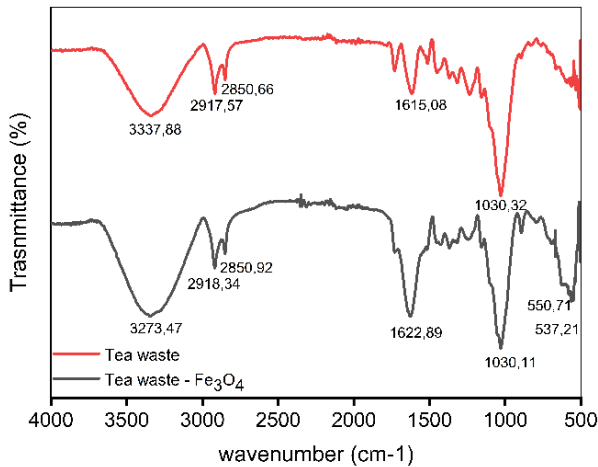


Figure 2. FTIR spectrum of tea waste and tea waste-Fe<sub>3</sub>O<sub>4</sub>.

### 3.2 Optimization of methylene blue adsorption capacity

The calibration curve of the MB is shown in Fig. 3. The MB absorbance is obtained from the wavelength at 663 nm. Then the absorbance values obtained from concentrations of 10, 20, 30, 40, and 50 ppm are made a curve that produce a linear equation that can be used to determine the concentration of the sample.

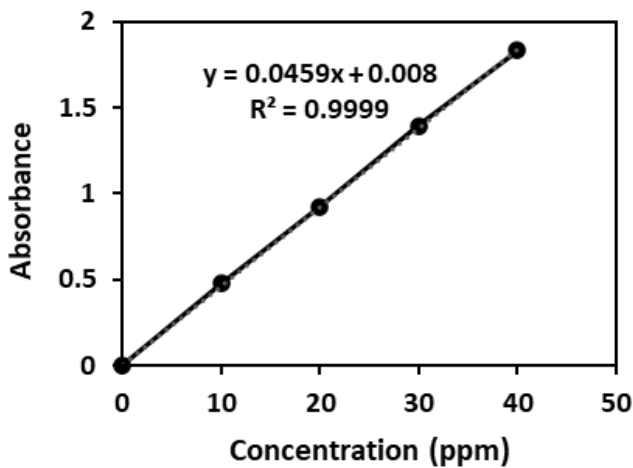


Figure 3. Methylene blue curve standard.

#### 3.2.1 pH optimization

The pH level can affect the charge or functional group on the adsorbent surface, to affects the adsorption capacity, where the adsorbent plays a role in adsorbing methylene blue [28]. The results of pH optimization can be seen in Fig. 4. At pH 2-10, there is an increase in the %removal adsorption value, but at pH 12 and 14, there is a decrease in the %removal adsorption value. This is because there are too many negative charges or hydroxyl

groups (-OH) in the adsorption environment, so that there is competition between ions to interact with waste, which can prevent waste from being adsorbed. The optimum pH condition occurs at pH 10 because the increase in pH will make the adsorbent environment more alkaline and have more negative charges from the (-OH) group, so that the absorption of methylene blue that occurs during adsorption will be better [29]. The absorption process is better because there is an electrostatic interaction between the adsorbent and methylene blue [1].

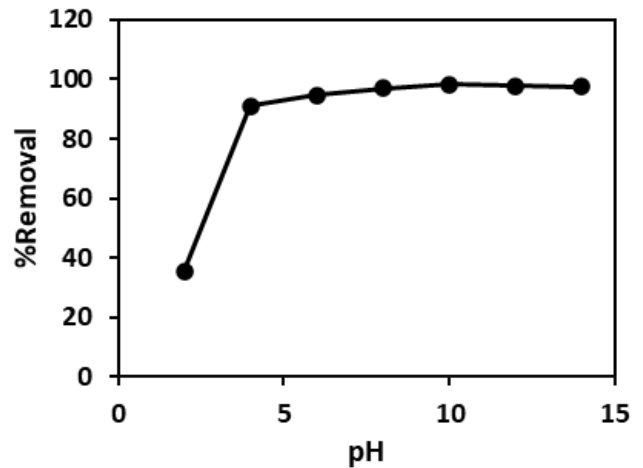


Figure 4. Correlation between the pH solutions and the %removal of MB.

#### 3.2.2 Contact time optimization

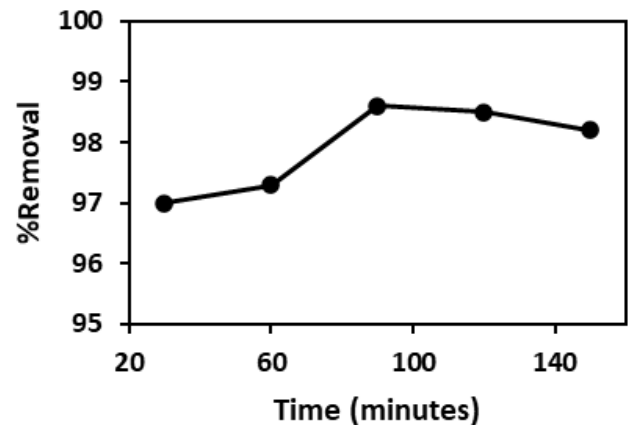


Figure 5. Correlation between the contact time and the %removal of MB.

The length of time used to adsorb greatly affects the absorption process that occurs, because the length of time used affects the interaction that occurs between methylene blue and adsorbents. The results of the contact time optimization can be seen in Fig. 5. At 30 to 90 minutes of contact time, there was an increase in the %removal value, but at the contact times of 120 and 150 minutes,

there was a decrease in the %removal value. This is because at the beginning of the time the surface of the adsorbent is not fully filled or has not saturated, thus facilitating the interaction process of methylene blue with the adsorbent, then after 90 minutes the adsorption process will become slower because the surface of the adsorbent is fully filled or has saturated, so that there is no increase in the % removal value [30]. So that at the beginning of adsorption, there is an increase in absorption, and then there will be a decrease in adsorption because it has reached an equilibrium point where the available active sites are saturated.

### 3.2 Methylene blue waste adsorption

The initial concentration of methylene blue waste was 22.77 ppm, then after adsorption, a final concentration of 0.22 ppm was obtained with a %removal value of 99%. The results of this adsorption are shown in Fig. 6, where the color difference between the waste before and after adsorption can be seen. Based on the Decree of the State Minister of Environment Number KEP-51/MENLH/10/1995 which stipulates that methylene blue waste that can be discharged into the environment must have a maximum concentration of 5-10 ppm on the condition that in fulfilling the waste level is not diluted with water, then the adsorbed methylene blue waste can be safely disposed of into the environment.



Figure 6. Methylene blue waste before and after adsorption.

### 3.2 Morphological analysis

SEM characterization was carried out to determine the morphology of the adsorbent surface [31]. The samples used are adsorbents before and after adsorption. In Fig. 7, it can be seen that the morphology of the adsorbent

surface before adsorption looks rough and hollow [17], while the morphology of the adsorbent surface after adsorption is denser and slightly hollow. This is because the waste has interacted with and been adsorbed by the active site in the adsorbent.

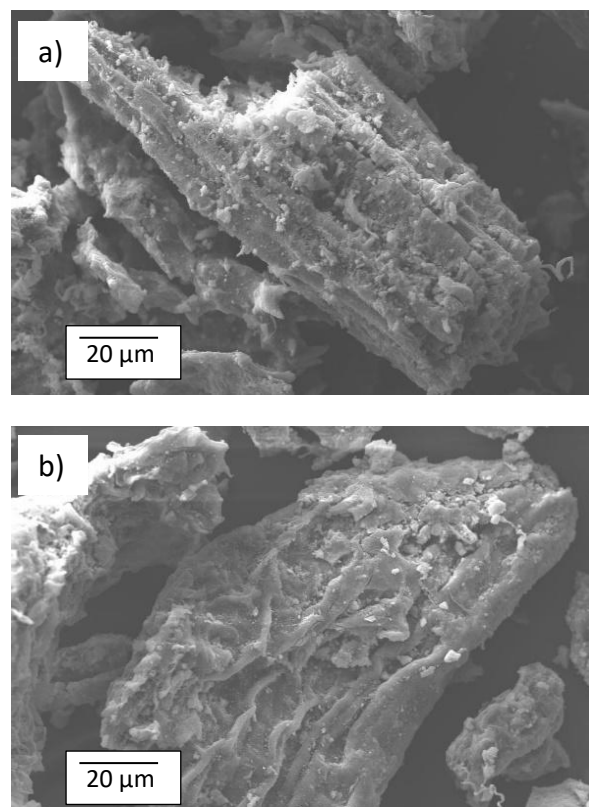


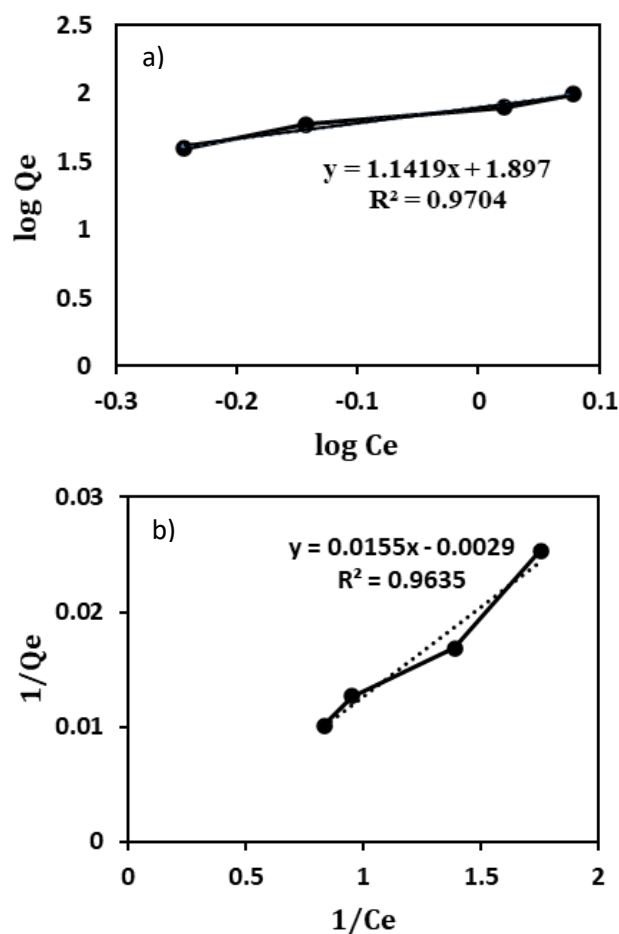
Figure 7. Morphology before adsorption (a) and after adsorption (b).

### 3.2 Isotherm adsorption

The determination of the adsorption isothermal model is important to determine the relationship between the amount of adsorbate absorbed by the adsorbent surface and the adsorbate concentration at equilibrium. This is done to describe the way molecules or ions interact with the active site on the adsorbent surface [22]. Determination of the isothermal model was carried out using data resulting from optimization of adsorption conditions. Langmuir assumes that surfaces are homogeneous, and in Freundlich adsorption isothermal model assumes that surfaces are heterogeneous [4]. The following is the isotherm curve of Langmuir and Freundlich in Fig. 8.

Based on the  $R^2$  value obtained in the Langmuir model  $R^2$  0.9635, and in the Freundlich model  $R^2$  0.9704. The adsorption is suitable for the Freundlich isotherm model rather than the Langmuir, which has an  $R^2$  value that is closest to 1 [32]. It can be assumed that the adsorption of

methylene blue using  $\text{Fe}_3\text{O}_4$  modified tea waste adsorbent is a multilayer adsorption on a heterogeneous surface.



**Figure 8.** Isotherm adsorption of Langmuir (a) and Freundlich (b).

#### 4. Conclusion

From the results of this study, tea waste adsorbent activated with HCl and modified with magnetite can be used effectively to adsorb methylene blue. The results showed that the adsorbent was optimal at pH 10 and a contact time of 90 minutes. The final concentration obtained after adsorption on tea wastes with optimal pH and time, namely 0.05 grams of adsorbent, 50 mL, and room temperature, was 0.22 ppm with a removal value of 99%. The adsorption data obtained from the adsorption process were in accordance with the Freundlich isotherm model, with an  $R^2$  value of 0.9704. The FTIR results proved that  $\text{Fe}_3\text{O}_4$  could be successfully synthesized, and based on the SEM results, the surface morphology of the adsorbent was hollow, so that the tea dregs were successfully adsorbed.

#### Author contributions

Yussi Pratiwi: Conceptualization, Supervision, Funding acquisition, Reviewing. Miftah Aulia: Methodology, Resources, Writing. Baiq Amelia Riyandari: Project administration, Formal analysis. Devi Nur Anisa: Visualization, Data Curation.

#### Conflicts of interest

The authors declare that they have no conflicts of interest.

#### Acknowledgement

This research is supported by research facilities of Chemical Research Laboratory at the State University of Jakarta.

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