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RESEARCH ARTICLE OPEN ACCESS

Impregnation of Synthesized Fe_3O_4- Fe_2O_3Nanoparticles Mixture from Iron Sand into Mesoporous Silica

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Abstract

Impregnation of three types of synthesized Fe_2O_3-Fe_3O_4 nanoparticles mixture with different $FeCl_3$ concentrations (0,072 M, 0,036 M, 0,03 M) had been succesfully done into mesoporous silica. Mixture of nanoparticles Fe_3O_4-Fe_2O_3 in mesoporous silica that produced (NP1@SiO_2, NP2@SiO_2, NP3@SIO_2) was characterized by morphology, magnetic properties and X-Ray Diffractogram. Mixture of Fe_3O_4-Fe2O_3 nanoparticles was successfully impregmanted into mesoporous silica forming a homogenous mixture of blackish gray which can not be distuingished between mesoporous silica with the mixture of Fe_3O_4-Fe_2O_3 nanoparticles and can be pulled by a magnet. The result of characterization with XRD which shows the peak of silica at 2θ = 20.75, 25.71,Fe_3O_4nanoparticle at 2θ = 30.08, 35.43, 56.94, 62.53 and Fe_2O_3at 2θ = 33.15, 35.63, 54.05 and the size of the silica pores in the meso range.

Keywords :Mixture of *Fe_3O_4-Fe_2O_3 nanoparticles,Impregnation,Mesoporous silica*

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INTRODUCTION

Iron sand is a very abundant natural material available in Indonesia. Iron sand in general has the main composition of iron oxide (Fe_2O_3 and FeO), silicon oxide (SiO_2), and other compounds with lower levels. Iron sand is an inorganic material that can be used as a basic material for the synthesis of nanoparticles Fe_3O_4. The chemical composition of iron sand can be known after testing, for example by using XRD (X-Ray Diffraction) or XRF (X-Ray Flouresence), so that it can be used in research[2].

Iron sand that has been processed into magnetic nanoparticles has a sale value more than natural iron sand, this is because magnetic nanoparticles can be used as industrial raw materials in the electronics field, as catalysts, as materials for the use of drug delivery systems (Drug Delivery System) , Magnetic resonance imaging (MRI), the separation of heavy metals for water purification and cancer therapy [4].

Fe_3O_4Nanoparticles can be synthesized physically and chemically. Physically that is by pulling with a magnet then milling / milling using High Energy Milling-Ellipse 3D Motion to nano size. However, chemical synthesis of nanoparticlesFe_3O_4 results in irregular particle size, the purity and reactivity of Fe_3O_4 produced is lower. Whereas chemically it can be synthesized by several methods one of which is the coprecipitation method. Chemically synthesized magnetic nanoparticles have an ordered particle size and higher reactivity.

S.Aini (2019) has succeeded in synthesizing a mixture of Fe_3O_4-Fe_2O_3 nanoparticles using basic materials ofSijunjung iron sand with a size between 5-20 nm. In 2018 S. Aini, has also succeeded in synthesizing mesoporous silica from silica sand in the form of hexagonal with a pore size between 5-30 nm. The mixture ofFe_3O_4-Fe_2O_3nanoparticles and mesoporous silica obtained by Aini is different from the nanoparticles obtained by other researchers[5][3].

The chemical properties of magnetic nanoparticles (Fe_3O_4) are highly reactive with oxidator, while Fe_3O_4 nanoparticles are easily agglomerated. Thus Fe_3O_4 in a small size must be protected from air during storage and use. One way to

protect Fe_3O_4 nanoparticles is to use silica as a protector / stabilizer, the advantage of using silica is that it is resistant to acids and resistant to heat. Then protection iron oxide with silica does not reduce the catalytic activity of various chemical reactions. One way to protect Fe_3O_4 nanoparticles is by impregnation.

Based on the description above, the authors are interested in knowing about the impregnation of a mixture of Fe_3O_4–Fe_2O_3 magnetic nanoparticles that have been synthesized with various types of FeCl concentrations into the mesoporous silica above.

MATERIALS & METHODS

Materials and Equipments

The material used to impregnation is a mesoporous silica and mixture of Fe_3O_4-Fe_2O_3 nanoparticles which is synthesized from natural materials,HClp.a,NH_4OH, ethanol,NaOH 8 M, H_2O.

The equipment used in this study are, magnet, Magnetic Stirrer and characterization using X-Ray Diffraction.

Method

0.63 gram of mesoporous silica dissolved in 70 ml of ethanol + 30 ml of aquabides, added a mixture of Fe3O4-Fe2O3 nanoparticles (16% w / w of SiO2). The two mixers were stirred for 12 hours at a temperature of 30°C. The product is taken with aquabides, dried at 110°C and calcined at 550°C for 5 hours. The results obtained were characterized using XRD.

RESULT AND DISCUSSION

Impregnation of Fe_3O_4-Fe_2O_3 nanoparticles into mesoporoussilicia

Impregnation of Fe_3O_4-Fe_2O_3 nanoparticles into mesoporoussilicia was successfully done. Three types of Fe_2O_3-Fe_3O_4 nanoparticle mixture synthesized with different $FeCl_3$ concentrations (0,072 M, 0,036 M, 0,03 M) is simbolyzed with NP1, NP2, NP3. The mixture of nanoparticles that been impregmanted into mesoporous silica is simbolyzed with NP1@SiO_2, NP2@SiO_2, NP3@SIO_2. The mixture of Fe_3O_4-Fe_2O_3 was succesfullyimpregmanted into mesoporous silica forming a homogenous mixture of blackish gray which can not be distuingished betweenmesoporous silica with the mixture of Fe_3O_4-Fe_2O_3 nanoparticles, as can be seen in picture 1 below.

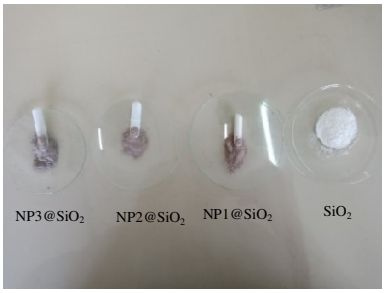

Fig 1.Mesoporous silica and NP@SiO_2picture.

The existence of Fe_3O_4-Fe_2O_3mixture in mesoporous silica can be characterized with XRD.

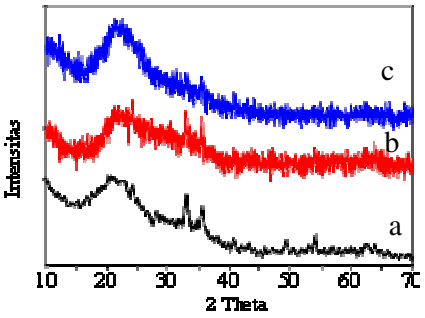

Fig 2.Fe_3O_4-Fe_2O_3diffactogram pattern of nanoparticles mixture in mesoporous silicaa.) NP1@SiO_2b.) NP2@SiO_2 c). NP3@SiO_2

The result of XRD characterization aims to examine synthesized Fe_3O_4-Fe_2O_3@SiO_2crystal structures. The data was obtained in the form of distance between fields, intensity and 2 theta angle of magnitude which then matched with X-ray diffraction pattern data JCPDS01-074-3486to SiO_2, JCPDS 01-076-8393to Fe_2O_3andJCPDS01-089-2355to Fe_3O_4so that the compounds contained in the sample can be identified. Fe_3O_4-Fe_2O_3@SiO_2has the main silica peak at 2θ = 20.75, 25.71, 49.44 and Fe_2O_3at the main peak 2θ = 33.15, 35.63, 54.05 thus Fe_3O_4withthe main peak 2θ = 30.08, 35.43, 56.94, 62.53.

As seen picture 2, the highest peak of Fe_2O_3dan Fe_3O_4at 2θ =33.15and35.43 are visible and wide in NP1@SiO_2 sample. Whereas in NP2@SiO_2 looks sharper and NP3@SiO_2 starts to be less clear the highest peak of $Fe_2O_3$and Fe_3O_4. It shows that Fe_2O_3 danFe_3O_4peaks were blocked due to mesoporous silica, the smaller the particle size, the more it enters the silica pores. NP1@SiO_2 has brownish black color due to the large particle size making it difficult to enter into mesoporus silica which causes Fe_3O_4-

Fe_2O_3 to oxidize. NP2@SiO_2 has blackish graycolor and can be pulled by magnet. Whereas, NP3@SiO_2 has darker blackish graycolor due to the smaller Fe_3O_4 particle size so that more Fe_3O_4 particles enter mesoporous silica, the magnetic attraction is higher than NP1 and NP2.

Mesoporous silica before and after impregnated with Fe_3O_4-Fe_2O_3 nanoparticles mixture hasmeso-sized pore, this can be proved with x-ray diffactogram pattern as shown in picture 3 below.

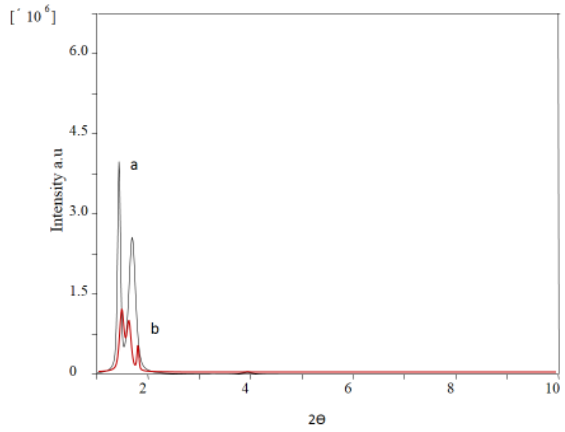

Fig 3.Small angle X-Ray Diffraction pattern mesoporous silica a.)Mesoporous silica beforeb.)Mesoporous silica after being impragnated with a mixture of Fe_3O_4-Fe_2O_3.

CONCLUSION

Based on the research, it can be concluded that 3 types of Fe_3O_4-Fe_2O_3 nanoparticles mixture had been successfully impregnated into mesoporous silica with evidence of the formation of a homogenous mixture with blackish gray color and can be pulled by a magnet. Another proof, the result of characterization with XRD which shows the peak of silica and Fe_3O_4-Fe_2O_3 nanoparticles and the meso-sized silica pore.

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