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Synthesis $NiFe_2O_4$ Nanoparticles by Co-precipitation Method for Degradation of Congo Red Dye

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Abstract

$NiFe_2O_4$ nanoparticle was synthesized using the co-precipitation method. This study done to the purpose for get the photocatalyst magnetic. The product of $NiFe_2O_4$ nanoparticles was characterized by XRD, FTIR, SEM-EDS and UV-DRS instrumentation. The result of characterization of $NiFe_2O_4$ nanoparticles was showed a sharp intensity peak at 2θ of 35.73º. The characterization using FTIR was showed the absorption band of Ni-O metal oxide at 779.24 cm^{-1} and the absorption band of Fe-O metal oxide at 694.37 cm^{-1}. Characterization using SEM-EDS showed the $NiFe_2O_4$ nanoparticles have agglomeration because the nanoparticles possess high surface energy. The composition element of $NiFe_2O_4$ nanoparticles was 47.84% O, 15.37% Fe, and 9.89% Ni. Meanwhile, the result of characterization using UV DRS was showed sharp peak intensity of wavelength at 319 nm. The band gap energy of $NiFe_2O_4$ nanoparticles with direct transition in 1.11 eV and indirect transition in 1.98 eV using the Tauc method. The small band gap energy to increases the photocatalytic activity of material. Then, the result of photodegradation toward Congo red was showed effectively at the concentration in 100 mg/L, the contact time in 25 minutes and the pH in pH 5.7 with removal effectivity was 71.104%.

Keywords: nanoparticle, congo red, photodegradation, co-precipitation

Abstract (Indonesian)

Sintesis nanopartikel $NiFe_2O_4$ telah dilakukan menggunakan metode kopresipitasi. Penelitian ini dilakukan dengan tujuan untuk mendapatkan suatu fotokatalis magnetik. Nanopartikel $NiFe_2O_4$ yang diperoleh dikarakterisasi menggunakan instrument XRD, FTIR, SEM dan UV DRS. Hasil karakterisasi $NiFe_2O_4$ menggunakan XRD menunjukkan intensitas puncak tertinggi pada sudut 2θ sebesar 35.73º. Hasil karakterisasi menggunakan FTIR menunjukkan pita serapan oksida logam Ni-O pada daerah bilangan gelombang 779,24 cm^{-1} dan pita serapan Fe-O pada bilangan gelombang 694,37 cm^{-1}. Hasil karakterisasi menggunakan SEM EDS menujukkan bahwa permukaan nanopartikel terbentuk aglomerasi karena memiliki energi permukaan yang tinggi. Komposisi unsur penyusun $NiFe_2O_4$ yaitu unsur O sebesar 47.84%, Fe sebesar 15.37%, dan Ni sebesar 9.89%. Sedangkan hasil karakterisasi menggunakan UV DRS menunjukkan intensitas serapan tertinggi pada panjang gelombang 319 nm. Energi celah pita diperoleh melalui perhitungan transisi langsung sebesar 1.11 eV dan transisi tidak langsung sebesar 1,98 eV. Energi celah pita yang kecil ini dapat meningkatkan aktifitas fotokatalitik material. Hasil uji degradasi terhadap zat warna congo red menunjukkan bahwa efektivitas degradasi terjadi pada konsentrasi 100 mg/L, dengan waktu kontak di 25 menit pada 5,7 dengan efektivitas degradasi sebesar 71,104%.

Kata Kunci: nanopartikel, Congo red, fotodegradasi, kopresipitasi

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INTRODUCTION

Organic dyes have many applications in industrial of textile, cosmetic, plastic, pulp, leather, etc. [1]. The azo dye of Congo red, is one of the organic dyes that are reactive and many used for industrial of textile. This dye is soluble in water and non-biodegradable [2] is an anionic organic dye with a benzidine structure that has molecule formula $C_{32}H_{22}N_6Na_2O_6S_2$ and weight molecule 696.99 g/mol. Congo red has changed the pigment from blue to red at pH 3.0-5.2 [3]. The chemical structure of Congo red showed in Figure 1.

Dye waste of congo red was treatment with various method, such as phytoextraction, adsorption, electro flotation, and photodegradation [4]. The degradation method was chosen because it is relatively inexpensive and easy to implement [5].

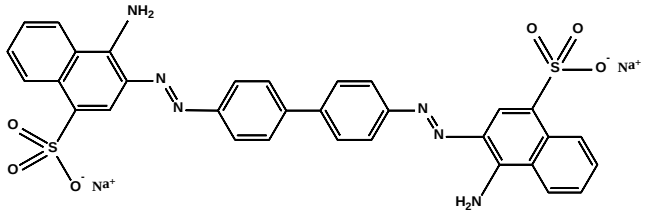


Figure 1. Chemical structure of Congo red [3]

Photocatalysis is a chemical reaction using catalyst material that activates the catalyst by lighting irradiation [6]. Photodegradation is the degradation of an organic compound to small compounds using photon energy and ultraviolet radiation [7].

The $NiFe_2O_4$ nanoparticles have one of the nanoparticles magnetic spinel ferrite. That is spinel inverse with the amount of Ni^{2+} and Fe^{3+} has same, where Ni^{2+} at the octahedral site and Fe^{3+} at the tetrahedral site, which has band gap energy 2.19 eV [8]. The $NiFe_2O_4$ nanoparticles is one of spinel ferrite that shows range a band gap in the visible light spectrum [9]. The $NiFe_2O_4$ nanoparticles also have a good biocompatibility properties, low toxicity, easy preparation, and high absorption ability [10]. Based on previous study, the $NiFe_2O_4$ nanoparticles is able to degrade of methyl orange and congo red dyes under UV irradiation [11][12].

The $NiFe_2O_4$ nanoparticles can be synthesized with any method such as co-precipitation, sol-gel, hydrothermal, powder metallurgy, and solid reaction method [13]. In this study, the method synthesis was used by the co-precipitation method using NaOH is one of a hydroxide base as deposition agent. The method of co-precipitation was chosen because can be used in low temperature. In this study the synthesis was carried out on room temperature. The principle is the precipitate of metal salt with hydroxide base, and its oxidized form was changed with heating [14].

The product of $NiFe_2O_4$ nanoparticles was characterized with XRD, FTIR, SEM-EDS, and UV DRS instruments. Then, $NiFe_2O_4$ nanoparticles were applied to photodegradation for the azo dye of Congo red with variation in concentration of congo red, time, and pH.

MATERIALS AND METHODS

Materials

All chemicals used were analytical grade, i.e. $NiSO_4.6H_2O$, $FeCl_3$ anhydrate, NaOH, HCl, $NaNO_3$, distilled water, demineralized water, Congo red, ethanol.

Synthesis of $NiFe_2O_4$ Nanoparticles [15]

$NiSO_4 \cdot 6H_2O$ 2.6259 g and $FeCl_3$ 8.1732 g were separately dissolved in 50 mL distilled water. Two solutions were mixed was added NaOH 1 M gradually and stirring up to pH 10. The solution was stirred for 60 minutes. The brown deposited sediment was washed with distilled water then dried at 75 ^oC until constant. The product of $NiFe_2O_4$ nanoparticles was calcined at 800 ^oC for 1 hour and the color was changed to black.

Characterization of $NiFe_2O_4$ nanoparticles

The product of $NiFe_2O_4$ nanoparticles was characterized with XRD PAN analytical X'pert PRO, FTIR Prestige 21 Shimadzu - Japan, SEM-EDS JEOL JSM 6510 (LA) and UV DRS by Pharmaspec UV-1700. The XRD analysis was observed at a wavelength of 1.5406 Å using voltage 40kV and generator current at 35 mA with Cu as anode.

The diffraction measurement taken at angle range 2θ= 10 until 90 at read speed 0.02 per second. The diffraction data are indexed with standard data JCPDS No. 10-0325 for $NiFe_2O_4$ nanoparticles. The crystal size of $NiFe_2O_4$ nanoparticles was determined using the following Scherrer equation (1):

$$D = \frac{k\lambda}{\beta \cos \theta} \quad (1)$$

Where D is the average crystal size (nm), k is a constant (0,9), λ is the wavelength of the X-Ray diffraction (1.5406 Å), β is the full width at half maximum, and θ is the diffraction angle [11][16].

The FTIR analysis to see band adsorption of metal oxide and functional group in $NiFe_2O_4$ nanoparticles. The SEM EDS analysis to see of morphology and composition element of $NiFe_2O_4$ nanoparticles. The UV DRS analysis to see band gap energy of $NiFe_2O_4$ nanoparticles.

Application of $NiFe_2O_4$ Nanoparticles to photodegradation of Congo Red

The wavelength measurement of Congo red observed at range 400-600 nm using Spectrophotometer UV-Vis Orion Aquamate 8000 and the absorbance measurements were carried out at the maximum wavelength is known. The measurement of pH*pzc* observed using solution of $NaNO_3$ with initial pH set from 2-12, then add 0.2 g of $NiFe_2O_4$ nanoparticles and stirred during 2 hours. Then, the final pH measured after the solution saved for 2 days.

The $NiFe_2O_4$ nanoparticles 0,05 g entered in beaker glass containing 25 mL congo red solution with variation of concentration at 50 ppm, 75 ppm, 100 ppm, 125 ppm, and 150 ppm. The solution is stirred using magnetic stirrer and pH is set according to pH_{PZC} under ultraviolet radiation 20S Watt during 60 minutes. The solution separated from nanoparticles using magnet permanent.

The best condition obtained to used next procedure with time variation at 10, 15, 20, 25 dan 30 minutes. Then, the best condition at concentration and time to use for procedure with pH variation at 3-8 with added HCL 0,1 M or NaOH 0,1 M.

Data Analysis

The absorbance data obtained were used to determine the effectivity degradation of $NiFe_2O_4$ nanoparticles. Based on the standard curve, it is then used to calculate degradation with the following formula (2):

$$\text{Removal efficiency (\%)} = \left(\frac{C_o - C_t}{C_o}\right) \times 100 \qquad (2)$$

Where C_o is the initial concentration, C_t is the final concentration of organic dye.

RESULTS AND DISCUSSION

Synthesis of $NiFe_2O_4$ Nanoparticles

The synthesis of $NiFe_2O_4$ nanoparticles using the co-precipitation method is mixed nickel sulfate and iron (III) chloride. The product of $NiFe_2O_4$ nanoparticles is brown powder. The substitution reaction formation of $NiFe_2O_4$ nanoparticles was shown as follows:

$$NiSO_4.6H_2O_{(aq)} + 2NaOH_{(aq)} \rightarrow Ni(OH)_{2(aq)} + Na_2SO_{4(aq)} + 6H_2O_{(l)} \qquad (3)$$

$$Ni(OH)_{2(aq)} \rightarrow NiO_{(aq)} + H_2O_{(l)} \qquad (4)$$

$$2FeCl_{3(aq)} + 6NaOH_{(aq)} \rightarrow 2Fe(OH)_{3(aq)} + 6NaCl_{(aq)} \qquad (5)$$

$$2Fe(OH)_{3(aq)} + NiO_{(aq)} \rightarrow NiFe_2O_{4(s)} + 3H_2O_{(l)} \qquad (6)$$

$$NiSO_4.6H_2O_{(aq)} + 2FeCl_{3(aq)} + 8NaOH_{(aq)} \rightarrow NiFe_2O_{4(s)} + Na_2SO_{4(aq)} + 6NaCl_{(aq)} + 10\ H_2O_{(l)} \qquad (7)$$

Characterization of $NiFe_2O_4$ Nanoparticles

The X-ray diffraction pattern of $NiFe_2O_4$ nanoparticles these Bragg's reflection peak as shown in Figure 2.

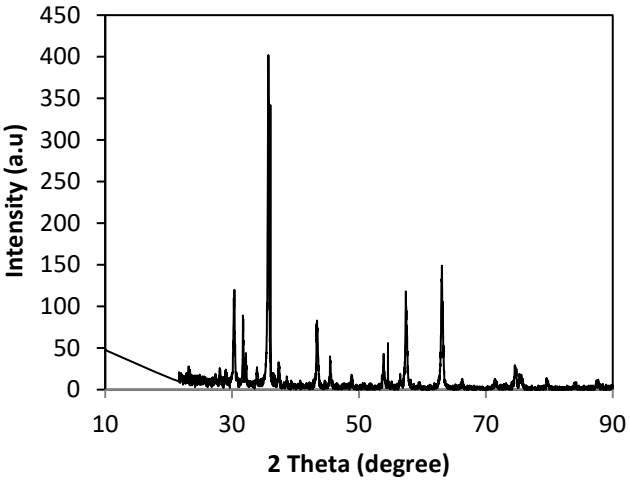


Figure 2. XRD pattern of $NiFe_2O_4$ nanoparticles

All the peaks are indexed with the standard JCPDS (Joint Committee for Powder Diffraction Standard) No. 10-0325 at 2θ of 30.32, 35.81, 37.27, 43.45, 53.89, 57.41, and 62.96°, respectively [17]. The high intensity diffraction peaks in diffractogram at 2θ of 30.32, 35.73, 37.36, 43.39, 53.86, 57.39, and 63.04, respectively. All the peaks data are similarities with standard data with a slight shift. The highest intensity diffraction peak at 2θ of 35.73° use for calculated of crystal size of $NiFe_2O_4$ nanoparticles. The crystal size was obtained using Scherrer equation is 80.52 nm.

The FTIR spectrum of nanoparticle $NiFe_2O_4$ was observed at the wavenumber range 4000-400 cm^{-1}, as shown in Figure 3.

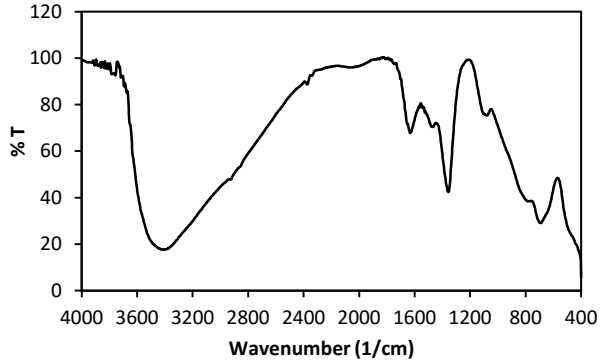
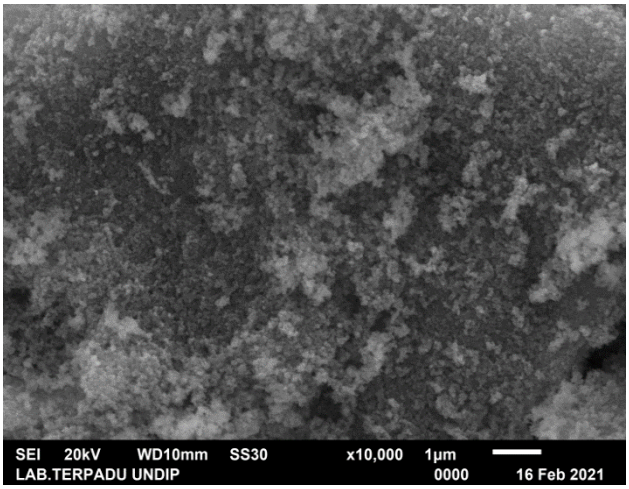


Figure 3. FTIR spectra of $NiFe_2O_4$ nanoparticles.

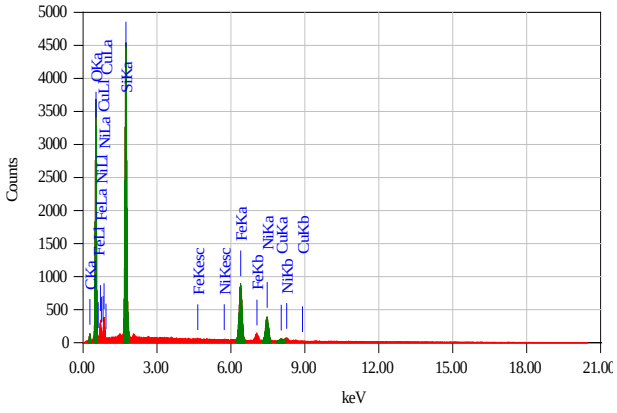
The FTIR spectrum have frequency absorption bands in wavenumber of 694.37; 779.24; 1080.14; 1357.89; 1473.62; 1629.85 and 3410.15 cm^{-1}. The vibration bands of metal oxide functional of spinel

ferrite are observed above wavenumber 1000 cm^{-1} and functional group in wavenumber at 1000-4000 cm^{-1} [12]. The absorption band located at 694.37 cm^{-1} possesses high-intensity is band of Fe-O metal oxide, and the absorption band located at 779.24 cm^{-1} with low intensity is band of Ni-O metal oxide. The absorption band at 3410.15 cm^{-1} is the bending vibration of the O-H functional group from absorbed water [11]. It shows the absorption band around wavenumber 650-700 cm^{-1} and absorption band around wavenumber 750-800 cm^{-1} is structure of metal ferrite [12].

The SEM analysis to determined morphology and the size distribution with 1000x magnification. Based on images in Figure 4, the surface of nanoparticles $NiFe_2O_4$ has uniform pore size in color white and formed agglomeration because the nanoparticle possess high surface energy [18]. The pose size using Image J application is 65.14 nm. So that, this confirmed of nanoparticles size which <100 nm.



(a)



(b)

Figure 4. (a) SEM image and (b) EDS spectra of $NiFe_2O_4$ nanoparticles.

Table 1. Element composition in $NiFe_2O_4$ nanoparticles

Element	Mass (%)	Atom (%)
C	7.70	3.52
O	47.84	53.07
Si	18.32	3.76
Fe	15.37	25.81
Ni	9.89	13.55
Cu	0.88	0.29
Total	100	100

The EDS spectra showed that the peak of O, Ni, and Fe have high intensity. Then, EDS data was shown in table 1. The element composition of $NiFe_2O_4$ nanoparticles was 47.84% O, 15.37% Fe, and 9.89% Ni. The composition atom of $NiFe_2O_4$ nanoparticles was 53.07% O, 25.81% Fe and 13,55% Ni. This composition was confirmed of ratio in structure of $NiFe_2O_4$ nanoparticles is successful synthesis. Based on atomic ratio from EDS data, this result was corresponds to the atomic ratio in molecule formula of $NiFe_2O_4$ nanoparticles which is 1:2:4 [19].

The UV-Vis spectra of $NiFe_2O_4$ nanoparticles was shown in Figure 5. The peak of highest absorbance in wavelength at 319 nm. The band gap energy of $NiFe_2O_4$ nanoparticles was calculated using the Tauc method. The graph was plotted between *hv* versus ahv^2. The band gap energy of $NiFe_2O_4$ nanoparticles was 3,15 eV. This band gap is wider than the result obtained in same method of co-precipitation in lower temperature, in range 1.68 and 1.70 eV [12] and sol-gel method in 1.97 eV [11]. The bandgap value of nanoparticles was affected by various factors, such as preparation method, calcination temperature, crystallite size, lattice parameter and impurities present in the sample [19]. The band gap energy of nanoparticles was affects the visible light absorption at the photocatalytic activity of material [8].

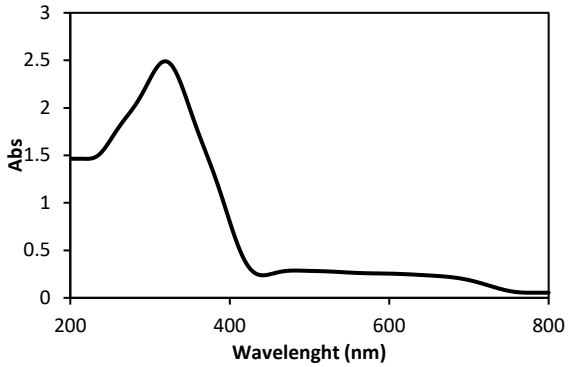


Figure 5. UV-Vis spectra of $NiFe_2O_4$ nanoparticles.

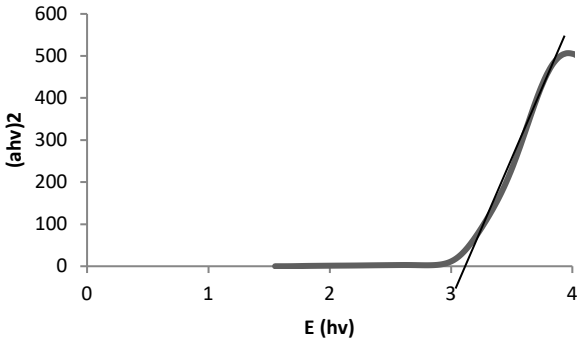


Figure 6. Band gap energy of $NiFe_2O_4$ nanoparticles

Application of $NiFe_2O_4$ Nanoparticles to photodegradation toward azo dye of Congo Red

The product of $NiFe_2O_4$ nanoparticles was determined value of pH*pzc* to known optimum condition of $NiFe_2O_4$ nanoparticles to degrade of dye. pH *point zero charge* (pH*pzc*) is condition where charge of nanoparticle or composite in neutral state. From the measurement of initial pH and final pH obtained the intersection with plotted between initial pH and ΔpH. The graph of $NiFe_2O_4$ nanoparticles showed at Figure 7.

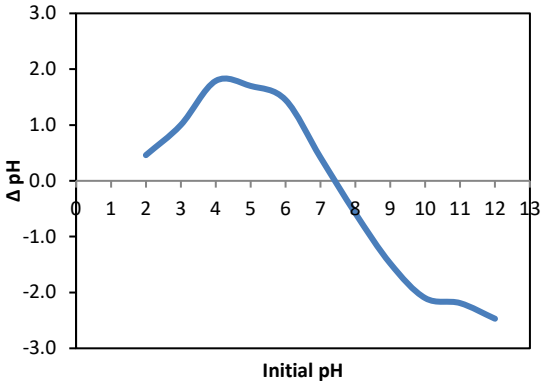
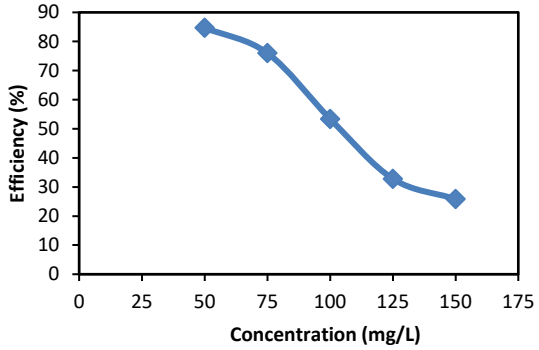


Figure 7. pH*pzc* of Congo red

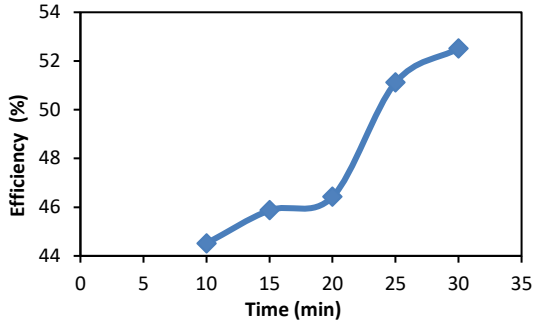
From the graph obtained the value of pH*pzc* of $NiFe_2O_4$ nanoparticles around at pH 7,4. The value of pH*pzc* the next to use for application for degradation of Congo red. If pH used below pH*pzc*, the surface of $NiFe_2O_4$ nanoparticles have positive charge and this condition is effective for interaction with Congo red which is the anionic dye. Whereas, if pH is above pH*pzc*, the surface of $NiFe_2O_4$ nanoparticles has a negative charge, there is repulsion and decreased removal effectivity [16].

The measurement absorbance of Congo red was observed at wavelength range 400-600 nm using spectrophotometer UV-Vis. From this measurement obtained of optimum wavelength at 498 nm.

The determination of optimum condition of Congo red was observed with 3 variables, variation of concentration, time, and pH. The removal efficiency of Congo red that showed at Figure 8.



(a)



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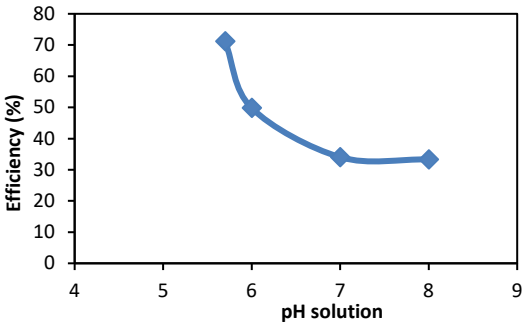

(c)

Figure 8. Removal effectivity of Congo red with the variation of concentration (a), time (b) and pH (c)

Based on the graph obtained, the removal efficiency for Congo red decreased when concentration increases. This showed that the increase of concentration should the followed to increase the amount of $NiFe_2O_4$ nanoparticles used for photodegradation. It because, the concentration is inversely proportional to the decrease of removal effectivity. So that, the same amount of $NiFe_2O_4$ nanoparticles is not enough to degrade the dye if the concentration get increase. The

decrease of removal effectivity was quite significant at concentration 75 to 100 mg/L, so the next procedure used concentration at 100 mg/L, because at this concentration is shows the differences was clear of the removal effectivity compared to concentrations of 50 and 75 mg/L.

The increase of removal effectivity quite significant of variation time at 25 minutes. There is a clear difference in increase of degradation at the 20th minute and the 25th minute. This showed that the long of contact time the followed by increase of the removal effectivity. Then, the optimum condition used to procedure of variation of pH that set from pH of 3-8. In this study, the only use that natural pH of dye at 5.7, 6, 7 and 8. It because, when the add amount of HCl in dye solution run into change the color of dye so that the process of degradation cannot to do. The absorption of dye is not in range wavelength of congo red if the solutions was changed the color. Based on graph in Figure 7c, the increase of pH the followed of decrease of removal effectivity. It because, the $NiFe_2O_4$ nanoparticles have a negatives charge when the pH solutions of congo red above pH*pzc*. So that, there is a repulsion between congo red solutions and $NiFe_2O_4$ nanoparticles caused by the congo red is an anionic dye. The decrease quite significant at pH 5.7 to 6. So that the optimum condition for process of degradation at the pH 6 or natural pH of dye.

CONCLUSION

The synthesized of $NiFe_2O_4$ nanoparticle with co-precipitation method has been successful. In this study using bottom up method, where is one of synthesis method of nanoparticles. The result of characterization using XRD showed highest intensity diffraction peak at 2θ of 35.73º use for calculated the crystal size and obtained 80.52 nm. The characterization using FTIR was showed absorption band at 694.37 cm^{-1} of Fe-O metal oxide and the absorption band at 779.24 cm^{-1} is band of Ni-O metal oxide. From the SEM image, the morphology of material have agglomeration because the nanoparticle possess high surface energy. The EDS data was confirmed of ratio in structure of $NiFe_2O_4$ nanoparticles is successful synthesis with element composition of $NiFe_2O_4$ nanoparticles was 47.84% O, 15.37% Fe, and 9.89% Ni. Band gap energy of $NiFe_2O_4$ nanoparticles with direct transition in 1.11 eV and indirect transition in 1.98 eV using Tauc method. Then, the result of photodegradation of Congo red effectively at concentration in the contact time in 25 minutes and pH in 6 or pH natural of dye.

ACKNOWLEDGMENT

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