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Available online at www.sciencedirect.com ScienceDirect Materials Today: Proceedings 5 (2018) 14970–14974 www.materialstoday.com/proceedings 2214-7853 © 2018 Elsevier Ltd. All rights reserved. Selection and/or Peer-review under responsibility of 3rd International Conference on Applied Physics and Materials Applications.

ICAPMA_2017 Synthesis of nanoparticles Fe₃O₄/PEG/PPy-based on natural iron sand Pintor Simamora, C.S. Saragiha, D.P. Hasibuana, Juniastel Rajagukguka,* aPhysics Department, Faculty of Mathematics and Natural Sciences, State University of Medan, 20221, Indonesia Abstract Magnetic nanoparticles of Fe₃O₄/PEG/PPy have been synthesized from natural iron sand by co-precipitation and ultrasonic treatment method.

The nanoparticles were synthesized using HCl as solvent and NH₃ as co-precipitate. The nanoparticles synthesized at 700C in two different treatments. PPy composites were varied to obtain the magnetic properties of samples. The measurements that have been done for both samples were XRD (X-Ray diffraction) and SEM (Scanning Electron Microscopy).

The XRD results showed that the samples were having a single phase in Fe₃O₄ and lattice parameter 8.393Å. Based on the results of SEM, the nanoparticles tend to form rough spherical in shape and distribution particles of Fe₃O₄/PEG/PPy A, B, C, and D sample were around 42-50 nm, 48-63 nm, 56-67nm and 49-67 nm. © 2018 Elsevier Ltd. All rights reserved.

Selection and/or Peer-review under responsibility of 3rd International Conference on Applied Physics and Materials Applications. Keywords: Nanoparticles; Fe₃O₄;

Polyethylene Glycol 6000; Polypyrrole 1. Introduction Disposal of liquid waste types including coloring materials into an aquatic environment becomes a serious problem at this time.

Where it will obstruct ultraviolet light (UV) into the water and reduce the efficiency of photosynthesis in aquatic plants [1]. Besides that, the most of the synthetic dyes are toxic, carcinogenic (causing cancer) and mutant (genetic mutations). Compared with the type of dye anion, the type of cation is more toxic dyes caused this type easily interact with the surface of the negatively charged cell membrane so that it can easily fit into the cell, thus causing health problems.

Therefore, the waste liquid is containing the type of dye must be sterilized before being discharged into the environment. * Corresponding author. Tel.: +62812-60786247; fax: +6261-66140002. E-mail address: juniastel@yahoo.com P. Simamora et al./ Materials Today: Proceedings 5 (2018) 14970–14974 14971 Several technologies have been used to reduce/remove heavy metals from aqueous solutions such as chemical redox followed by precipitation, ion exchange, membrane processes, elektrodialysis and adsorption [2].

The main drawback of the deposition is large consumption of reagent, high mud composition and not efisien set of metal ions to used directly back. Cation exchange, membranes processes and elektrodialysis process are not economically because of their high operational costs [3]. Of these technologies, adsorption is a versatile and cost-effective technique to remove contaminants from the water so that the process is attracting attention in recent years [4].

Currently, the nanotechnology has been used in many applications such as energy utilization, industry, sensors and maintenance of the environment [5]. This technology is a quick and effective solution to overcome problems that can not be solved using conventional technology. The nanostructured materials have been widely used to absorb heavy metals from water / wastewater and has proven advantageous as an adsorbent for a very large surface area and short diffusion length, causing in a high adsorption capacity and high adsorption efficiency.

In recent years, magnetic separation technology has been widely used in the field of separation and adsorption [6]. Therefore, Fe₃O₄ can be applied in the field of the environment, especially used as adsorbent in binding heavy metals or dyes. To obtain Iron Oxide (Fe₃O₄) in the nanometer scale, then do some methods such as sol-gel, chemical solutions, sonochemical, solvothermal, freeze drying, hydrothermal [7], laser pyrolysis technique, microwave plasma [8] and coprecipitation method [9].

In this study, the manufacture of magnetic nanoparticles do with menggunakan coprecipitation method because this method uses low temperatures and very simple. Two different treatment will be done to make nanoparticles. However, with this method has the disadvantage that nanoparticles have been synthesized to be susceptible to agglomeration, to minimize polymer additions it can minimize the occurrence of agglomeration. One polymer that can be used is Polyethylen glycol (PEG 6000).

In this research, it was conducted by making nano Fe₃O₄ magnetic particles originating from iron sand in the Deli Serdang, North Sumatera. The Iron Oxide nano-particles will be synthesized with polypyrrole (PPy) with coprecipitation method and sonication [10]. Iron Oxide (Fe₃O₄) nanoparticles was combined with polypyrrole (PPy) to achieve excellent absorption.

Currently, the polypyrrole (PPy) has attracted much attention due to its unique properties such as have high electrical conductivity with relative stability good environment, non-toxic, relatively low cost and ease of preparation are advantageous in some applications. For the first time nanocomposite PPy / Fe₃O₄ used as adsorbent and effectively reduce levels of heavy metals Cr (VI).

Along with the development of nanocomposite technology PPy / Fe₃O₄ continuously developed. 2. Materials and methodology 2.1. Materials Natural iron sands taken from Buaya River in Deli Serdang, North Sumatera. Polypyrrole (PPy) received from sigma-Aldrich, Hydrochloride Acid (Merck 37 %), Amonia Solution (Merck 32%), Polyethylene glycol (PEG 6000) from Merck. Fig 1.

Synthesis Fe₃O₄ nanoparticles. 14972 P. Simmora et al./ Materials Today: Proceedings 5 (2018) 14970–14974 2.2. Synthesis of Fe₃O₄ based on natural iron sand by co-precipitation method. The Fe₃O₄ nanoparticles through co-precipitation method were prepared by following the procedure.

The first, Natural iron sand was taken from Buaya River in Deliserdang, North Sumatera. The iron sand was synthesized using 250 ml 12 M HCl, stirred for 90 minutes at 300 rpm using magnetic stirrer, then filtered using paper filter. Then the filtered iron sands was added 1 mmol of polyethylene glycol for 90 minutes. Two glasses of 100 ml 6.5

M Amonia Solution prepared by stirring with 300 rpm at 70°C. Until precipitation appeared at the bottom of both glasses. The precipitate material then was separated from the solution, washed with aquades until its pH has reached 7, dried it at 100°C for 5 hours, until Fe₃O₄ nanoparticles powder were obtained as shown in Fig. 1. 2.3.

Synthesis of Fe₃O₄/PPy nanocomposites The polymerization was carried out in a 500 mL three-necked, round-bottomed flask.

Fe₃O₄ (5 g) from the above methods (co-precipitation methods), dispersed in the 300 mL D.I. water and subjected to ultrasonic treatment for 1 h, followed by an addition of pyrrole (Py). The polymerization was initiated by an addition of FeCl₃ (66 g/L) and lasted for 8 h with stirring at 0–50°C. The product was washed by D.I.

water and ethanol for 3 times and dried in a vacuum at 55°C for 24 h. Based on the type of Fe₃O₄ and the amount of Ppy used for the preparation of the composites, Fe₃O₄/PPy nanocomposites were named as A (Fe₃O₄/PEG, 0 g of pyrrole), B (Fe₃O₄/PEG, 2.5 g of pyrrole), C (Fe₃O₄/PEG, 5 g of pyrrole), D (Fe₃O₄/PEG, 7.5 g of pyrrole). Fig. 2 shows the structure of Fe₃O₄/PPy. Fig. 2. Structure Fe₃O₄/PPy.

3. Results and discussion The synthesized nanoparticles from natural iron sand that we obtained are black in color shown in Fig. 3 (A) and responsive to external magnetic field. From Fig. 3(B) -(D) the color changes to brown-black. The structures characterization by using XRD are shown in Fig. 4.

It shows that both samples have single phase of Fe₃O₄ with cubic spinel structures, and with lattice parameter of 8.393 Å. The average particle size were estimated using Scherrer equation. $d = \frac{k\lambda}{\cos\theta} \cdot \frac{1}{B}$ (1) Where d is crystalline size, k is a Scherrer constant, λ is wavelength of the x-ray and B is full width at half maximum (FWHM).

The pattern of bare Fe₃O₄/PEG and Fe₃O₄/PEG/PPy nanocomposites were shown in Fig. 4 where measured by XRD instrument. It can be seen from the XRD pattern that Fe₃O₄ diffraction peaks have been maintained well in addition of PPy and indicates the deposition of conductive polymer layer has no negative influence on the crystalline structure of nano Fe₃O₄. The particles size calculated using Scherrer equation were 30.44 nm, 41.28 nm, 49.72 nm and 45.89 nm for A until D samples respectively.

Based on these results, it is clearly that the nanoparticles had been successfully synthesized from natural iron sands using this technique. The structural morphology of nanoparticles was observed with high resolution by the Scanning Electron Microscope (SEM). The nanoparticles tends to form rough spherical in shape. The diameter was in the range of 100 nm. As shown in Fig. 3(a)-(d). Particles size distribution of Fig.

5(a) varied around 42 nm – 50 nm, (b) varied around 48 nm – 63 nm, (c) varied around 56 nm-79 nm and (d) varied around 49 nm-67 nm. P. Simamora et al./ Materials Today:

Proceedings 5 (2018) 14970–14974 14973 Fig. 3. (A) Nanoparticles Fe₃O₄/PEG, (B) Nanoparticles Fe₃O₄/PEG/PPy 2.5 g, (C) Nanoparticles Fe₃O₄/PEG/PPy 5 g, (D) Nanoparticles Fe₃O₄/PEG/PPy 7.5 g. Fig. 4. XRD pattern of (A) sample Fe₃O₄/PEG (B) sample Fe₃O₄/PEG/PPy 2.5g, (C) sample Fe₃O₄/PEG/PPy 5g, (D) sample Fe₃O₄/PEG/PPy 7.5 g. The values are consistent with the particles size that we calculated using Scherrer equation.

Average particles size of (d) sample is smaller than the (c) sample. This is due to (c) sample was agglomeration as shown Fig. 5(c). The nanoparticles Fe₃O₄/PEG/PPy composites was also exhibited larger size without changing their spherical shapes. 4. Conclusions Magnetic nanoparticles of Fe₃O₄/PEG/PPy have been synthesized from natural iron sand by co-precipitation and ultrasonic treatment method.

PPy composites were variated to obtain the magnetic properties of samples. The XRD results showed that the samples were having a single phase is Fe₃O₄, cubic spinel and lattice parameter is 8.393Å. Based on the results of SEM, the nanoparticles tends to form rough spherical in shape and distribution particles of 14974 P. Simmora et al./

Materials Today: Proceedings 5 (2018) 14970–14974 Fe₃O₄/PEG/PPY A, B, C, and D sample were arround 42-50 nm, 48-63 nm, 56-67nm and 49-67 nm. The nanoparticles Fe₃O₄/PEG/PPy composites was also exhibited larger size without changing their spherical shapes. Fig. 5. SEM imaging of nanoparticles (a) nanoparticles Fe₃O₄/PEG,(b) nanoparticles Fe₃O₄/PEG/PPy 2.5

g, (c) nanoparticles Fe₃O₄/PEG/PPy 5 g, (d) nanoparticles Fe₃O₄/PEG 7.5 g

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