Eksakta : Berkala Ilmiah Bidang MIPA
VOLUME 22 NO 04 2021, pp 238-247
ISSN : Print 1411-3724 — Online 2549-7464
DOI : https://doi.org/10.24036//eksakta/vol22-iss4/284

Eksakta
Berkala Ilmiah Bidang MIPA
http://www.eksakta.ppj.unp.ac.id/index.php/eksakta

Article

Preparation and Characterization of Magnetite Fe_3O_4-Activated Carbon Composite as Adsorbent Cr(VI) Ion

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Article Info

Article history :

Received September 5, 2021
Revised September 12, 2021
Accepted September 12, 2021
Published December 30, 2021

Keywords :

Magnetite, activated carbon, Cr(VI), adsorption, toxic metal

Abstract. Metal ion such as Cr(VI) is one toxic heave metal at the trace level, thus it is necessary to remove metal ion Cr(VI) ion environmental. The absorption of Cr(VI) ions was carried out using a Fe_3O_4 magnetite activated carbon composite. Activated carbon prepare from palm shells (*Elaeis guinensis Jack*) was composite with magnetite Fe_3O_4 by co-precipitation method. Magnetite Fe_3O_4-activated carbon composite is characterized by SEM, VSM and XRD. SEM image show that Fe_3O_4 a deposit on the surface of activated carbon. The degree of magnetization of Fe_3O_4 with VSM obtained 20.99 emu/g. The pattern of XRD diffractogram show that diffraction peak at 2θ which was 6.5495°; 30.1146°; 35.3581°; 43.063°; 57.1369°; 62.5918°. The spectra of FTIR show that functional groups exist in composites such as carboxyl, carbonyl and hydroxyl groups. Adsorption of Cr(VI) ion occurs at pH 5, contact time is 30 minutes and a maximum concentration of 200 mg/L Cr(VI) with a maximum adsorption capacity was 43.4 mg/g.

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1. Introduction

The rapid development of the industry and the increase in population contributed greatly to the pollution of heavy metals in the environment [1]. Pollution caused by heavy metals is the main concern in this time. Chromium is a metal that widely used in several industries. Chromium basically exists in two forms namely chromium (VI) and chromium (III). Chromium (III) is proved to be biologically important for mammals because it maintains glucose, lipids and protein metabolism. Conversely, chromium (VI) can diffuse as Cr_2O_4 or $HCrO_4$ through cell membranes and oxidized biological molecules with toxic results. Cr(VI) is a toxic metal even on parts per billion level. Anthropogenic sources of Cr(VI) metal ions in environmental samples derived from seepage or electroplating waste disposal, metal crafts, nuclear power, leather tanning, photography and the textile industry [2]–[11].

The influence of Cr(VI) metal on chronic conditions can cause dermatitis on the skin, corrosive and local effects can occur in the lungs [11]. Chromium (VI) is one of the metals that can considered one of toxic pollutants because it is most toxic, carcinogenic and mutagenic form for living organisms. In addition, it leads to liver damage, pulmonary congestion and causes skin irritation resulting in ulcer formation [5].

Several methods of removing metals in waste have been carried out including adsorption, ion exchange and separation with membranes. The adsorption process is more widely used in industry because it has many advantages, namely it is more economical and does not cause toxic side effects and is able to eliminate organic materials by utilizing its properties as an adsorbent [11]. The efficiency and cost effectiveness of the adsorption process lies in the selection of particle adsorbents [12]. Activated carbon is one of the inexpensive adsorbents for removing organic and inorganic pollutants from wastewater [13].

The traditional method for separating former adsorbents is by filtration. However, it has a weakness that is causing blockage of the filter and loss of carbon in the mud [5][14]. In recent years, separation technology using magnetite has been widely used in the fields of separation and adsorption [15][16][17]. Separation using magnetite has become one of the promising techniques for environmental water purification. Magnetic solid phase extraction (MSPE) is a technique that has received substantial consideration in the separation and recovery of metals due to the many advantages it offers compared to conventional methods. This technique is based on the extraction of different analyte from solution using a solid adsorbent with magnetic properties Applied magnetite activated carbon composites for adsorption of contaminants in water, has been done to adsorption organic wastes such as phenol, chloroform and chloro-benzene with high adsorption capacity [10].

Rossier et al have synthesized magnetite cobalt nanoparticles coated with a thin carbon shell (Co@C NPs) using a scalable method based on hydrogen reduction synthesis. The carbon shell of these cobalt nanoparticles serves as a guard to prevent the release of cobalt into water at neutral pH and contains oxidized components that promote flexible functional groups and further surface modification The particles are used for the adsorption of gold ions from solution [18]. Ghasemi et al have synthesized Fe_3O_4 nanoparticles modified with the functional group with ethylene diamine tetra acetic acid (EDTA) to produce new super-paramagnets. The adsorbent is used to remove Ag(I), Hg(II), Mn(II), Zn(II), Pb(II) and Cd(II) from different samples of wastewater and soil [19]. Anis Ariyani, 2018 has conducted research on the manufacture of magnetite Fe_3O_4-activated carbon composite with a ratio of iron oxide: activated carbon 1: 1, 1: 2 and 1: 3. The adsorbent used for the adsorption of Cs and Sr [2], preparation and characterization magnetite Fe_3O_4 nanoparticle have studied [20]. Nedhaji, 2013 has made activated carbon made from corncob biomass and magnetized by magnetite nanoparticles (MCCAC) is used for the adsorption of hexavalent chromium from aqueous solutions [21].

In this research, the preparation of activated carbon from palm shell waste and activated physically and chemically. Carbon is activated by H_3PO_4 10% and activated carbon is mixed with magnetite Fe_3O_4 by co-precipitation using a mixture of $FeCl_3$ and $FeSO_4$ to form a composite and composite adsorbent used for adsorption of Cr(VI) ion.

2. Experimental Section

2.1. Materials and Equipment

Materials is used in this experiments; Palm shells, H_3PO_4(Merck), aquabides, $FeCl_3.6H_2O$ (Merck), $FeSO_4.7H_2O$ (Merck), NaOH (Merck), HNO_3(Merck), ammonia solution 30% were purchase from Merck with analytical grade. Equipments is used; oven, standard set of glassware, furnace, analytical balance, desiccator, whatman filter paper, Jaw Crusher, Pulverizer, Magnetic Stirrer Sieving Shaker, 100 mesh sieve, UV-Vis spectrophotometer, X-Ray Diffraction (XRD, Hitachi), Vibrating Sample Magnetometer (VSM, Hitachi), and Scanning Electron Microscope (SEM, Hitachi).

2.2. Preparation of Activated Carbon.

Activated carbon was made from palm shells taken from the Palm Oil Mill in Bulian, Batang Hari Regency Province of Jambi, Indonesia. The palm shell is washed thoroughly dried at a temperature of 105 °C, crushed with a grinder and ground with a milling ball until smooth, sifted with a particle size of 100 um. Carbon is activated by 10% H_3PO_4 for 24 hours with a ratio of 1 : 2, and after mixing then washed with distilled water, and dried at 105 °C for 2 hours in the oven. Activated carbon in pyrolysis by flowing N_2 gas at 500-700 °C for 2 hours, activated carbon is cooled at room temperature and sieved with a particle size of 100 um.

2.3. Preparation of Magnetite Fe_3O_4-Activated Carbon Composite

The preparation of magnetite Fe_3O_4–activated carbon composites following method [5]. Activated carbon composites are prepared by mixing 6.5 grams of activated carbon with 400 ml of an iron salt solution consisting of 7.8 grams (28 mmol) $FeCl_3.6H_2O$ and 3.9 grams, (14 mmol) $FeSO_4.7H_2O$ added to the mixture at 70 °C. The mixture is then stirred for 30 minutes before adding 100 ml of 5M NaOH drop wise to obtain a black precipitate composite. The composite formed was washed with demineralized water and then dried in an oven at 100 °C for 2 hours. After cooling, the resulting magnetic adsorbent is repeatedly washed with water until the pH becomes neutral. The magnetite Fe_3O_4 activated carbon composite was separated using simple magnetic, dried and stored in an airtight container.

2.4. Characterization of Activated Carbon Magnetite Fe_3O_4 Composite.

The activated carbon-magnetite Fe_3O_4 composite was characterized surface morphology by Scanning Electron Microscope (SEM), X-ray Diffraction (XRD). The magnetic properties of the magnetite activated carbon (MAC) composite were characterized by a Vibrating Sample Magnetometer (VSM). The functional group of surface adsorbent was determined by FTIR and the analysis of Cr(VI) metal ion when the equilibrium is measured with AAS.

2.5. Adsorption Procedure.

The Adsorption procedure of Cr(VI) is carried out a batch technique. The amount of each adsorbent was set at 0.1 g, and the volume of the Cr(VI) solution was set at 20mL. The initial concentration of Cr(VI) solution is from 25-200 mg/L, adsorption is carried out at 293 °K for 15-90 minutes, the pH of Cr(VI) solution is adjusted with 0.1M HNO_3 or 0.1M NaOH solution is carried out when we investigate the effect of pH on adsorption of Cr(VI) at pH 2-6. The amount of Cr(VI) ion was adsorption at time t (Q_t) is calculated by the equation:

$$Q_t = \frac{Co-Ce}{m} . V$$

C_o = initial Cr(VI) concentration (mg/L)
C_e = Cr (VI) concentration at time t (mg/L)
V = volume of Cr(VI) solution (mL)

m = mass of adsorbent (g)

3. Results and Discussion

1. Characterization of Magnetite Fe_3O_4-Activated Carbon (MAC) Composite.

Structure and morphology magnetite Fe_3O_4-activated carbon composite was carried out by XRD and SEM. Based on the XRD pattern of magnetite, the XRD peak can be indexed diffraction peak at $2\Theta = 6.5495; 30.1146; 35.3581; 43.0631; 57.1369; 62.5918$ with the maximum peak at the peak of 35.33581. The peaks produced in this study have similarities or values close to the typical peak of PCPDFWIN Number 11-0614. Which shows the cubic Fe_3O_4 phase on the AC surface. The XRD pattern of magnetite Fe_3O_4-activated carbon can bee seen Figure 1.

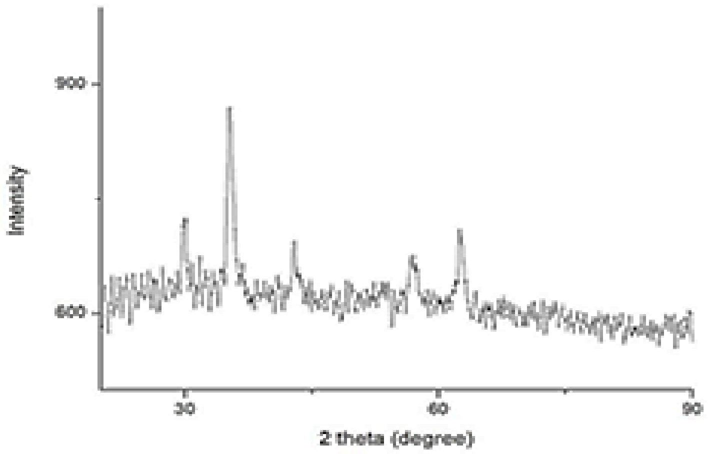


Figure 1. The pattern XRD magnetite activated carbon composite.

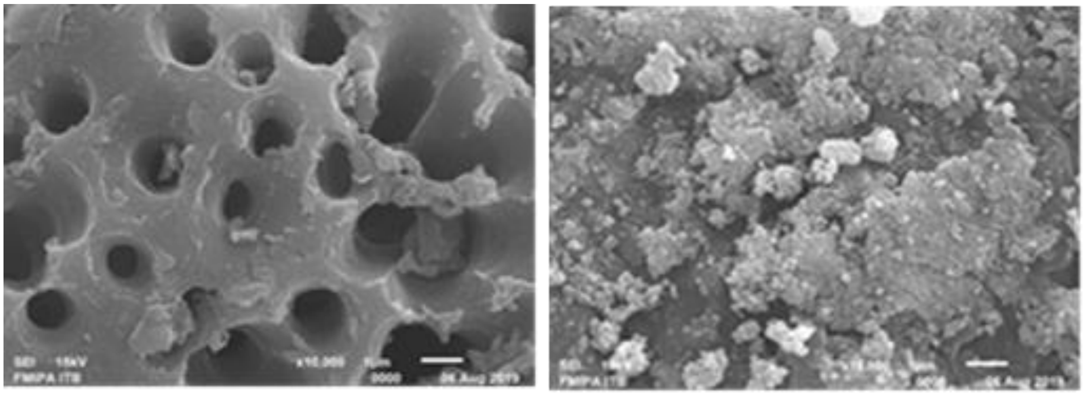
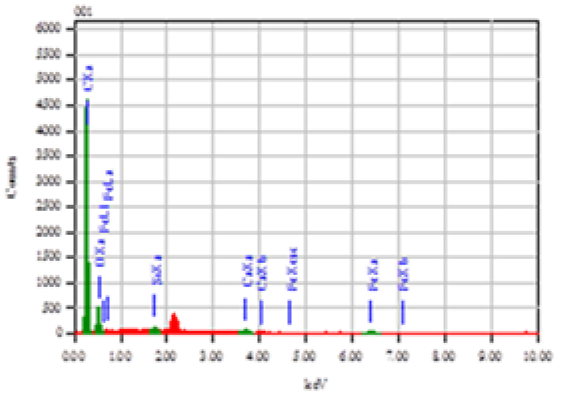

a b

Figure 2. Image morphology surface of activated carbon (a) and magnetite Fe_3O_4-activated carbon (b) 10,000x magnification.

The surface micrographs activated carbon (AC) and magnetite Fe_3O_4-activated carbon composite (MAC) are shown in Figure 2a and 2b. The surface morphology of activated carbon (2a) and the magnetite distribution of nanoparticles on the surface of activated carbon (2b). From Figure 2a and 2b can be seen that some of the magnetite particles have stuck to the surface of the activated carbon powder. The surface of the AC has an irregular shape with an average particle size of about a few micrometers [22] whereas magnetite particles tend to form a spherical configuration covering the activated carbon. The external surface of activated carbon is full of cavities as reported [23]. The morphology of the spherical to cubic magnetite Fe_3O_4 activated carbon is seen on the surface of activated carbon. Similarly, spherical magnetite nanoparticles were have been reported [23][10]. In previous studies it has been reported that the surface morphology of activated carbon adsorbents and activated carbon magnetite, where the activated carbon surface has very large pores, this can be seen in the image with a magnification of 5000x, while in activated carbon magnetite the pores are slightly closed, this is due to presence of magnetite particles attached at the surface of the magnetite activated carbon, with a magnification of 5000x it can be seen that the Fe_3O_4 magnetite particles are white to gray in color [22]. The results of elemental analysis measured by EDX can be seen in Figures 3a and 3b, while the elemental composition contained in activated carbon and MAC before and after adsorption can be seen in Table 1.



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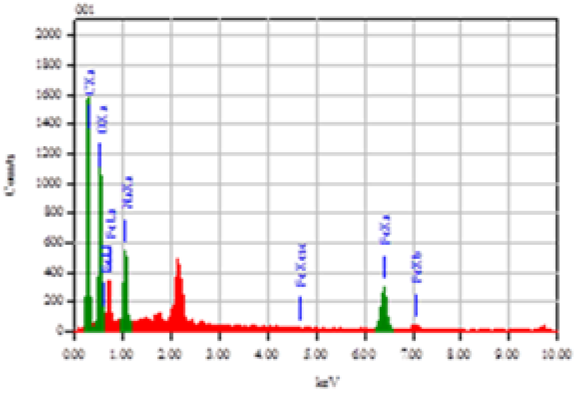


b

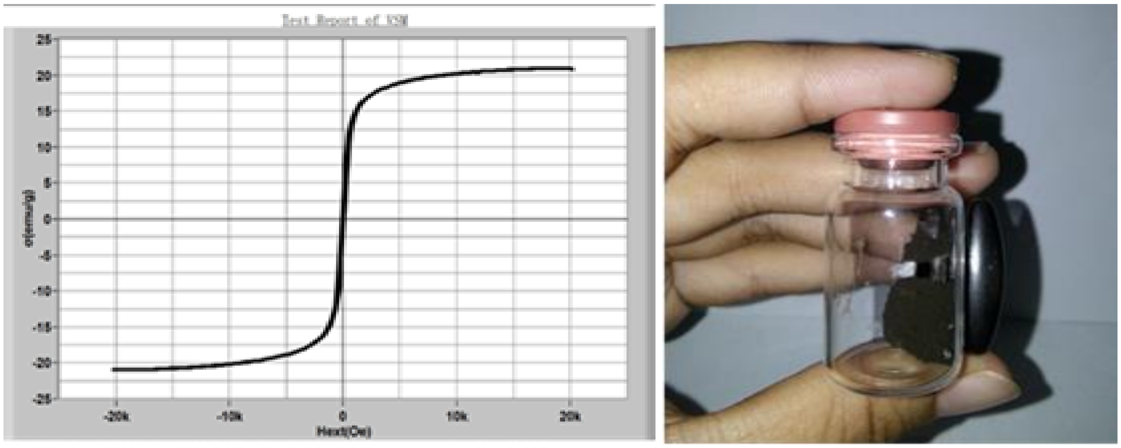
Figure 3. Elemental analysis of MAC before adsorption (a) and after adsorption of Cr(VI) (b).

The composition of active carbon elements can be seen in Table 1. The element carbon contained in activated carbon as 77.04 %. Whereas MAC Fe_3O_4 contains element C of 48.25%, element Fe of 16/37% and element O of 30.7%. This shows that there is a magnetite iron oxide compound (Fe_3O_4) deposit in activated carbon.

Table 1 : Composition of activated carbon (AC) and magnetite activated carbon (MAC) before adsorption Cr(VI).

	AC		MAC-Fe_3O_4	
Element	Mass (%)	Atom (%)	Massa (%)	Atom (%)
C	77.04	82.71	48.25	62.45
O	20.56	16.57	30.70	29.83
Si	0.43	0.20	-	-
Ca	0.77	0.25	-	-
Fe	1.20	0.28	16.37	4.56
Na			4.68	3.16

Characterization of the magnetite properties can be seen in **Figure 4a** and activated carbon which is attracted by an external magnet can be seen in F**igure 4b.** From the results of the VSM curve it can be seen that the VSM magnetization curve of the composite at room temperature. MAC composites exhibit paramagnetic behavior that is characterized by strong magnetic susceptibility. The MAC saturation magnetization value is 48.43 emu/g which is more than enough for conventional magnetic separation when compared with the magnetization value of 4.4 emu/g reported elsewhere. Similarly spherical magnetite nanoparticles were have been reported [21].



a b

Figure 4. VSM curve of magnetite activated carbon (a) dan magbetite activated carbon (b)

2. Sorption Study.

Effect of pH Cr(VI).

The effect of initial pH on adsorption of Cr(VI) onto MAC is carried out by varying the pH from 2 to 6 and shown in Figure 5. pH is one of the important parameters in the adsorption process. The initial pH of Cr(VI) solution was studied using a mass of 0.1 g MAC adsorbent and the volume of 20 mg/L Cr(VI) solution was 20 mL. The pH of the Cr(VI) solution affect the ionization state of the functional group on the MAC surface. Cr(VI) in the aqueous phase exists in different anionic forms such as chromate (CrO_4^{2-}), dichromate ($Cr_2O_7^{2-}$), or hydrogen chromate ($HCrO_4^-$) [x]. The dominant form of Cr(VI) at low pH is $HCrO_4$ [21]. The shift of $HCrO_4^-$ to other forms CrO_4^{2-} and $Cr_2O_7^{2-}$ increases with pH (ref). From these results it can be seen that Cr(VI) can adsorption Cr(VI) in several forms such as $HCrO_4^-$ at pH 3. Some functional groups of activated carbon such as phenolic (-OH) bind electrostatically negatively charged complex metal ions.

The decrease in adsorption increases with an increase in pH this is due to a decrease in electrostatic attraction between the adsorbent and the ion. At low pH, Cr(VI) becomes protonated so that the adsorption capacity becomes large; thus, the electrostatic tensile strength becomes high, resulting in an increase in Cr(VI) anionic adsorption capacity. The decrease in adsorption capacity above pH 3 occurs because the surface active site of the adsorbent becomes saturated by anionic such as CrO_4^{2-} and $Cr_2O_7^{2-}$ thus slowing the adsorption of ions towards adsorbent surface. An increased pH will caused the surface functional group to become negative and Cr ions form $Cr_2O_7^{2-}$ which is also negatively charged, resulting in a repulsive force between the functional group and Cr ions and caused the adsorption capacity Cr decreased [23]. The same thing was reported by Zhang et al, 2011, the pH for Cr(VI) adsorption using carbon silica composite adsorbent was obtained at pH 2-3 [9]. Mutongo et al (2020) obtained the pH for Cr adsorption using magnetite nanoparticles at pH 2-4 [23].

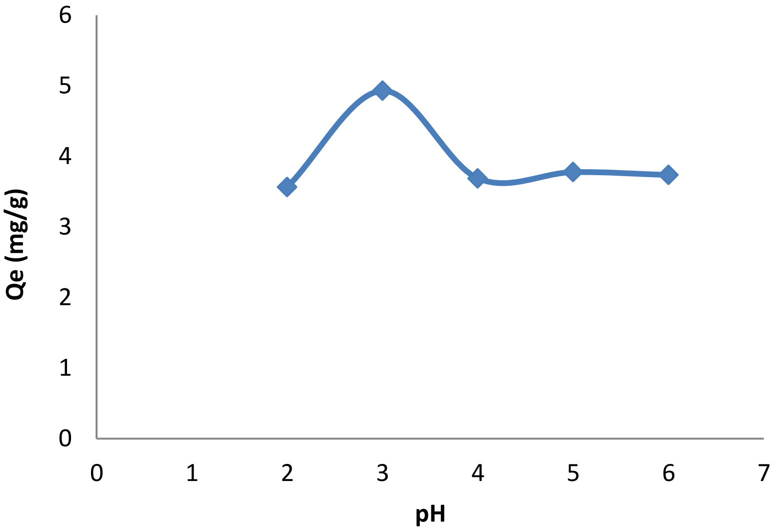


Figure 5. Effect of pH for adsorption of Cr(VI) on MAC.

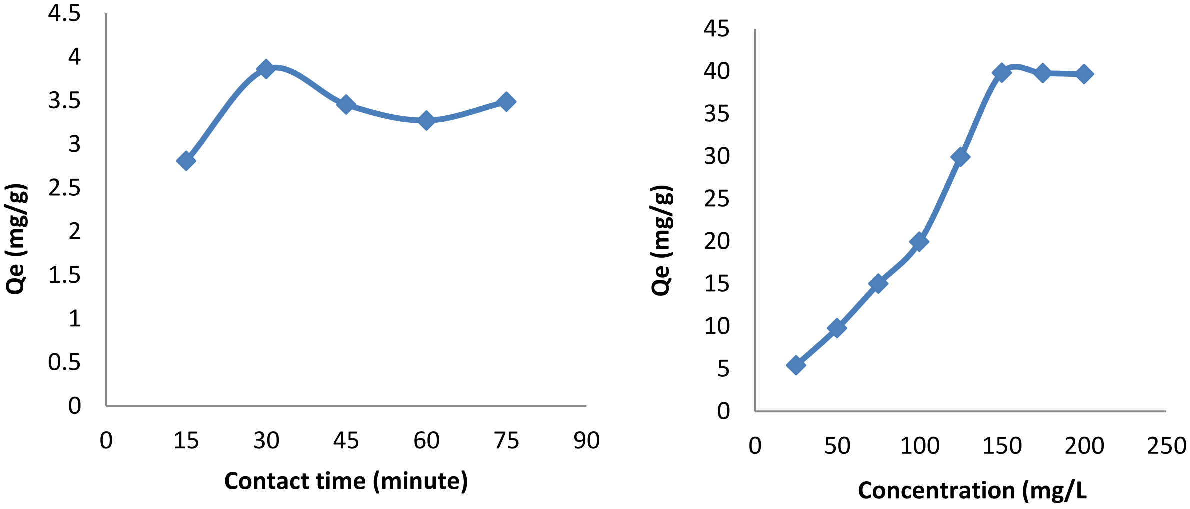
Effect of Contact time and initial concentration.

Contact time is one of the factors that affect the adsorption capacity. Contact time is related to the rate of reaction, which the concentration of a species changes with time. The determination of the contact time is used to obtain the optimum stirring time during the batch process so that the adsorbent can adsorption metal ions to maximum extent. The adsorption of an adsorbate will increase the longer the contact time. The long of contact time allows more diffusion and attachment of adsorbate molecules to take place.

The effect of contact time on adsorption of Cr(VI) on MAC can be shows at Figure 6a. Adsorption of Cr(VI) on MAC adsorbent with mass MAC 0.1 g over a period of 15–75 minute using 25 mg/L Cr(VI) 20 mL at pH 3. The adsorption capacity of Cr(VI) increases with increasing time from 15-30 minutes and reaches a state of equilibrium after 30 minutes with a maximum adsorption capacity of 3.859 mg/g. The rapid increase in adsorption capacity at the initial stage shows that there are many active sites on the MAC surface that are easily accessed, causing many Cr(VI) ions to be adsorbed on the MAC surface, and at 45-75 minutes reach equilibrium because the active sites of the functional groups on the MAC have filled by Cr (VI) ions.

The longer the contact time, the more interactions that occur between the adsorbent and the metal ion solution, so that more Cr metal ions are adsorbed on the MAC adsorbent surface. The adsorption of Cr(VI) ions is fast at the beginning of the adsorption time due to the availability of a large number of active sites on the MAC adsorbent surface [13].

At low initial concentrations of Cr(VI) the available surface ratio is larger, so the absorption becomes independent of the initial concentration. However, in the case of higher concentrations, this ratio is low. The percentage of removal depends on the initial concentration. The curve also shows that adsorption causes saturation, suggesting possible coverage of the Cr(VI) monolayer at adsorbent surface [13]. The effect of initial concentration Cr(VI) to adsorption capacity on MAC can be show in Figure 6b. Adsorption of Cr(VI) at various initial concentrations (25-200 mg/L) in 30 minutes using 0.1 g adsorbent MAC at pH = 3. The adsorption capacity increased with increasing concentration in the range of 25-200 mg/L, this shows that the number of active site on the surface of the adsorbent of that can interact with Cr(VI) ion. The amount of chromium removed from the solution increased with an increase in the concentration of Cr(VI). This may be due to the higher interaction between metal ions and metal adsorption sites of the adsorbent. The adsorption capacity increases with the difference in concentration between the liquid phase and the solid phase, and the driving force and mass transfer rate increase and accelerate [8]. The MAC adsorption rate is a function of the initial metal concentration. This phenomenon can be assumed that the high initial concentration of Cr(VI) ion provides the driving force needed for Cr(VI) to overcome the mass transfer resistance between the aqueous phase and activated carbon, moreover, competition between Cr(VI) ion.



a b

Figure 6. Effect of contact time (a) and initial concentration (b) of adsorption Cr(VI) on MAC.

4. Conclusion

Magnetite Fe_3O_4-activated carbon (MAC) were prepared from palm shell as precursor carbon and $FeCl_3$ and $FeSO_4$ as a magnetite agent form with co-precipitation method with ratio 1 : 2. MAC has been for adsorption of Cr(VI) from aqueous media. The MAC was characterized with XRD, SEM and VSM. The MAC used for adsorption of Cr(VI) with parameter adsorption for pH at 2-6, contact time 45 minute and initial concentration Cr(VI) range 10-200 mg/L with adsorption capacity 45.43 mg/g. MAC is one of the adsorbents that has the potential to be used for the removal of Cr ions in solution media

5. Acknowledgement

This study was supported by Hibah Penelitian Unggulan Fakultas Sains dan Teknologi Universitas Jambi.

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