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Synthesis and Characterization of Electrochemical Properties of Manganese Ferrite Nanoparticles ($MnFe_2O_4$) from Iron

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Abstract
Synthesis and characterization of the electrochemical properties of manganese ferrite nanoparticles ($MnFe_2O_4$) from iron ore have been done. The aim of this research was to determine the potential of iron ore as a source for the manufacture of $MnFe_2O_4$ and to know electrochemical characterization. The precipitation method was used in this research. Iron ore samples were taken from Tanah Laut Regency, South Kalimantan, Indonesia. The sample of iron ore was purified first to synthesis $MnFe_2O_4$ nanoparticle. Manganese salt $MnCl_2$ is used as a source of manganese. Characterization of samples use TEM and potentiostat. Glucose oxidase (GOD) is used as a sample to be given electrochemical properties of the sample. The GOD concentration used is 0.2; 0.4; 0.6; and 0.8 ppm. The range of $MnFe_2O_4$ nanoparticles was successfully made with sample diameters ranging from 1.5 to 12.5 nm. The current values obtained on $MnFe_2O_4$ nanoparticles range from $0.226 - 0.322$ mA. The sensitivity of $MnFe_2O_4$ nanoparticles is around 0.16 mA/ppm. The higher the concentration used, the greater the current produced.

Keywords: manganese ferrite, co-precipitation, electrochemical, glucosa oxidase

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Introduction

In recent years, various applications of technology have used ferrite nano materials due to the unique magnetic properties and electrical properties of these materials. It is well known that the physical and chemical properties of nano-sized magnetic materials are very different from bulk due to the influence of their surface (ratio of surface to large volume) and the effect of quantum confinement (properties that depend on size). Mangan ferrite nanoparticles ($MnFe_2O_4$) are one of the nano ferrite materials that are very important because of their magnetic applications, such as recording media devices, drug delivery, ferrofluid, biosensors and catalysis [1–5].

It has been known that the $MnFe_2O_4$ material has an inverted spinel structure because 80% of Mn ions occupy the tetrahedral site (A-site), which is surrounded by four O_2 ions, and the 20% left Mn

ion occupies an octahedral site (B-site), surrounded by six O_2-ions [6]. However, manganese ferrite has a FD3M space group.

In order to overcome these difficulties and to meet the requirements for new applications, some wet chemical processes like pulse laser deposition [6], solid state reaction [7], ball-mill [8], co-precipitation technique [9], micro-emulsion process [10], hydrothermal method [11], sol-gel citrate [12, 13], solution spray [14], and the detonation of emulsion explosive [15] have been considered for the production of nanoscale ferrites.

In this research, $MnFe_2O_4$ nanoparticles were synthesized using the co-precipitation method. This study uses iron sources from iron ore from Tanah Laut Regency, South Kalimantan. The samples produced will be characterized using TEM and tested for electrochemical properties using a potentiostat. This research use Glucose Oxidase (GOD) to test $MnFe_2O_4$ nanoparticle.

Methods

The materials used were iron ore are 37% HCl (aldrich), 0.2M $FeSO_4 \cdot 7H_2O$ (Aldrich), 5% NH_4OH, (Aldrich), 0.1M $MnCl_2$ (Aldrich), aquades, aquabides, 96% alcohol. Sample preparation of iron ore is done by using hammer and permanent magnets. Iron ore is washed and then dried. The sample is destroyed using hammer. Iron ore powder is separated from impurities using a permanent magnet. After that, sieving was done using a 230 mesh filter to homogenize the powder size. The uniform powder size is then cleaned using a permanent magnet from impurities to obtain a higher level of purity.

The process of synthesis $MnFe_2O_4$ was done by co-precipitation technique. First, add 37% 100 ml HCl for every 6 g of iron ore that has been mashed, the sample is placed in a glass bottle for 1 day so that the iron ore powder becomes smoother. Then, the solution is inserted 5 ml into the glass. Samples added 0.2 M 0.4 ml $FeSO_4 \cdot 7H_2O$ into the beaker. The sample is heated at 70°C for 15 minutes, then enter $MnCl_2$ 0.1 M 0.2 gram. Samples were dripped with a solution of 5% NH_4OH until the solution turned solid black. Samples were washed using distilled water and 96% alcohol 3 times followed by ultrasonication. $MnFe_2O_4$ is dried in an oven at 70°C to dry to get a powder-shaped sample. The sample was characterized using TEM and electrochemical using a potentiostat.

Results and Discussion

The sample preparation result is shown in Figure 1. Iron ore have ±98% element of ferrite (Fe) [16]. The average crystal size of the $MnFe_2O_4$ nanoparticles produced based on Figure 2 is a range from 1.5 - 12.5 nm.

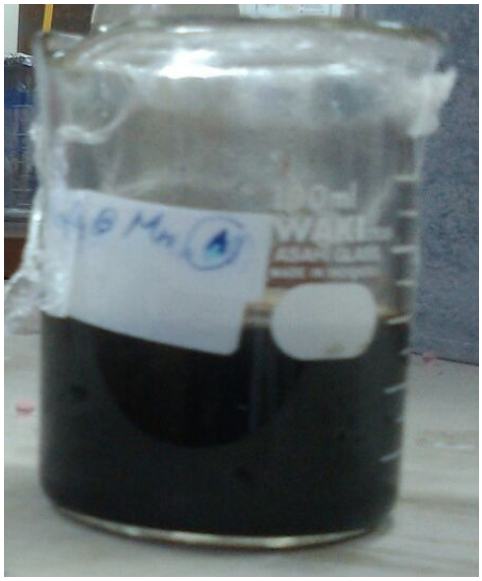

Figure 1: Sample of $MnFe_2O_4$ nanoparticle.

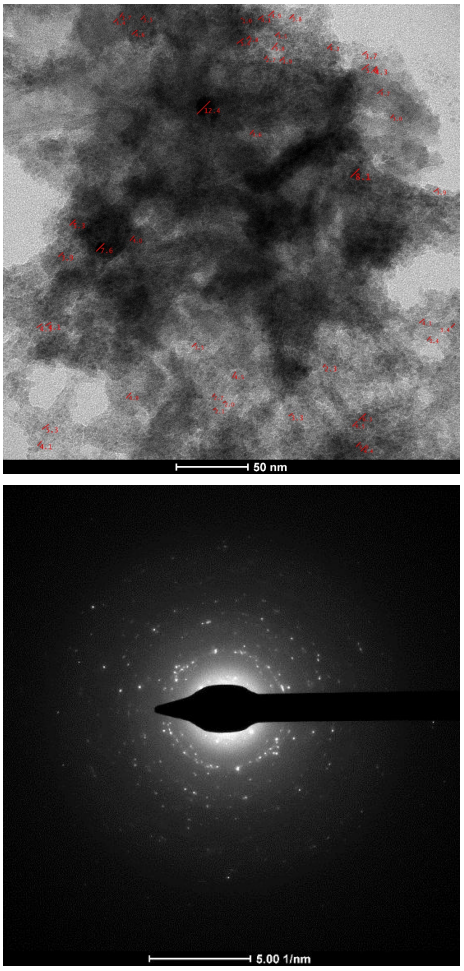

Figure 2: Results of TEM of $MnFe_2O_4$ nanoparticle.

The crystal size obtained shows that the coprecipitation method succeeded in producing ferrite manganese nanoparticles. However, $MnFe_2O_4$ still agglomerated. The result obtained is suitable with previous studies [17–20]. The resulting $MnFe_2O_4$ nanoparticles show high crystallinity (Figure 2 bottom). From the figure, there are miller indices (220), (311), (400), (333), (440).

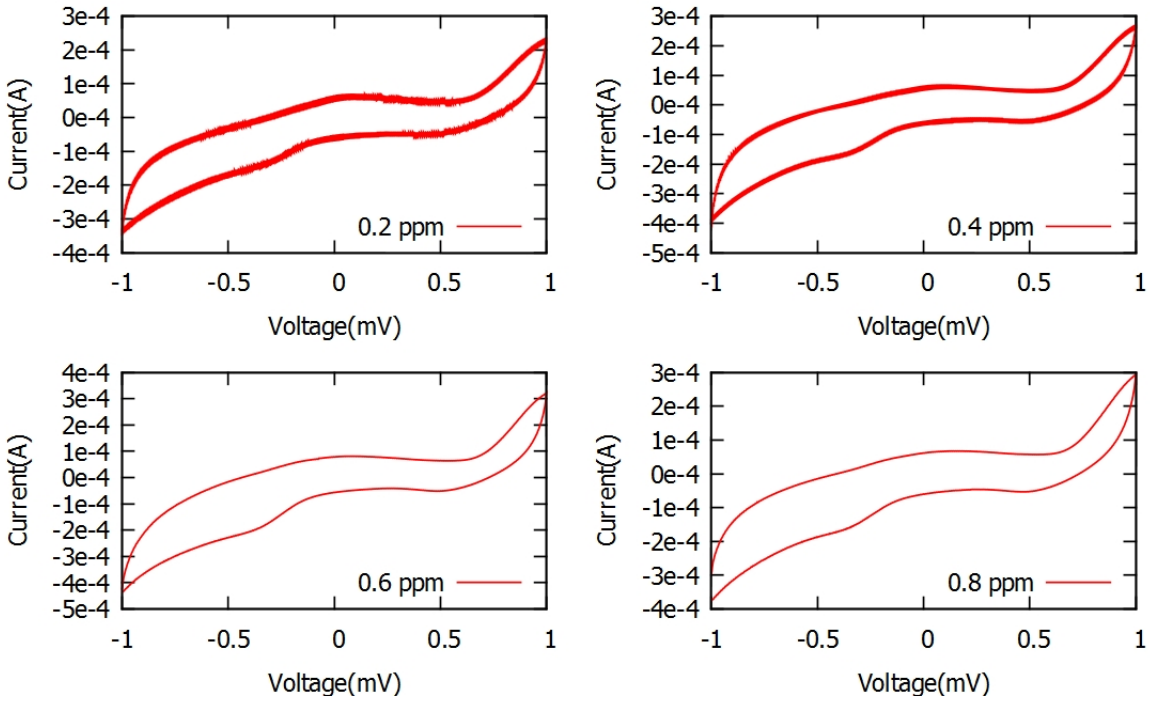

Figure 3: Results of electrochemical $MnFe_2O_4$ nanoparticle using biomaterial GOD ranging from 0.2 to 0.8 ppm.

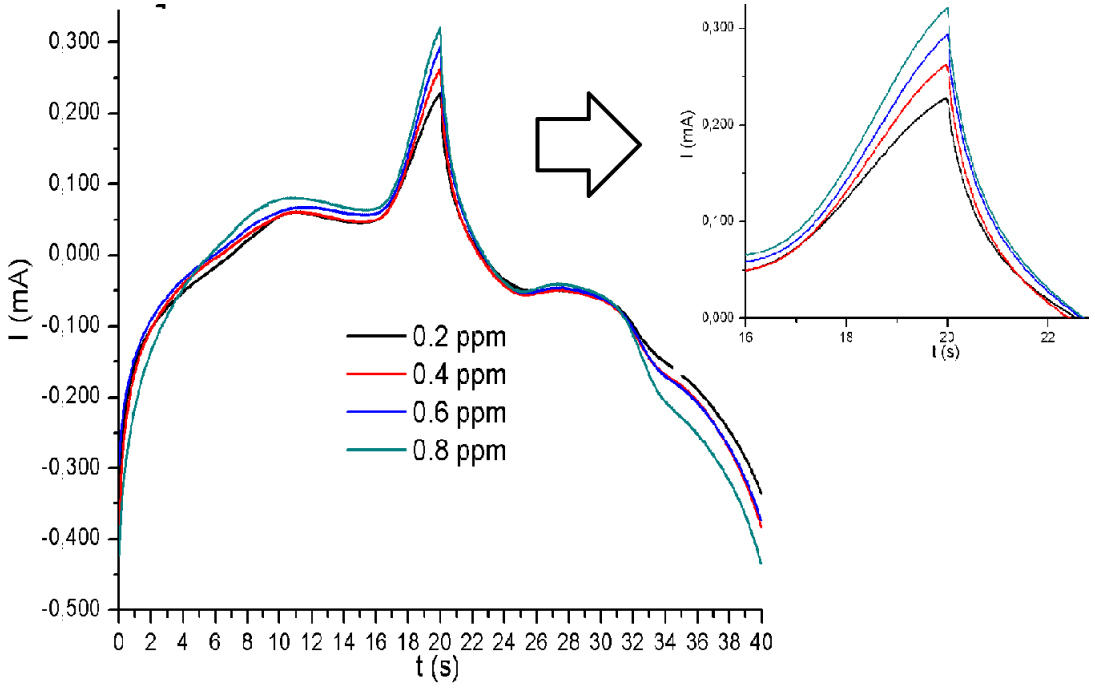

Figure 4: The currents produced by varying the concentration of GOD range from 0.2 to 0.8 ppm.

Figure 3 is the result of the cyclic voltammetry of the performance of GOD / $MnFe_2O_4$ / graphite electrodes on the effect of glucose solution concentration in the form of a cyclic voltmeter which shows that there is a peak indicating that there is a glucose oxidation reaction by the GOD enzyme. From the peak of oxidation at the cyclic voltage, we can know the value of the oxidation current from the oxidation reaction produced at each concentration. The value of the current produced is recorded as a result of the influence of glucose substrate concentration. From the oxidation current data obtained from each peak, a curve can be made with relation to glucose concentration to obtain a sensitivity value from the electrode in detecting glucose and to describe the reaction rate of the enzyme catalyst. Sensitivity is the difference between the highest and lowest glucose oxidation current values generated against the difference between the highest and lowest glucose concentrations.

Table 1: Current of concentration from GOD.

Concentration (ppm)	I (mA)	V (V)	t (s)
0.2	0.226	0.9995	20
0.4	0.263	0.9975	20
0.6	0.294	1.000	20
0.8	0.322	1.000	20

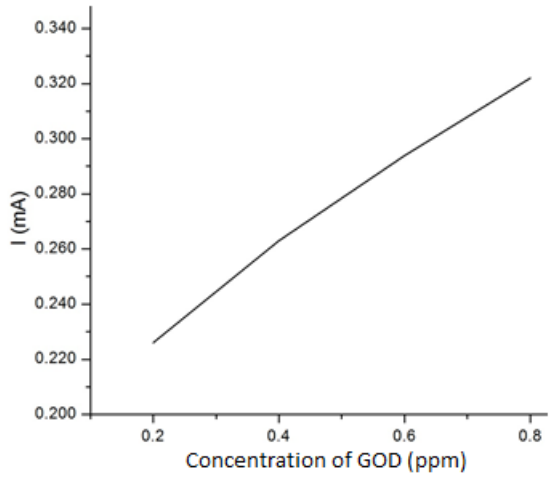

Figure 5: Relations between concentration and current in the $MnFe_2O_4$ sample.

The current value generated at a concentration of 0.2; 0.4; 0.6; and 0.8 ppm are 0.226; 0.263; 0.294; and 0.322 mA respectively (Table 1). The value of the current produced is proportional to the concentration used (Figure 5). The higher the concentration, the higher the current is obtained. The sensitivity value obtained from the performance of this electrode is 0.16 mA/ppm. This means that every increase of one ppm of glucose substrate produces a current value of 0.16 mA. The maximum time to reach the peak current occurs for 20 seconds (Figure 4).

Conclusion

$MnFe_2O_4$ nanoparticles have been successfully synthesized by ferrite originating from iron ore. The resulting particle size has a range of $1.5 - 12.5$ nm. $MnFe_2O_4$ nanoparticles have a sensitivity of 0.16 mA/ppm using the enzyme glucose oxidase (GOD). The greater the ppm used, the greater the current produced.

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