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Synthesis of Lead Oxide Using the Oxidizing Agent $(NH_4)_2S_2O_8$ with Variaous Amounts of NaOH for Decolorization of Remazol Black B Solution

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Abstract

In this study, the lead oxide has been successfully synthesized using oxidizing agents $(NH_4)_2S_2O_8$ and applied to decolorize the Remazole Black B (RBB) solution. Lead oxide was synthesized by the batch method through the mechanism of reduction-oxidation and characterized using SEM-EDX and XRD. Decolorizing RBB solution was performed using a modified Fenton method. This study aims to determine the optimum performance of RBB decolorization from lead oxide. The results showed that the crystal formed contains Pb and O. Pb oxide obtained was α-PBO_2 with the orthorhombic crystal system. Based on characterization, the lead oxide obtained has a surface morphology of coral flowers-like. Synthesis utilizing equilibrium composition (1:2) and a drying temperature of 100°C yielded Pb oxide, which has a better potential as a modifier in RBB decolorization with a decolorization percentage of 80.07%. The process was then carried out for advanced synthesis less and excess of molar variations. The ratio of lead oxide: NaOH (1:4) showed optimum performance in decolorizing RBB with a decolorization percentage of 80.07 %.

1. Introduction

Increased production in the batik industry results in increased waste discharged into the environment, especially those associated with batik textile dyes. The waste discharged is an organic compound that cannot be degraded biologically, hence causing harm to the aquatic environment. This dye waste contains chemicals that pollute the environment, such as remazol black B, remazol brilliant orange 3r, remazol golden yellow, and remazol red [1]. The natural destruction of these dyes can be carried out by decomposition by sunlight. However, this reaction takes a long time compared to the accumulation of dyes in the bottom of the water, so the photodegradation process of this dye waste is not significant [2].

One of the solutions for overcoming this waste is degrading the dye through the PbO_2-modified Fenton method. The Fenton method is a method for degrading organic compounds by forming hydroxyl free radicals. However, the drawback of this method is the use of PbO_2 electrodes as a source of Pb^{2+} contamination which can harm the environment. Given the dangerous level of Pb^{2+} and there is still residual Pb^{2+} which is still dissolved and becomes a pollutant, this dissolved Pb^{2+} must be treated and not be disposed of into the environment before processing; therefore, recycling Pb^{2+} into Pb oxide is required.

In this research, the synthesis of Pb oxide was carried out using variations in the ratio of NaOH to the oxidizing agent $(NH_4)_2S_2O_8$, which would be tested for its ability to decolorize artificial RBB dyes.

2. Methods

2.1. Materials and Instruments

Lead(II) nitrate ($Pb(NO_3)_2$, Merck), sodium hydroxide (NaOH, Merck, 99%), ammonium persulfate ($(NH_4)_2S_2O_8$, Merck), remazol black B (RBB, Sigma-Aldrich), hydrogen peroxide (H_2O_2, 30%, Merck), distilled water. UV-Vis spectrophotometer (T60 UV-

Visible Spectrophotometer), SEM-EDX (Phenom Pro X), XRD.

2.2. Preparation and Synthesis of Pb Oxide from $Pb(NO_3)_2$

Pb powder (8.28 gram) of $Pb(NO_3)_2$ was dissolved in 50 mL distilled water to obtain a concentration of 0.5 M $Pb(NO_3)_2$ solution, and 1 M NaOH solution was added. After the mixture changed color from clear to milky white, the mixture was allowed to stand and filtered to get the precipitate. The dried precipitate was dissolved in 100 mL of distilled water, added with NaOH solution, which had been dissolved in 100 mL of distilled water with excess concentration (1:4) and an oxidizing agent $(NH_4)_2S_2O_8$ with a molar ratio of 1:1 with $[Pb(OH)_4]^{2-}$. The mixture changed color to blackish green. After being left for a while, the solution was filtered and dried at different drying temperatures (room temperature (desiccator), 100°C oven, 200°C oven, 300°C furnace, and 400°C furnace). The dry precipitate was used for the RBB decolorization test using the Fenton-modified method to determine the best drying temperature.

Pb Oxide was synthesized by varying the molar ratio of Pb Oxide: NaOH at 1:0.5, 1:1, 1:3, and 1:4. The dried precipitate of $Pb(OH)_2$ was dissolved in distilled water, and excess NaOH was added at a concentration ratio of 1:4 in 100 mL of distilled water. After the mixture changed color to cloudy clear, the oxidizing agent $(NH_4)_2S_2O_8$ dissolved in 100 mL of distilled water was added to the mixture. Each solution was dried using the optimum drying temperature obtained from the decolorization test using Pb Oxide with an equilibrium ratio. Each dry sample was used to decolorize the RBB dye using the Fenton method. Pb oxide with the best molar ratio in decolorizing dyes was characterized using SEM-EDX and XRD.

2.3. Decolorization of RBB dyes

Decolorization was performed using Pb Oxide with an equilibrium ratio and dried at optimal drying temperature. 0.05 g of Pb Oxide was added to 25 ppm RBB solution and 25 mL of 3% H_2O_2 reagent drop by drop. The solution was allowed to stand for 30 minutes and then filtered. The absorbance of the filtrate was measured with a UV-Vis spectrophotometer at a wavelength of 597 nm. The best temperature determination is seen from Pb Oxide, which decolorized the most optimal dyes.

2.4. Characterization using Scanning Electron Microscope and Energy Dispersive X-Ray

Characterization using SEM-EDX aims to determine the surface morphology, composition, and size of the synthesized products. The sample used for characterization was lead oxide using the drying method and the molar ratio of $Pb(NO)_3^{2-}$: NaOH, which was the most optimal in decolorizing RBB solutions.

2.5. Characterization using X-ray Diffraction (XRD)

The characterization was conducted to determine the crystallinity of lead oxide using X-rays at 2θ = 10 – 90°. The samples used for the characterization were Pb oxide using the drying method and the molar ratio of $Pb(NO)_3^{2-}$: NaOH, which was the most optimal in decolorizing RBB solutions. The results of the XRD diffractogram obtained were used to determine the lead oxide phase, the crystal system, and the structure of synthesized lead oxide.

3. Results and Discussion

3.1. Synthesis of Pb Oxide

$Pb(NO_3)_2$ (8.28 grams) was dissolved in 50 mL of distilled water to obtain a concentration of 0.5 M. The $Pb(NO_3)_2$ solution was clear in color The solution was added with $Pb(NO_3)_2$ and NaOH with a mole ratio of 1:2 under stirring. Dissolving NaOH in water will produce heat because the reaction is exothermic. The reaction that occurs can be seen in reaction (1) [3].

$$Pb^{2+} + 2\ ^-OH \rightarrow Pb(OH)_2\downarrow \quad (1)$$

Pb^{2+} reacts with ^-OH to produce a white precipitate of $Pb(OH)_2$. This white precipitate of $Pb(OH)_2$ can be converted to PbO_2 through the complex formation pathway according to the reaction (2) [3].

$$Pb(OH)_2\downarrow + 2^-OH \rightarrow [Pb(OH)_4]^{2-} \quad (2)$$

The precipitate dissolves over the reagent to form tetrahydroxoplumbate(II) because lead hydroxide is amphoteric [4]. The solution was then added with excess NaOH with a mole ratio of 1: 4. So, as much as 1.99 grams of NaOH was dissolved in 100 mL of distilled water. Adding NaOH provides sufficient hydroxide ions and an alkaline medium to precipitate Pb^{2+} as a white precipitate of lead hydroxide, $Pb(OH)_2$. This precipitate was formed because the solution was supersaturated. The formation of a precipitate was caused by the product of the mixed compound ions (Q_c) exceeding the K_{sp} $Pb(OH)_2$ value, the Q_c $Pb(OH)_2$ value = 25 x 10^{-3}, while the K_{sp} $Pb(OH)_2$ value = 2.8 x 10^{-16}. This is in accordance with the relationship between the solubility product and the relationship between the Q_c and K_{sp} values that precipitation occurs when the $Q_c > K_{sp}$ value. After that, the oxidizing agent $(NH_4)_2S_2O_8$ was added with a mole ratio of 1:1. $(NH_4)_2S_2O_8$ dissolved in 100 mL of distilled water and then mixed with $[Pb(OH)_4]^{2-}$ solution. The mixture of the three solutions produced a black-green color. The solution was allowed to stand for a few moments so that the precipitate formed completely. The reaction that takes place can be seen in Equation [4]:.

$$[Pb(OH)_4]^{2-} + S_2O_8^{2-} \rightarrow PbO_2\downarrow + 2H_2O + 2SO_4^{2-} \quad (3)$$

The solution that forms the precipitate is filtered to separate the filtrate from the precipitate. The precipitate obtained was given drying variations at room temperature (desiccator), 100°C oven, 200°C oven, 300°C furnace, and 400°C furnace. The drying variation aims to determine a drying method with optimal catalyst capabilities.

Figure 1. Synthesis of Pb oxide at an equilibrium ratio of 1:2 with variations in drying (a) desiccator (b) oven 100°C (c) oven 200°C (d) furnace 300°C (e) furnace 400°C

The results of drying variations on Pb oxide can be seen in the color difference on desiccator drying; the color of Pb oxide is darker than the other four variations. This is because it causes a change in the formed lead oxide phase at different temperatures. The increase in heating temperature can cause lead oxide to lose oxygen or the composition of oxygen in a lead oxide can be reduced, so lead oxide undergoes a phase transition.

3.2. Effect of Pb Oxide Drying Variations on RBB Decolorization

In this study, a 25 ppm RBB solution was decolorized with a fixed mass of Pb oxide of 0.05 grams. The Pb oxide used has varied drying methods, namely with room temperature (desiccator), 100°C oven, 200°C oven, 300°C furnace, and 400°C furnace, and the addition of a fixed concentration of 3% hydrogen peroxide as much as 25 mL. The method used in this decolorization process is Fenton modification because it uses H_2O_2 and modifies the catalyst used, namely lead oxide. The purpose of different drying methods was to determine the effect of the drying method on the ability of lead oxide to decolorize Remazol black B solution using the modified Fenton method. The parameters of this study were the concentration of Remazol black B solution before and after decolorization treatment and the percentage value of decolorization. In the decolorization process, an exothermic reaction occurs, which is indicated by an increase in temperature and produces bubbles. In an exothermic reaction, the enthalpy change is negative. The enthalpy reaction of H_2O_2 and PbO_2 is shown in reactions (4) and (5).

$$H_2 + O_2 \rightarrow H_2O_2 \qquad \Delta H = -411.25 \text{ kJ} \qquad (4)$$

$$Pb + \tfrac{1}{2}O_2 \rightarrow PbO_2 \qquad \Delta H = -335.2 \text{ kJ} \qquad (5)$$

After decolorization, the solution was allowed to stand for 30 minutes and then filtered to separate the filtrate and precipitate. The purpose of standing for 30 minutes is to limit the reactions that occur between RBB dyes, hydrogen peroxide, and Pb oxide. Furthermore, absorption measurements were carried out using a UV-Vis spectrophotometer at the maximum wavelength of 597 nm. Absorbance measurements for each decolorized solution were repeated three times to obtain more accurate data. The influence of the optimum temperature of the synthesized Pb oxide was observed in the RBB process.

Based on the decolorization percentage data obtained (Table 1), it can be seen that the synthesized Pb oxide can be used as an effective catalyst in the decolorization process of remazol black B using the modified Fenton method. Table 1 shows that the optimal decolorization process was obtained using Pb oxide after drying in an oven at 100°C. The lowest decolorization process came from drying with a furnace at 400°C. This is because Pb(IV) has a melting point of 290°C, so when drying is carried out at a higher temperature of 400°C, the Pb oxide phase changes [5]. Research conducted by Cao *et al.* [6] stated that when the temperature is above 160°C, PbO_2 will decompose more quickly to Pb_3O_4. Whereas, when the temperature is below 120°C, the decomposition process of PbO_2 to Pb_3O_4 will be slower [6]. This difference in the drying method also causes differences in electron structures that affect the energy levels of the electrons. The energy level of the electrons will affect the reactivity of the Pb oxide powder [7]. A one-way ANOVA test was conducted to determine the effect of different drying methods in significantly decolorizing the dye solution. The ANOVA test has a null hypothesis: the average decolorization percentage is not significantly different. The calculated F value was greater (41,184.6) than the table F value (3,478). This resulted in the null hypothesis being rejected so that it can be stated that the percentage of lead oxide decolorization given the different drying methods was significantly different.

Table 1. Percentage of RBB dye decolorization on drying variations of Pb oxide

Variation of drying	Absorbance	Decolorization (%)
Desiccator	0.240	57.36
Oven 100°C	0.198	64.73
Oven 200°C	0.304	46.47
Furnace 300°C	0.217	61.33
Furnace 400°C	0.332	41.52

In decolorizing the RBB solution using lead oxide, a spontaneous reduction-oxidation reaction between precipitated lead (PbO_2) and H_2O_2 occurred PbO_2 underwent reduction to become Pb^{2+}, whereas H_2O_2 underwent oxidation. The reactions that occur are seen in reactions (6) for reduction and (7) for oxidation.

$$PbO_2 + 4H^+ + 2e^- \rightarrow Pb^{2+} + 2H_2O \qquad E_o = +1.455 \text{ V} \quad (6)$$

$$H_2O_2 \rightarrow O_2 + 2H^+ + 2e^- \qquad E_o = -0.68 \text{ V} \quad (7)$$

$$PbO_2 + H_2O_2 + 2H^+ \rightarrow Pb^{2+} + O_2 + 2H_2O \qquad E_{ocell} = +0.775\text{V} \quad (8)$$

The reaction that takes place is a spontaneous reaction based on a positive theoretical cell potential. Based on reactions (6-8), the reaction between PbO_2 and H_2O_2 can produce Pb^{2+} ions on the surface so that Pb^{2+} will interact with H_2O_2 to produce hydroxyl radicals (•OH) which can degrade remazol black B compounds and produce new radicals so that the reaction can occur sequentially and rapidly. The PbO_2 surface is a place that can facilitate the formation of hydroxyl radicals (•OH) because Pb^{2+} is available on the surface.

Based on research conducted by Hasibuan *et al.* [8], using Pb oxide in the reaction using H_2O_2 can initiate the formation of hydroxyl radicals. The hydroxyl radical (•OH) has a high standard reduction potential value of 2.8 V. The formation scheme that occurs is shown in reactions (9) to (13).

$PbO_2 + H_2O \rightarrow PbO_2 [\bullet OH] + H^+ + e^-$ (9)

$R + [\bullet OH] \rightarrow \bullet R + H^+ + e^-$ (10)

$\bullet R + H^+ + e^- \rightarrow CO_2 + H_2O + minerals$ (11)

where R is the RBB substance molecule [8]

$Pb^{4+} + 2H_2O_2 \rightarrow Pb^{2+} + 2HO_2\bullet + 2H^+$ (12)

$Pb^{2+} + 2H_2O_2 \rightarrow Pb^{4+} + 2^-OH + 2\bullet OH$ (13)

In reaction (13), Pb^{2+} ions are oxidized to Pb^{4+} and react with excess H_2O_2 causing OH radical scavenging ($HO_2\bullet$). This process inhibits and weakens decolorization because these species are less reactive in comparison (OH) and decrease the number of radicals. The available energy from the redox reaction allows for a back-and-forth reaction of Pb^{4+}, which is formed from reaction (13) to become Pb^{2+} which can further react to form another HOO• radical. This reaction has a positive E_0 cell.

$Pb^{4+} + 2e^- \rightarrow Pb^{2+}$ $E_0 = +1.690$ V (14)

$2H_2O_2 \rightarrow 2HO_2\bullet + 2H^+ + 2e^-$ $E_0 = -1.495$ V (15)

$Pb^{4+} + 2H_2O_2 \rightarrow Pb^{2+} + 2HO_2\bullet + 2H^+$ $E_{ocell} = +0.195$ V (16)

In conditions that are not ideal, the radicals formed in reactions (12) and (13) will react again with hydrogen peroxide, Pb^{2+}, and Pb^{4+} contained in the Fenton modification method. The reactions that occur are shown in (17) to (20) [8].

$Pb^{2+} + 2HO_2\bullet \rightarrow Pb^{4+} + 2^-OH$ (17)

$\bullet OH + H_2O_2 \rightarrow 2HO_2\bullet + H_2O$ (18)

$Pb^{4+} + 2HO_2\bullet \rightarrow Pb^{2+} + 2O_2 + 2H^+$ (19)

$Pb^{2+} + 2HO_2\bullet \rightarrow Pb^{4+} + 2HO_2^-$ (20)

It can be concluded that the reaction of H_2O_2 and PbO_2 that occurs is a spontaneous reaction and a reaction that produces heat (exothermic) which is indicated by the presence of bubbles.

OH radicals produced from the reduction of oxidation of Pb^{2+} and H_2O_2 can oxidize various types of pollutants, including textile dyes, pharmaceutical waste, and other organic pollutants, in a short time. The RBB decomposition process is described through the radical scheme at the reaction (21) and (22) [8].

$3\bullet OH$ + remazol black B → intermediate product (21)

$\bullet OH$ + intermediate product → $H_2O + CO_2$ + minerals (22)

3.3. Effect of $Pb(NO_3)_2$ and NaOH Amount Ratio in Decolorizing RBB Dyes

This study aims to determine the decolorization ability of RBB solutions using the Fenton method with Pb oxide synthesized in different ratios. In the decolorization process, 25 mL of 3% H_2O_2, 100 mL of RBB dye solution with a concentration of 25 ppm, and synthesized Pb oxide powder with a ratio of 1:0.5, 1:1, 1:3, and 1:4 each as much as 0.05 grams. Pb oxide synthesis products in the ratio of under and over amounts are shown in Figure 2.

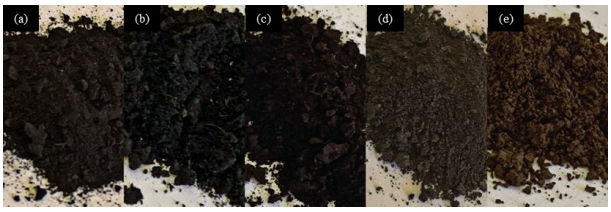

Figure 2. Pb oxide synthesis product with variations in the ratio of the amount of NaOH (a) 1:0.5 (b) 1:1 (c) 1:2 (d) 1:3 and (e) 1:4

The parameters measured in this test were the concentration of RBB solution before and after the decolorization process. In this test, 0.05 grams of Pb oxide powder was reacted with 100 mL of 25 ppm RBB sample in a beaker glass. Then, 25 mL of 3% H_2O_2 was added. The reaction is an exothermic reaction indicated by changes in temperature (heat) produced and bubbles during the decolorization process. After adding peroxide, the solution was allowed to stand for 30 minutes. Then the solution was filtered to separate the precipitate and filtrate. Furthermore, the absorbance measurement of the decolorized solution was carried out using a UV-Vis spectrophotometer at a wavelength of 597 nm. The results of the decolorization process of the RBB solution on the difference in the ratio of the amounts of $Pb(NO_3)_2$ and NaOH are shown in Table 2.

Table 2. Percentage of decolorization of the RBB solution on the difference in the ratio of the amounts of $Pb(NO_3)_2$ and NaOH

Mole Ratio	Sample concentration (ppm)	Decolorization (%)
1: 0.5	15.98	36.68
1: 1	14.03	44.41
1: 2	8.9	64.73
1: 3	6.9	72.66
1: 4	5.03	80.07

From the data presented in Table 2, the synthesized Pb oxide is effective as a catalyst in the RBB decolorization process using the modified Fenton method. Based on the data obtained in the table above, the ratio of the largest amount, namely 1:4, performs the most optimal decolorization process compared to other ratios. The excess NaOH will increase the ^-OH ion, which helps the $Pb(OH)_2$ deposition process. In the excess amount of NaOH, the decolorization activity is expected to be better because when the number of ^-OH ions is large and the pH increases, the adsorption of dye molecules by Pb oxide increases [6, 9]. When the number of OH^- ions is excessive, it is suspected that another modifier will form, which works synergistically with PbO_2.

One way ANOVA test was conducted to determine the effect of differences in the ratio of the amount of NaOH in decolorizing the dye solution significantly or not. The ANOVA test has a null hypothesis, namely the average percentage of decolorization is not significantly different. In calculations, the calculated F value is greater than the table F value. The table F value is 3.478 while the calculated F value obtained is 25258.94935.

This results in the null hypothesis being rejected so that it can be stated that the percentage of lead oxide decolorization given the different treatment of the ratio of the amount of NaOH is significantly different.

3.4. Morphology and Content Test Using SEM-EDX

The purpose of sample testing using SEM-EDX is to determine the surface morphological structure and elemental information that make up the lead oxide surface. The test sample is a synthetic product with a ratio of 1: 4 which is dried in an oven at 100°C.

The surface morphology of the synthesized Pb oxide can be seen by Scanning Electron Microscopy (SEM) testing. The SEM results of the Pb oxide sample are shown in Figure 3.

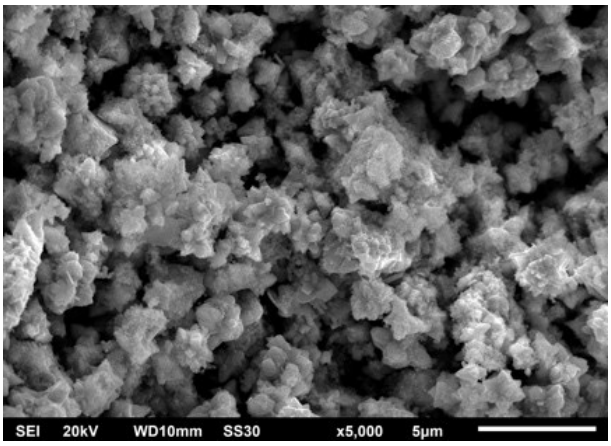

Figure 3. SEM image of Pb oxide product in ratio 1:4 with oven drying treatment at 100°C with 5000× magnification

Based on Figure 3, it can be seen that the surface morphology of the Pb oxide product with oven drying at 100°C has a porous structure. The lead oxide analyzed has an average particle size of 0.622 μm.

Testing using Energy Dispersive X-Ray (EDX) aims to determine the elemental content of the sample surface. The results of the EDX test are presented in Figure 3 and Table 3.

Table 3. Percentage composition of Pb oxide product

Contained Elements	Relative Mass (%)
Pb	90.92
O	9.08

Based on Table 3, the crystals synthesized are Pb oxides characterized by the presence of Pb and O in the crystals. The results of the EDX characterization showed that lead was the main constituent of the sample, indicated by a higher percentage than oxygen. Meanwhile, oxygen is the sample's oxide form being analyzed, indicating the resulting product is lead oxide.

3.5. X-ray Diffraction Pattern Analysis (XRD)

XRD analysis tests crystal structure, chemical elements and compounds, lattice parameters, lattice volume, and others. The test method using XRD will not damage the material to be tested [10]. The samples characterized were Pb oxide synthesis products with a ratio of 1: 4 which were treated with drying in an oven at 100°C. XRD results are shown in Figure 4. Identification of the crystalline phase of the sample was carried out by adjusting the position of the measured diffraction peaks with the available database (peak list). Adjustments are made using the Match! Software.

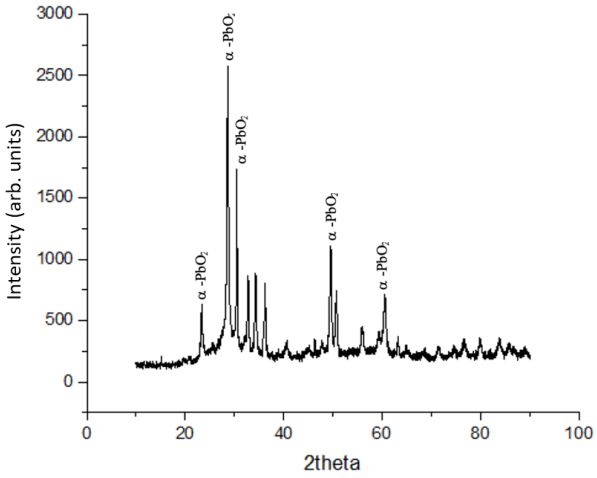

Figure 4. The results of the characterization of Pb oxide using XRD

The results of the X-ray diffraction pattern show that a crystal structure has been formed in the sample that corresponds to the material phase α-PbO_2 which is characterized by the formation of diffraction peaks with fairly high intensity at angle 2θ of 23.360 (1 1 0), 28.510 (1 1 1), 30.000 (0 2 0), 32.550 (0 0 2), 34.240 (0 2 1), 36.290 (2 0 0), 49.530 (2 0 2), 50.740 (2 2 1), 60.600 (3 1 1). These data are in line with data previously reported by Singh and Srivastava [11] that phase α-PbO_2 has a miller index value (hkl) (1 1 0; 1 1 1; 0 0 2; 0 2 1; 2 0 0; 1 1 2). Based on the XRD diffraction pattern data, it can be concluded that the synthesis product of Pb oxide is α-PbO_2 with scrutinyite mineral phase and orthorhombic structure. The data is in accordance with the report [12].

4. Conclusion

Using the oxidation-reduction method, synthesizing lead oxide from $Pb(NO_3)_2$, NaOH, and $(NH_4)_2S_2O_8$ produced dark reddish-brown lead oxide powder. Lead oxide in the oven drying variation at 100°C decolorized the best compared to the other four drying variations. The lead oxide ratio of 1:4 on drying decolorized the RBB dye solution better than other ratios. Based on surface morphology, composition, and product crystallinity tests, it can be concluded that the lead oxide obtained was α-PbO_2.

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