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# Synthesis of Manganese Oxide – Magnetite ($Fe_3O_4$) for The Degradation of Methylene Blue

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Manganese oxide; $Fe_3O_4$; Fenton heterogeneous catalyst; Methylene blue

**Abstract.** Methylene blue is a dye that is challenging to break down in aquatic environments. A promising approach to degrade methylene blue involves using metal oxide-based materials. Magnetite ($Fe_3O_4$) is beneficial for easy separation after catalytic reactions, while manganese oxide is effective in oxidation processes. Combining manganese oxide and magnetite can create materials with synergistic properties that improve the degradation of methylene blue through photocatalytic mechanisms or modified Fenton reactions. The development of manganese-magnetite oxide ($Fe_3O_4$) composites offers a potential solution for efficient, eco-friendly, and reusable catalysts. This study aims to investigate the synthesis of manganese-magnetite oxide and its performance in methylene blue degradation as an innovative solution to water pollution. The $MnOx-Fe_3O_4$ composite was successfully synthesized using the hydrothermal method at 110°C for 6 hours. XRD results show that increasing $Fe_3O_4$ concentration enhances the $Fe_3O_4$ peak, while the Hausmanite peak disappears, overlapping with the $Fe_3O_4$ peak, as supported by SEM results. The $MnOx-Fe_3O_4$ composite (1:10 ratio) was most effective in degrading methylene blue, achieving 92% degradation of 100 mL of 20 ppm methylene blue with a catalyst concentration of 0.1 g and 5 mL of $H_2O_2$ using the Fenton method.

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## 1. Introduction

Methylene blue (MB) dye is frequently employed due to it's vivid hue and is difficult to break down in aquatic environments, it poses a high toxicity risk to aquatic life (Mar'af and Hendrawati, 2018). Several methods used to reduce the dye content in textile industry waste include biological methods, coagulation, electrocoagulation, adsorption, ozonation and chlorination. However, these methods are less effective in dealing with hazardous substances in textile industry waste which often endanger the surrounding environment, especially waters (Hardian et al, 2021).

Using materials based on metal oxides is one possible technique for methylene blue degradation. Manganese oxide exhibits strong catalytic activity throughout the oxidation process, whereas magnetite ($Fe_3O_4$), a magnetic substance, offers advantages in separation following catalytic events. Through modified Fenton reactions or photocatalytic processes, manganese oxide and magnetite can combine to create materials with complementary qualities that improve the efficiency of methylene blue breakdown.

The synthesis of manganese oxide-magnetite ($Fe_3O_4$) composite materials is an attractive strategy for developing catalysts that are efficient, environmentally friendly and easy to re-use. This study aims to explore the synthesis method of manganese-magnetite oxide and evaluate its performance in the degradation of methylene blue, in order to provide an innovative solution to the problem of water pollution.

The ability of manganese oxide as a catalyst is influenced by its physical and chemical properties. Different synthesis methods and synthesis conditions also affect the structure and surface characteristics and its ability to act as a catalyst (Hafizhah et al., 2015). Awaluddin et al., 2025 successfully synthesized cryptomelane using the precipitation method with precursor concentrations of $KMnO_4$ and glucose of 0.04 M and 0.06 M with a percentage of methylene blue degradation reaching 97.01% for 120 minutes and an initial concentration of methylene blue of 30 ppm. Subkhan (2018) synthesized manganese oxide Manganosite using the sol gel method by mixing 47.25 grams of glucose ($C_6H_{12}O_6$) added to the $KMnO_4$ solution successfully degraded methylene orange by 92.09% in 90 minutes. Prasetya (2019) successfully synthesized birnessite type manganese oxide by sol gel method using $KMnO_4$ and citric acid precursor successfully degraded Methylene Blue up to 97.63%. Synthesis of manganese oxide with glucose and $KMnO_4$ using hydrothermal method has never been reported, so the synthesis of manganese oxide using hydrothermal method is expected to find manganese oxide that has different structure and surface characteristics.

By creating composites, manganese oxide's catalytic activity can be increased by overcoming agglomeration, increasing surface area, and speeding up the production of hydroxyl radicals. Due to its many uses, including drug delivery, MRI, catalysts, sensors, data storage, and the adsorption of water contaminants, magnetite ($Fe_3O_4$) is gaining attention. Its high absorption capacity and reaction to magnetic fields make it easier to separate substances in solution (Li et al., 2018).

$MnO_2/Fe_3O_4$ composites with unique properties have been successfully created by a variety of techniques, including sol-gel, precipitation, and ultrasonic. At pH 9, the hydrothermally produced α-$MnO_2/Fe_3O_4$ nanocomposite can break down nearly all 2,4-dichlorophenol in 80 minutes and can be recycled at room temperature using a magnet (Dubey et al., 2022). When $MnO_2$ is added to $Fe_3O_4$/CuO, waste dye decolorization through Fenton oxidation is more effective and has a high reusability after six uses (>90 %) (Ghasemi et al., 2021). Furthermore, the Mn-doped $FeO_4$/RGO catalyst demonstrated great stability and promise for wastewater treatment, was successful in degrading RhB using the Photo-Fenton method at neutral pH, and was simple to separate using a magnetic field (Chen et al., 2019).

In this study, the synthesis of manganese oxide composite - $Fe_3O_4$ was carried out using the hydrothermal method, the reactants used were $KMnO_4$ and glucose, then supported with pure $Fe_3O_4$ and expected to produce a crystal structure, morphology, and surface area that are different from the others. This composite is expected to have high activity and can be reused.

**2. The Methods**

**2.1 Tools and materials**

The materials used in this research were Potassium Permanganate ($KMnO_4$) (Merck), HCl (Merck), $C_6H_{12}O_6$ (Merck), $Fe_3O_4$, methylene blue, Whatman No.42 filter paper, distilled water and $H_2O_2$

(Merck). The equipment used in this research was an autoclave, hot plate, oven, centrifuge, X-Ray diffraction (XRD), Scanning Electron Microscopy (SEM) (6510 LA), UV-Vis spectrophotometer (Shimadzu, IR Prestige-21), pH-meter, analytical balance (Shimadzu), magnetic stirrer, volume pipette, measuring pipette, mortar pestle, tongs, heat-resistant gloves, thermometer, cuvette, measuring flasks, petri dishes, and a set of glassware commonly used in chemical research.

### 2.2 Synthesis of manganese oxide – magnetite ($Fe_3O_4$) using the hydrothermal method

Hydrothermal synthesis was carried out by mixing 2.765g of $KMnO_4$ added with 4.725g of glucose ($C_6H_{12}O_6$), then stirred until dissolved, after which $Fe_3O_4$ was added with variations (0, 1:1, 1:5, 1:10) of the weight of $KMnO_4$. After that, the mixture was put into a Teflon-coated stainless steel autoclave, temperature 110 °C for 6 hours. The products formed were washed with 0.1M HCl and distilled water, respectively. The product was dried at 120 °C for 2 hours for characterization.

### 2.3 Characterization of manganese oxide – magnetite ($Fe_3O_4$)

The pure compounds were characterized using XRD and SEM. Characterization of manganese oxide - magnetite ($Fe_3O_4$) using SEM aims to determine the surface shape and particle uniformity of manganese oxide - magnetite ($Fe_3O_4$). XRD is used to determine the structure, level of crystallinity, and level of purity of manganese oxide using X-ray diffraction. at a scan speed of 4° min-1 from 5 to 80°.

### 2.4 Degradation of methylene blue

*2.4.1 Degradation of methylene blue using the photocatalytic method*

Take 100 mL of MB liquid waste solution using a volume pipette. Then put it in a 250 mL beaker and add 0.1 gr of catalyst. Stirred using a hotplate stirrer for 120 minutes with the help of light and a centrifuge for 20 minutes at 2000 rpm to separate the catalyst. Next, testing was carried out with a UV-Vis spectrophotometer with a wavelength of 663 nm.

*2.4.2 Degradation of methylene blue using the photo Fenton method*

Take 100 mL of MB liquid waste solution at a concentration of 20 ppm using a volume pipette. Then put it in a 250 mL beaker then add 0.1 gr catalyst and 5 mL $H_2O_2$. Then stirred using a hotplate stirrer at room temperature for 120 minutes with the help of light. Next, centrifuge for 20 minutes at a speed of 2000 rpm to separate the catalyst. Carry out the test with a UV-Vis spectrophotometer with a wavelength of 663 nm.

*2.4.3 Degradation of methylene blue using the Fenton method*

Take 100 mL of MB liquid waste solution using a volume pipette. Then put it in a 250 mL beaker, then add 0.1 gr of catalyst and 5 mL of $H_2O_2$. Then stirred using a hotplate stirrer at room temperature for 120 minutes without light. Then centrifuge 20 minutes at 2000 rpm to separate the catalyst. Next, testing was carried out with a UV-Vis spectrophotometer with a wavelength of 663 nm

## 3. Result and Discussion

### 3.1 **Result of manganese oxide – magnetite synthesis**

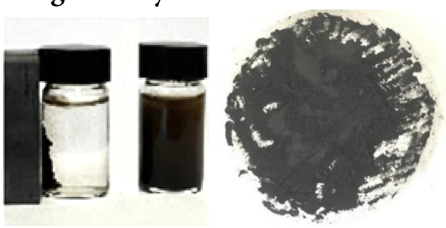

**Figure 1.** Image of the MnOx-$Fe_3O_4$ composite resulting from synthesis.

The MnOx-$Fe_3O_4$ composite interacts with magnets and is perfectly attracted. As seen in Figure 1. This shows the paramagnetic nature of the MnOx-$Fe_3O_4$ combination, in accordance with the research of Dubey *et al.* (2022).

### 3.2 Analysis of lattice structure in MnOx–$Fe_3O_4$ composites

An X-ray diffractometer was used to characterize the structure of each synthesized solid. The structure and crystal phase of the composite samples were investigated using X-ray diffraction (XRD). The XRD results of MnOx and the MnOx – $Fe_3O_4$ Composite may be shown in figures 2 From the XRD data, the MnOx sample is a mixture of several manganese oxide phases with a very small particle size and an irregular structure. Numerous circumstances, including the synthesis process, the synthesis environment, or the presence of contaminants in the sample, might result in this disordered structure (Sari et al., 2021).

In the synthesized composite MnOx/(2:3)/6 Jam, the hausmanite crystal phase at 2θ, namely $31{,}480^0$ and $49{,}620^0$ with reflection fields of 112 and 204, was also obtained, then the cryptomelane crystal phase at 2θ, namely $24{,}380^0$ with a reflection field of 202 and the birnessite crystal phase at 2θ, namely $51{,}810^0$ with a reflection field of 004, was obtained in accordance with ICDD data from each crystal phase is shown in Figure 2.

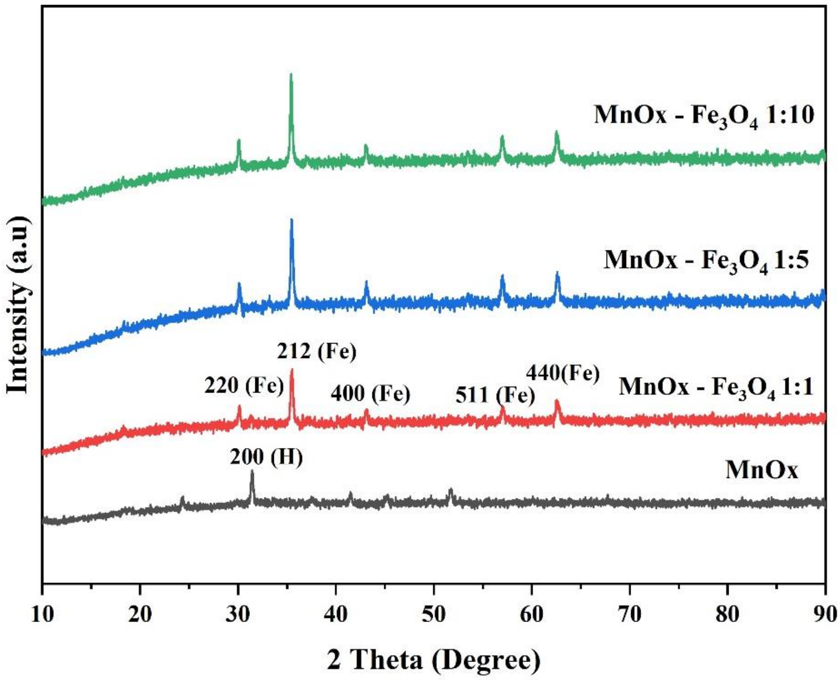


**Figure 2.** XRD patterns of MnOx/$Fe_3O_4$ material composite after the reaction.

The XRD results after adding $Fe_3O_4$ can be seen that the peak formed is the peak belonging to $Fe_3O_4$ which is in accordance with JCPDS No. 19-0629. Based on the image above, the peak that appears in the MnOx-$Fe_3O_4$ composite synthesized using the hydrothermal method is a typical peak of magnetite characteristics, as evidenced by the presence of peaks that appear at diffraction angles of around 30, 35, 43, 57 and 62° with Miller indices of 220, 212, 400, 511 and 440 respectively. The research results are in line with the research results that have been reported previously (Dubey, 2022 and Wang, 2015). So magnetite has been successfully synthesized using the hydrothermal method. The more $Fe_3O_4$ added, the higher the resulting peak, indicating that the MnOx surface is covered by $Fe_3O_4$.

The initial peak of MnOx generated vanishes with increasing $Fe_3O_4$ addition, allowing the manganese oxide to shift structurally and become amorphous, which prevents detection in XRD examination. $Fe_3O_4$ can cover or conceal the diffraction peaks of MnOx because it has a higher diffraction intensity than MnOx in a specific $2^{\theta}$ angle range. The diffraction from MnOx becomes very faint or even invisible if the $Fe_3O_4$ level is high enough.

By a redox process, the addition of $Fe_3O_4$ can affect the oxidation state of Mn in MnOx. $MnO_2$ has a more amorphous structure or develops a new phase that differs from hausmanite or cryptomelane. $Mn^{3+}$ in hausmanite can be oxidized to become $Mn^{4+}$, which is more stable in the form of $MnO_2$. Consequently, only the $Fe_3O_4$ peak is left once the first phase vanishes.

### 3.3 Surface morphology analysis of MnOx–$Fe_3O_4$ composites

When 1:1 $Fe_3O_4$ is added, the SEM image (Figure 3) reveals that MnOx has an uneven granular or particle-shaped morphology with a rough and dense surface that overlaps. Smaller grains ($Fe_3O_4$ nanoparticles) then bond together to form larger agglomerations or clusters. An agglomeration is a group of nanoparticles that come together as a result of interactions between them. The particles seem to have an uneven shape and tend to be cubic or polygonal as more $Fe_3O_4$ is added. Although particle sizes vary, a group of particles usually appears to be pretty uniform. The probability of very high contact forces between particles is indicated by the existence of aggregation or clumping (He,Y, 2018).

The SEM results show that the addition of $Fe_3O_4$ initially sticks to the MnOx surface and then combines and forms a larger surface, which shows that the $Fe_3O_4$ has mixed completely and covered the MnOx surface. The more $Fe_3O_4$ is added, the particles appear larger and the crystalline structure is more cubic or polygonal in shape. Indications are that $Fe_3O_4$ has begun to interact with MnOx, causing morphological changes. In accordance with the XRD results which show that the more $Fe_3O_4$ produced, the higher the Fe peak produced.

**$MnO_x$** **$MnO_x$ – $Fe_3O_4$ (1:1)** **$MnO_x$ – $Fe_3O_4$ (1:5)** **$MnO_x$ – $Fe_3O_4$ (1:10)**

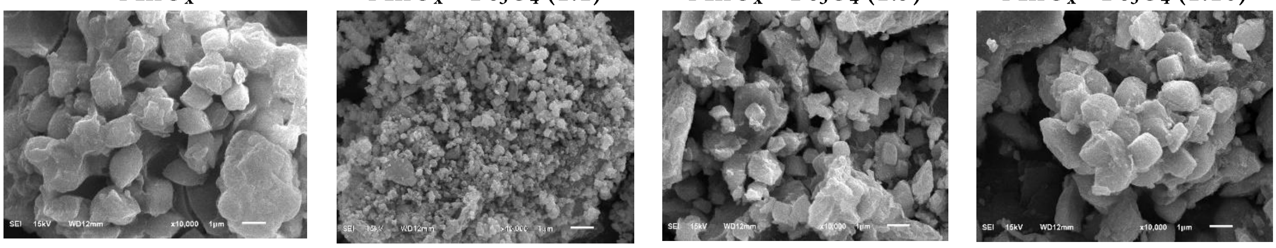

**Figure 3.** SEM images of $MnO_x$ and $MnO_x$-$Fe_3O_4$ Composites 1:1, 1:5 and 1:10.

### 3.4 Analysis photocatalytic activity

The composites using the photocatalytic method MnOx were able to degrade 17.67%, MnOx – $Fe_3O_4$ 1:1 19.43%, MnOx – $Fe_3O_4$ 1:5 22.64%, MnOx – $Fe_3O_4$ 1:10 24.97% after 120 minutes (see Figure 4). Photocatalytic methods with manganese-magnetite oxide are less effective for the degradation of methylene blue due to limitations in light absorption, fast electron-hole recombination, and low adsorption capacity.

$Fe_3O_4$ has a relatively small energy gap (~0.1 eV - 0.5 eV), so it is less effective in absorbing visible and ultraviolet light. Manganese oxide ($MnO_x$) itself has photocatalytic activity, but its efficiency depends on the phase and crystal composition. In the photocatalysis mechanism, electrons ($e^-$) excited from the valence band to the conduction band must remain free long enough to react with oxygen or water, forming reactive species such as hydroxyl radicals (•OH). In the manganese-magnetite oxide system, electrons and holes tend to recombine quickly before they can react, reducing the effectiveness of methylene blue degradation.

It was found that degradation using the photo Fenton method of MnOx composite was able to degrade 51.45%, MnOx – $Fe_3O_4$1:1 66.43%, MnOx – $Fe_3O_4$ 1:5 73.85%, MnOx – $Fe_3O_4$ 1:10 78.30% as shoen in Figure 5. The photo-Fenton method is a combination of the Fenton reaction and ultraviolet (UV) irradiation which aims to increase the production of hydroxyl radicals (•OH) to degrade organic pollutants such as methylene blue. In the photo-Fenton method, regeneration of $Fe^{2+}$ by UV light aims to increase •OH production, but if electron-hole recombination on the catalyst surface occurs quickly, the number of radicals available to degrade methylene blue becomes less. So it is less able to degrade methylene blue.

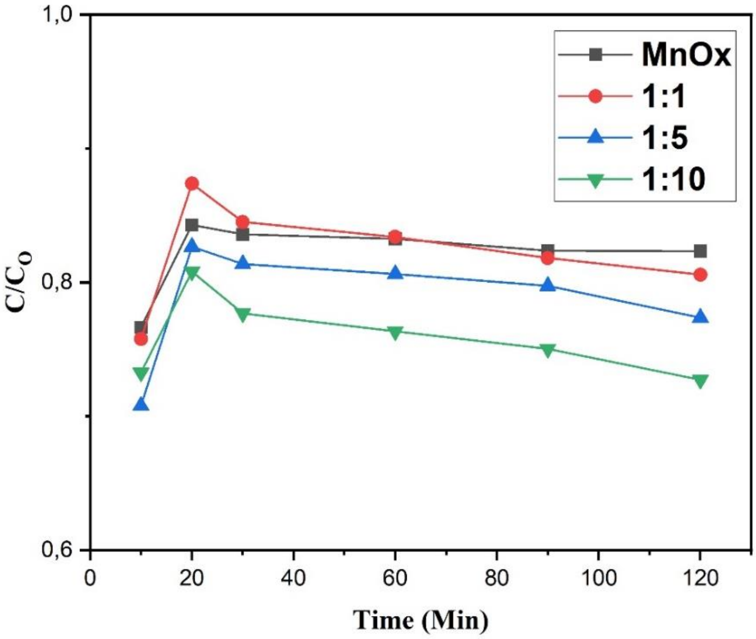


**Figure 4.** Graph of methylene blue degradation results using the photocatalytic method.

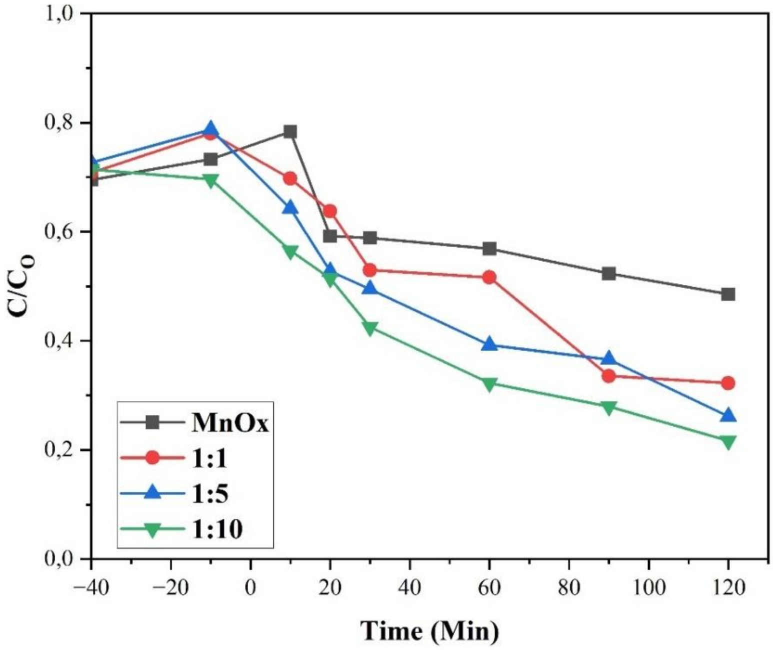


**Figure 5**. Graph of methylene blue degradation results using Fenton photo method

Meanwhile, the results of research using the Fenton method obtained several optimum points for the MB degradation process with the best composite effect in Figure 6, obtained MnOx 42.22%, MnOx – $Fe_3O_4$ 1:1 51.50%, MnOx – $Fe_3O_4$ 1:5 84.67%, MnOx – $Fe_3O_4$ 1:10 92.54%. In the Fenton system $H_2O_2$ reacts with iron ions ($Fe^{2+}$) to produce hydroxyl radicals (•OH). This hydroxyl radical is a very strong and non-selective reactive species, so it is able to oxidize various organic compounds, including methylene blue, into simpler and less dangerous compounds (Ghasemi, 2021).

When $Fe_3O_4$ is combined with manganese oxide (MnOx) in a composite, both support each other in increasing degradation activity. MnOx can speed up the oxidation reaction, while $Fe_3O_4$ helps in the catalyst separation process after use. $Fe_3O_4$-MnOx composites often have a larger surface area compared to single oxides, which increases the number of active sites for degradation reactions. The addition of $Fe_3O_4$ can increase the number of reaction centers where hydroxyl radicals (•OH) are formed. $Fe_3O_4$ can act as a catalyst in the Fenton process, which functions to increase the degradation rate. The more $Fe_3O_4$, the more these radical formation reactions occur, which accelerates degradation.

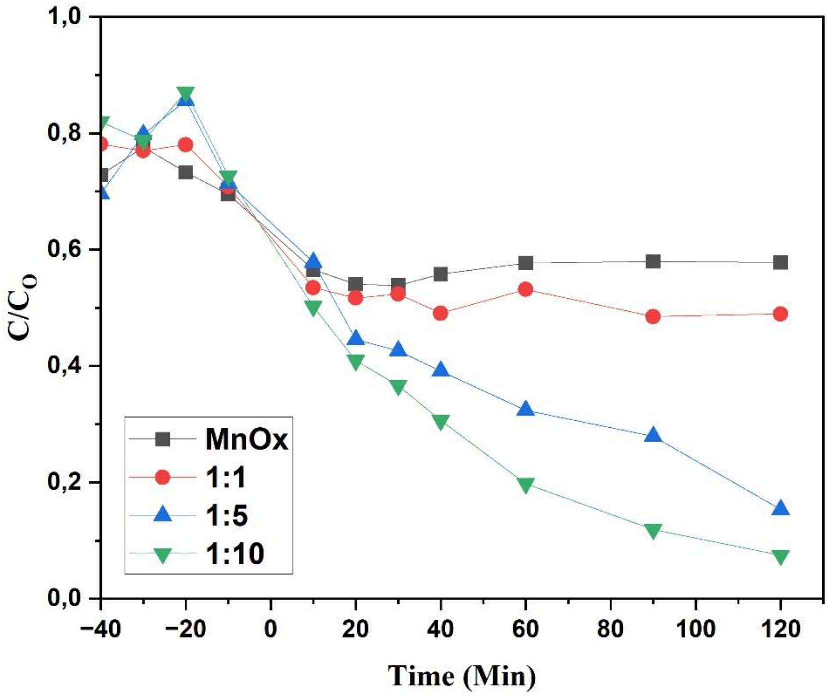


**Figure 6.** Graph of methylene blue degradation results using the Fenton Method

In the Fenton process, the production of highly reactive hydroxyl radicals (•OH) occurs directly through the Fenton reaction without the need for light as a trigger. These hydroxyl radicals are among the most powerful oxidants available for the degradation of organic compounds such as methylene blue. Photo-Fenton also produces •OH, but this reaction requires UV light to activate the reaction, which can limit its performance depending on the light intensity and exposure time. Photocatalytic, on the other hand, relies on catalyst materials such as $TiO_2$ that are only active under light, which reduces their efficiency under low or limited light conditions.

The Fenton method tends to be cheaper and easier to implement because it only requires simple chemicals such as $H_2O_2$ and $Fe^{2+}$, and does not require additional devices such as UV lamps for activation. Photo-Fenton and photocatalytic require devices to regulate and provide light sources, which can increase operational costs and system complexity. So in this study the Fenton method is more effective in degrading (92%) than Photo-Fenton (78%) and Photocatalytic (24.97%).

In this study, the Fenton method is the best method for degrading methylene blue. In the Fenton method, $Fe^{2+}$ ions from $Fe_3O_4$ and $Mn^{3+}/Mn^{4+}$ ions from MnOx play a role in activating $H_2O_2$ to produce highly reactive hydroxyl radicals (•OH). These radicals attack the aromatic structure of methylene blue, causing degradation into simpler and colorless compounds. The synergistic effect between MnOx and $Fe_3O_4$ contributes to increased degradation efficiency. MnOx helps accelerate the regeneration of $Fe^{2+}$ from $Fe^{3+}$, thereby strengthening the catalytic cycle. In addition, the combination of the two materials provides more active sites and expands the range of operating conditions.

Although quantitative recycling tests have not been conducted thoroughly, the MnOx–$Fe_3O_4$ composites show promising potential for reuse based on the magnetic properties of $Fe_3O_4$ which allows easy separation of the catalyst after reaction, as previously reported by Ghasemi and Dubey. The hydrothermal synthesis method used is relatively simple, therefore, further development has high potential to be applied to the treatment of liquid waste containing synthetic dyes.

## 4. Conclusion

The synthesis of MnOx–$Fe_3O_4$ was successfully carried out using the hydrothermal method at 110 °C for 6 hours. The addition of $Fe_3O_4$ significantly influenced the structural and catalytic characteristics of manganosite. XRD analysis showed that increasing the $Fe_3O_4$ content enhanced the diffraction peak intensity associated with $Fe_3O_4$, while the hausmannite peak diminished due to overlap with $Fe_3O_4$ reflections, consistent with the SEM observations. The optimal composition for methylene blue degradation was MnOx–$Fe_3O_4$ (1:10), achieving 92% degradation of 100 mL of 20 ppm methylene blue using 0.1 g of catalyst and 5 mL of $H_2O_2$ via the Fenton method.

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