



# ADSORPTION CHARACTERISTIC OF IRON ONTO POLY[EUGENOL-CO-(DIVINYL BENZENE)] FROM AQUEOUS SOLUTION

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**Abstract.** A study on the adsorption characteristic of iron onto Poly[eugenol-co-(divinyl benzene)] (EDVB) from aqueous solution has been conducted. EDVB was produced and characterized by using FTIR spectroscopy. The adsorption was studied by a batch method by considering the factors affecting the adsorption such as initial metal ion concentration, adsorption selectivity, and mechanism of adsorption using a sequential desorption method. The adsorption of iron onto EDVB followed a pseudo-2 order kinetics model with the rate constant of  $0,144 \text{ L}^2.\text{mmol}^{-1}.\text{min}^{-1}$ . The adsorption isotherm was studied with Tempkin, Langmuir and Freundlich models. The adsorption capacity ( $Q_{\text{max}}$ ) obtained by Langmuir isotherms was  $250 \text{ mg.L}^{-1}$  while the equilibrium value was  $0,8 \text{ Lmg}^{-1}$ . A competitive adsorption study showed that EDVB is adsorbed selectively towards iron rather than chromium, coppers and cadmium ions. The interaction type of iron onto EDVB was determined by a sequential desorption.

**Keywords:** Polyeugenol; divinyl benzene (DVB); adsorption; iron; Fe

## I INTRODUCTION

The contamination of heavy metals in water as one of industrial wastes containing some toxic properties in environment is deemed as a serious problem; hence, it must be removed from environment [1,2,3]. The one of important impacts of heavy metal contamination in environment is related to its accumulation to the soil after a long time of period. The presence of heavy metals in the soil is in view of the adsorption processes, chemical reactions and ion exchange in the soil [1]. Heavy metals can be toxic and non-biodegradable pollutants, which bring some impacts on environment, public health and economic [2]. Water with high iron content considered as heavy metal can be very objectionable either in taste, odor or in appearance [4,5]. Fe metal is the one of essential heavy metals to some extent is needed by organism, but in excess amount it has some toxic properties. The exceeding amount of Iron in common groundwater usually causes the water to appear yellow and it also becomes one

of the problems complained by the consumers for drinking water. The necessary of Iron for normal function in the human body is in the range of 3000-5000 mg. Fe as a secondary contaminant with the recommended limit of 0.3 mg/L according to the U.S. EPA., The excess of Iron can cause hepatoma, which is the primary cancer of liver that becomes the most common cause of death among patients with hemochromatosis, and can damage to the gastrointestinal track and liver [4,6]. The recovery of heavy metals can be done by various methods such as chemical precipitation, ion exchange, electrochemistry removal, membrane separation, and adsorption. Adsorption is one of the most effective and simple methods for treatment of hazardous toxic waste from solution and an economically viable alternative if a low-cost adsorbent is utilized [7,8,9]. The study on Fe metal ion adsorption has also been performed using various natural materials, such as natural zeolite and modified zeolite [10], chitin, natural clay and activated clay [6], unmodified raphia

palm [11], chitosan/ attapulgite composite [12], and maize husk [5]. In addition, there are still many other natural materials. Clove oil is one of the essential oils that have a high economic value and are produced in relatively large quantities. These oils can be obtained by extraction and distillation from the flower buds, flower stalks, leaves and leaf stalks of plants clove (*Eugenia caryophyllata*). The main components of clove oil are eugenol and kariofilena with the eugenol content of about 80% (v/v). Eugenol has several functional groups (allyl group, methoxy, and hydroxy) so it is possible to be modified into other compounds. The synthesis of polymer-eugenol and the some application in metal ion adsorption and membrane transport has been done by Ref. [13,14,15,16]. Polymer-eugenol which is modified with the DVB (divinyl benzene) as the cross linker has been done by Ref. [17,18,19], thus far its application as Iron adsorbent has not been studied. The research of polyeugenol as the selective adsorbent to Fe(III) has been done by Ref. [15]. EDVB as the modified eugenol polymer which has an additional active group  $-OH$  and  $-OCH_3$  attached to the benzene allows it to bind more cations, so it would be expected that EDVB may be used as an adsorbent of metal cations, especially Iron.

## II MATERIALS AND METHODS

All reagents were analytical grade without any further purification. Divinylbenzene (DVB), dibenzyl ether (DBE),  $BF_3O(C_2H_5)_2$ , anhydrous  $Na_2SO_4$ , NaOH,  $HNO_3$ , KCl, HCl,  $Na_2EDTA$ ,  $FeCl_3 \cdot 6H_2O$ ,  $CrCl_3 \cdot 6H_2O$ , and  $CuCl_2 \cdot 2H_2O$ ,  $CdCl_2 \cdot H_2O$  were purchased by Merck. Eugenol was supplied by Indesso Aroma, Purwokerto, Indonesia. All the organic chemicals were used as received. Double distilled water meanwhile was obtained from Chemistry Laboratories, UGM. Universal pH indicator. Instrumentations used as follow: Infrared spectroscopy (FTIR, Shimadzu Prestige-21), atomic absorption spectrophotometer (AAS, Perkin Elmer 3000), scanning electron microscope (JSM-6360LA), pH meter and analytical balance (Mettler Toledo AB54-S). The equipment was used such as glassware, plastic equipment (mouthpiece and PE bottle), magnetic stirrer, and shaker.

### The Synthesis of EDVB

The synthesis of EDVB in this study employed the basic ingredients of eugenol and catalyst  $BF_3O(C_2H_5)_2$  as conducted by Ref. [19]. EDVB was obtained by mixing eugenol (5.8 g) and DVB at the weight of 6 % relative to weight of

eugenol (0.348 g) in the flask. Then, 1 mL of  $BF_3O(C_2H_5)_2$  was added as the catalysts. Polymerization was carried overnight and terminated by adding 1mL of methanol. Red gel was obtained and dissolved in diethyl ether and then washed using the deionized water until reaching the neutral pH. The anhydrous  $Na_2SO_4$  was added into an organic layer. The solvent was evaporated and the residue was dried in a desiccators. Polymers formed were weighed and the yields were calculated. The products were characterized by Infrared Spectrometer.

### Adsorption experiment

Adsorption experiment was performed in a batch method by shaking 20 mg of EDVB with 20 mL of initial concentration of iron solution on shaker at a desired temperature. The effect of pH on the adsorption was investigated by the adsorption of iron on EDVB at pH variation. Iron solution was adjusted to pH 1.0; 2.0; 3.0; 4.0; and 4.5 by  $HNO_3$  and NaOH. The effect of interaction time and adsorption kinetic was investigated by the adsorption of iron onto EDVB at variation of times at the optimum pH. The variation of interaction time was carried out within 30, 120, 180, 360, 720, 1080, and 1440 min. The effect of initial concentration and isotherm adsorption was investigated by the adsorption of iron onto EDVB at the optimum pH and time with variation of initial metal solution concentrations. The variation of initial metal concentration was conducted by adjusting the concentration of metal solution to 25, 50, 100, 150, and 200 mg.  $L^{-1}$ .

### Selectivity of metal ion adsorption

The selectivity of adsorption was studied by performing variation of mixed metal solutions, i.e., a mixture of metal ions Iron-Cr(III), Iron-Cu(II) and Iron-Cd(II). Each variation also has the variation of mole ratios, i.e. 1:0, 1:1, 1:2, 2:1. The mixed metal solution (20 ml) at pH 4 was interacted with EDVB (20 mg) for 24 hours, and filtered and the filtrate was analyzed by AAS to determine the metal content of Fe (III), Cr (III), Cu (II) and Cd (II).

### Determination of adsorption mechanism

The solution of iron (20 mL, 100 mg.  $L^{-1}$ ) was interacted with 0.1 g EDVB at the optimum pH and time. Supernatant solution and EDVB with the adsorbed iron were filtered, and then EDVB with the adsorbed iron was washed in distilled water and dried. Then, EDVB with the adsorbed iron were conducted with solvents i.e. distilled water, KCl, HCl,  $Na_2EDTA$  to determine the interaction of sequential desorption of Iron from EDVB.

### Trapping mechanism

EDVB with the adsorbed Fe was added by 20 mL distilled water, and shaken for 1 hour. The Iron metal concentration in the filtrate was analyzed by AAS.

### Ion exchange mechanism

After trapping mechanism with the distilled water, EDVB remaining adsorbent was dried and added with 20 mL of 0,1 M KCl. The mixture was shaken for 2 hours. The Iron concentration in the filtrate was analyzed using AAS.

### Hydrogen bonds formation mechanism

The remainder EDVB of the ion exchange mechanism which has been dried was added with 20 mL of 0,1 M HCl. The mixture was shaken for 36 hours. The Iron concentration in the filtrate was analyzed using AAS.

### Complex bonds formation mechanism

EDVB which has been dried from the previous step was added with 20 mL of 0.1 M Na<sub>2</sub>EDTA. The mixture was shaken for 36 hours. The Iron concentration in the filtrate was analyzed using AAS.

## III RESULTS AND DISCUSSION

The polymer of EDVB has been successfully synthesized using BF<sub>3</sub> catalyst through a cationic polymerization. As shown in Fig. 1 the FTIR spectra of eugenol and EDVB, characteristic band for eugenol is because the absorption of allyl group (C-Csp<sup>2</sup> stretching) provides the band at 1636,5 cm<sup>-1</sup>, and presents signal at 650-900 cm<sup>-1</sup> which are the signal of benzene aromatic rings, vinyl group at 995,27 cm<sup>-1</sup> (-CH=CH<sub>2</sub> bending), O-H stretching at 3448,72 cm<sup>-1</sup>, C-H stretching of alkyl methyl (-CH<sub>3</sub>) and methylene group (-CH<sub>2</sub>) were showing the signal respectively at 1365,60 cm<sup>-1</sup> and 1435,04 cm<sup>-1</sup>, C-H bending of 1,2,4-substituted aromatic group can be observed at 817,82 cm<sup>-1</sup>, then -C-O-C- stretching present at signal 1300-1000 cm<sup>-1</sup>. In Fig. 1 the polymerization of eugenol and DVB can be characterized by the losses of allyl group at 1636,5 cm<sup>-1</sup> and vinyl group at 995,27 cm<sup>-1</sup>. Chemical structure of divinylbenzene, eugenol and EDVB can be seen in Fig. 2.

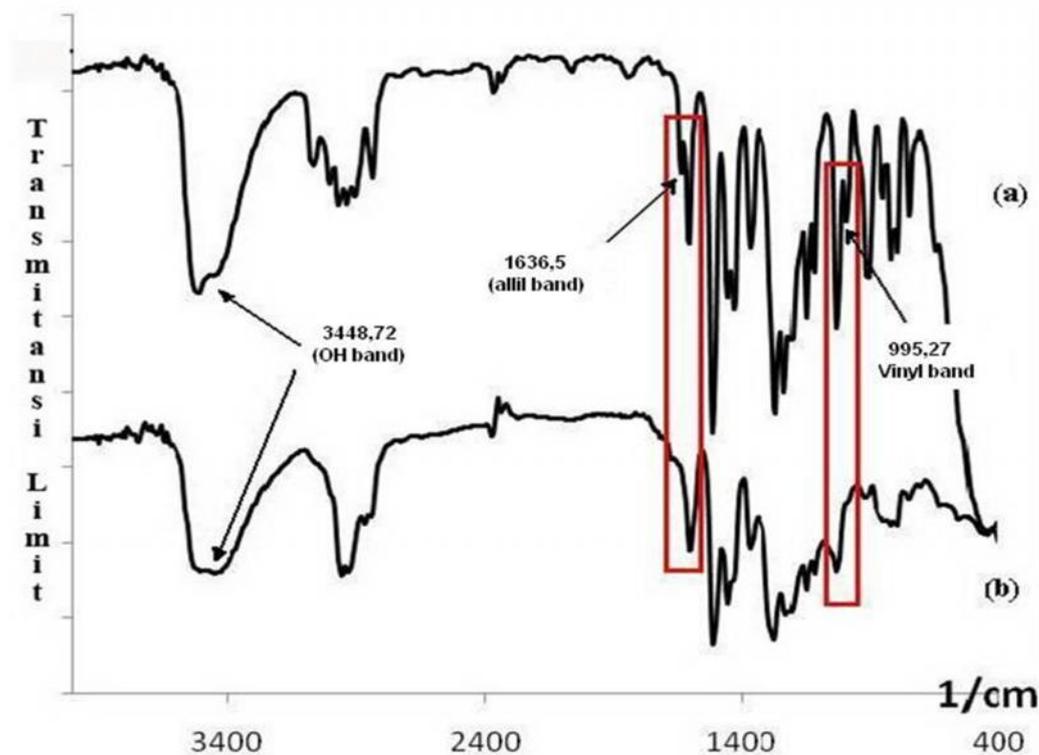


Fig. 1 IR Spectra of (a) eugenol; (b) EDVB

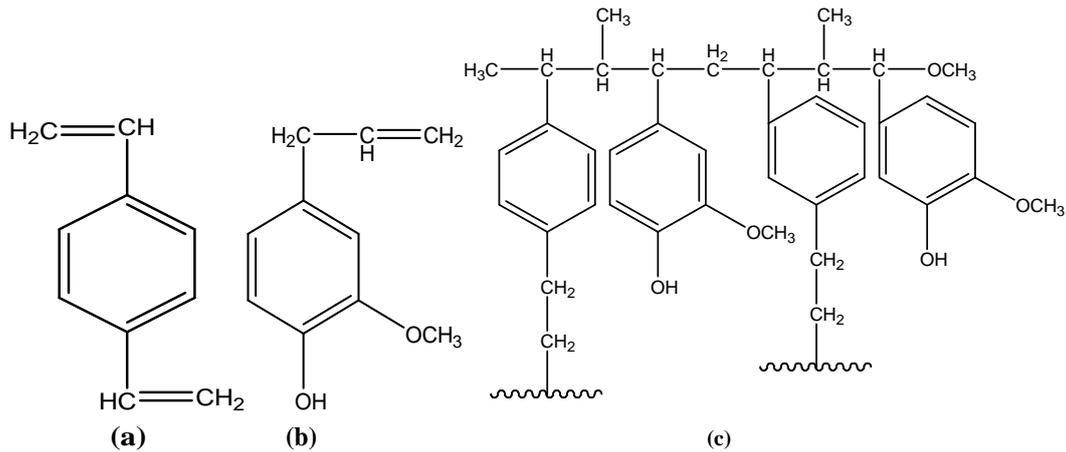


Fig. 2 Chemical structures of (a) divinylbenzene; (b) eugenol; (c) EDVB

pH is an important parameter in the adsorption process. It can cause the changes of charge on the adsorbent surface and metal ion speciation in solution. The selected pH was in the range below 5 because the speciation of metal ions Iron at pH 5 was formed as the precipitate of  $\text{Fe}(\text{OH})_3$  and before the deposition of Fe ions as  $[\text{Fe}(\text{OH})_4]^{5+}$  in a very little [20]. In this study, the optimum pH adsorption was obtained at pH 4.5. It was possible because the speciation ion  $\text{Fe}^{3+}$  was protonated to  $[\text{Fe}(\text{OH})]^{2+}$ ,  $[\text{Fe}(\text{OH})_2]^+$ , and  $[\text{Fe}_2(\text{OH})_2]^{4+}$ . Hence, it was possible if the surface of the adsorbent, which had a cluster of the active O at O-H, interacted with the complex iron ions in the form of hydrogen bonds. In addition, it was also possible if the adsorbent surface speciation was derived from phenolic compounds into phenolic ions in alkaline pH solution conditions [21]. Thus, it indicates that at pH 4.5 phenolic ions can interact optimally with the positive charged metal ions.

The determination of optimum interaction time iron onto EDVB was conducted by interacting EDVB with iron solution at various times (30, 120, 180, 360, 720, 1080, and 1440 minutes). The adsorption was done at optimum pH. Adsorption kinetics were determined by comparing the value of  $R^2$  of several kinetics models, such as using first order, second order, pseudo first-order, pseudo second order and Langmuir-Hinshelwood kinetics models. The first-order and second-order were determined respectively using the Eq. (1) and (2) [22].

$$\ln C_t = -k_1 t + \ln C_0 \quad (1)$$

$$\frac{1}{C_t} = k_2 t + \frac{1}{C_0} \quad (2)$$

where  $C_t$  is the concentration of residual Iron at  $t$  ( $\text{mmol. L}^{-1}$ ),  $C_0$  is the initial concentration of

iron ( $\text{mmol. L}^{-1}$ ),  $k_1$  is the first-order reaction rate constant,  $k_2$  is the second-order reaction rate constant Pseudo first-order reaction kinetics and pseudo second order were determined respectively using Lagergren in Eq. (3) and (4) [23,24].

$$q_e - q_t = \log q_e - \left(\frac{k_3}{2.303}\right)t \quad (3)$$

$$\frac{t}{q_t} = \frac{1}{k_4 q_e^2} + \left(\frac{1}{q_e}\right)t \quad (4)$$

$q_e$  is the initial concentration of iron,  $q_t$  is the concentration of iron after equilibrium at time  $t$ ,  $k_3$  is the pseudo first-order reaction rate constant,  $k_4$  is the pseudo second-order reaction rate constant. Langmuir-Hinshelwood kinetics model was used to determine the equilibrium constant using the Eq. (5) [25]

$$\left[\ln\left(\frac{C_0}{C_a}\right) / (C_0 - C_a)\right] + K = k_1 t / (C_0 - C_a) \quad (5)$$

where  $C_0$  is the initial concentration of iron,  $C_a$  is the residual concentration of iron,  $K$  is the equilibrium constant, and  $k_1$  is reaction rate constants. Based on the linearity data in Table 1, the adsorption of Iron onto EDVB followed the pattern of the kinetics pseudo second-order. It means that the adsorption reaction of iron onto EDVB was influenced by three reactants, where the one reactant had the excessive concentration; thus, it can be regarded as a catalyst. The calculation of the adsorption kinetic using the Langmuir-Hinshelwood model obtained reaction rate constants ( $k$ ) of  $7,446 \times 10^{-6} \text{ min}^{-1}$  and the equilibrium constant ( $K$ ) of  $8,513 \text{ mol. L}^{-1}$ .

To determine the adsorption isotherm of iron onto EDVB, the initial iron concentration was varied in various concentrations, i.e., 25, 50,

100, 150, 200 mg. L<sup>-1</sup> at the optimum pH (4.5) and time interaction (6 hours). The effect of concentration on the adsorption of iron onto the EDVB is shown in Fig. 3.

Table 1 Kinetics parameters of iron adsorption onto EDVB  
Kinetics parameter of adsorption of Iron on EDVB

Kinetic models	reaction rate constant (k)	Linearity (R <sup>2</sup> )	Equilibrium constant (K) (mol. L <sup>-1</sup> )
Order 1	1,0 × 10 <sup>-2</sup> (L. min <sup>-1</sup> )	0,951	-
Order 2	0,153 (L <sup>2</sup> . mmol <sup>-1</sup> . min <sup>-1</sup> )	0,837	-
Pseudo order 1	6 × 10 <sup>-3</sup> (L. min <sup>-1</sup> )	0,874	-
Pseudo order 2	0,144 (L <sup>2</sup> . mmol <sup>-1</sup> . min <sup>-1</sup> )	0,999	-
Langmuir-Hinshelwood	7,446 × 10 <sup>-6</sup> (min <sup>-1</sup> )	0,912	8,513

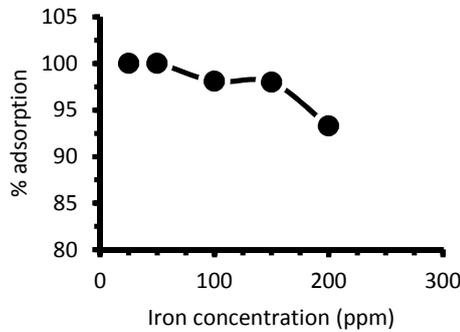


Fig. 3 Effect of concentration to Iron adsorption onto EDVB

Adsorption isotherms were used to determine the form of interaction between the solutions with the adsorbent and to determine the adsorption capacity of adsorbent. In this study Langmuir, Freundlich, and Tempkin isotherm model were used to fit the experiment data, as respectively expressed by Eq. (6), (7), and (8) [26].

$$\frac{C_e}{q} = \frac{1}{K q_{max}} + \frac{1}{q_{max}} C_e \quad (6)$$

$$\log q = \log K_F + \frac{1}{n} \log C_e \quad (7)$$

$$q_e = B_1 \ln K_t + B_1 \ln C_e \quad (8)$$

where  $C_e$  is the equilibrium concentration (mg.L<sup>-1</sup>),  $q$  is the amount adsorbed at equilibrium (mg.g<sup>-1</sup>),  $q_{max}$  is the capacity when high concentration (mg.g<sup>-1</sup>),  $K$  is the Langmuir constant (L.mg<sup>-1</sup>),  $K_F$  is the Freundlich capacity factor (mg.L<sup>-1</sup>/n.L/n.g<sup>-1</sup>),  $\frac{1}{n}$

is the Freundlich intensity parameter,  $B_1 = \frac{RT}{b}$ , where  $T$  is the absolute temperature (K),  $R$  is the ideal gas constant (8,314 kJ/kmol.K),  $K_t$  is the equilibrium constant (L.mg<sup>-1</sup>), adsorption variation energy (kJ.mol<sup>-1</sup>). Figure 4 presents three adsorption isotherms models with a good linearity, but the linearity ( $R^2$ ) is shown at best in the Tempkin isotherm model considering the effect of interaction between the adsorbate with the adsorbate, the heat of adsorption of molecules on the surface layer decreasing linearly due to the interaction between the adsorbate and another [27]. Table 2 presents the parameters of each adsorption isotherm model.

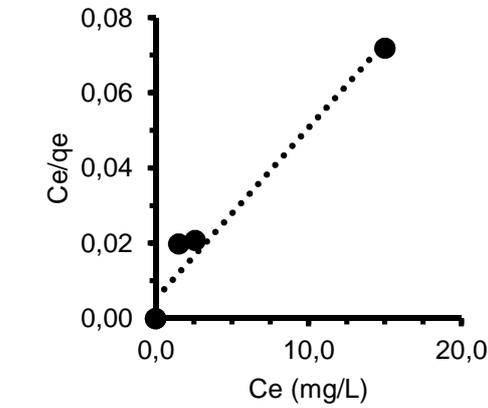
Table 2 Adsorption isotherm parameters

Isotherm Model	Parameters	R <sup>2</sup>
Langmuir	$K$	0,8 (L. mg <sup>-1</sup> )
	$q_{max}$	250 (mg. g <sup>-1</sup> )
Freundlich	$K_f$	1,0093 (mg. g <sup>-1</sup> )
	$1/n$	0,055
Tempkin	$K_t$	3,01912 (L. mg <sup>-1</sup> )
	$B$	55,35 (kJ. mol <sup>-1</sup> )

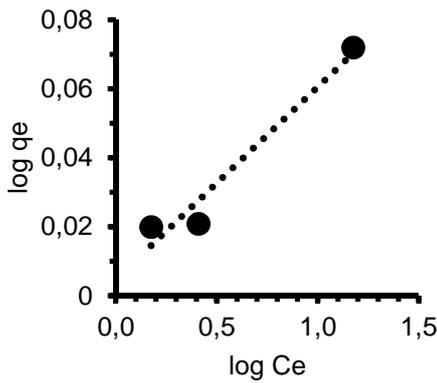
Langmuir model explains about adsorption, occurred in monolayer. The chemical bonding occurred between the adsorbate and the adsorbent is quite strong, and then when the adsorbent surface is covered by the adsorbate, adsorbent will not adsorb another adsorbate, although the temperature and concentration increase. Langmuir isotherms obtain the adsorption capacity ( $Q_{max}$ ) 250 mg.g<sup>-1</sup>, its value is better compared to chitin, chitosan, clay, raphia palm fruit endocarp and chitosan/antapulgite composite which respectively have 1,4537; 90,09; 30,0; 16,3500; 62,50 mg.g<sup>-1</sup> [6,7,11,12]. Then the equilibrium obtains value 0.8 L. mg<sup>-1</sup> (Fig. 4).

Selectivity experiment was conducted to determine whether EDVB has adsorption selectivity for iron or as a universal adsorbent of metal ions. EDVB was interacted with the mixture of metal, i.e., Iron-Cr(III), Iron-Cu(II) and Iron-Cd(II) solutions at several mol ratios i.e. 1:0, 1:1, 1:2, and 2:1 at the optimum pH and time interaction. The results of the adsorption selectivity of metal mixtures onto EDVB are shown in Fig. 5. The figure presents about the selectivity of other metals with an insignificant influence on the adsorption of iron onto EDVB, and only Chromium ions (mol ratio 1:1), which showed a considerable

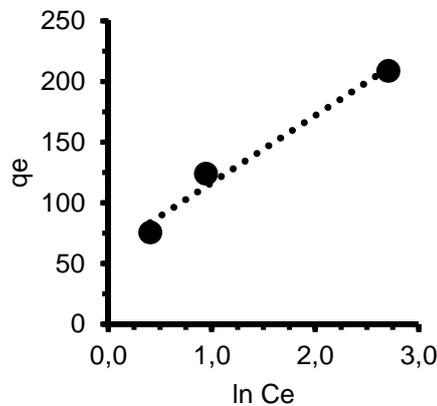
adsorption competition. This is possible due to Chromium has the physical and chemical properties of metal which is almost similar to Iron as hard acid metal according to Hard Soft Acid Base rules. EDVB as a Hard Base adsorbent are suitable for Hard Acid metal i.e. iron and chromium.



(a)



(b)



(c)

Fig. 4 Adsorption isotherm models of Iron adsorption onto EDVB: (a) Langmuir; (b) Freundlich; (c) Tempkin

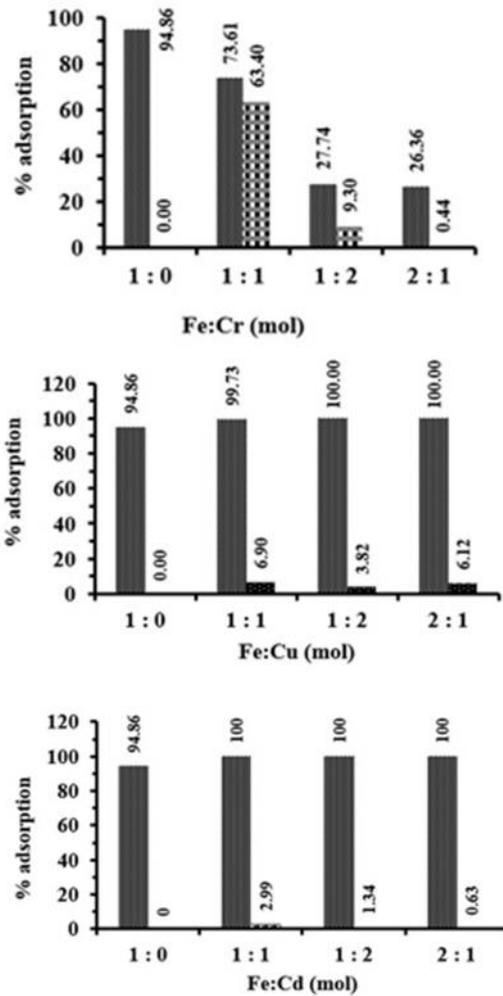


Fig. 5 Selectivity of metal ion adsorption onto EDVB

The determination of adsorption mechanism was performed with a sequential desorption method using several different solvents. It was performed to identify the type of bonding occurred between Iron ions onto EDVB. A sequential desorption was done with H<sub>2</sub>O (distilled water), KCl 0,1 M, HCl 0,1 M and 0,1 M Na<sub>2</sub>EDTA as the solvent. Desorption by H<sub>2</sub>O proved about a bonding mechanism of trapping, KCl proved about a mechanism of ion exchange, HCl proved about the existence of hydrogen bond formation and Na<sub>2</sub>EDTA proved about the bonding mechanism of complex formation. The results of sequential desorption are presented in Fig. 6. Based on desorption data, it was found that HCl had the greatest percentage at 31,32%, followed by Na<sub>2</sub>EDTA at 1,84%. These results indicated that the bonding occurred between the iron onto EDVB was hydrogen bonding. It was possible because Iron ions were interacted at pH 4.5 in the form of [Fe(OH)]<sup>2+</sup>, [Fe(OH)<sub>2</sub>]<sup>+</sup>, and [Fe<sub>2</sub>(OH)<sub>2</sub>]<sup>4+</sup>, so the hydrogen of -OH

ligand could bind the hydrogen ions with the oxygen atom in the active group of EDVB [20]. Approximately 1,84% of iron was also desorbed by Na<sub>2</sub>EDTA. It was possible because the most iron bound to the EDVB through the formation of chelate complexes. An interaction between iron onto EDVB is shown in the IR spectra of Fig. 7. As shown in the figure, some changes of peak before and after adsorption in the areas of -OH group absorption peaks shift toward lower band were from 3448,72 cm<sup>-1</sup> to 3433,29 cm<sup>-1</sup>, accompanied by an increase in

peak intensity and widening of the band. The strong peaks changed from 15,88 to 13,104 while the width of the shift changed from 312,45 to 402,458. In the areas of -OCH<sub>3</sub> (1033,85 cm<sup>-1</sup>) had not shifted the peak absorption, but there was a strong change in the peak of 16,17 to 14,931 and widening the band occurred from 77,06 to 93,439. The absence of peak shift indicates that the active -OCH<sub>3</sub> group interaction occurs only in the form of stretching rather than bending.

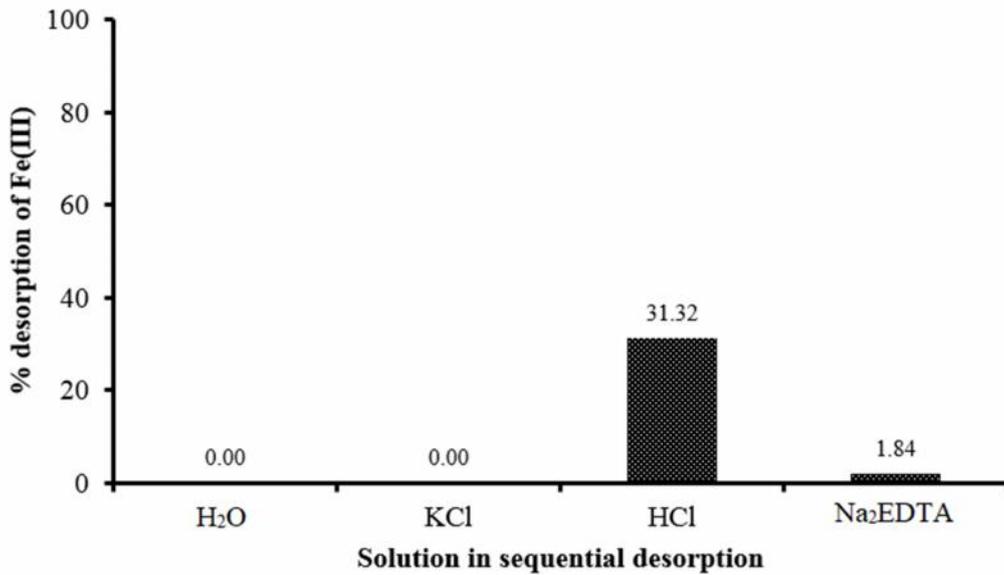


Fig. 6 The sequential desorption of iron in various solvents

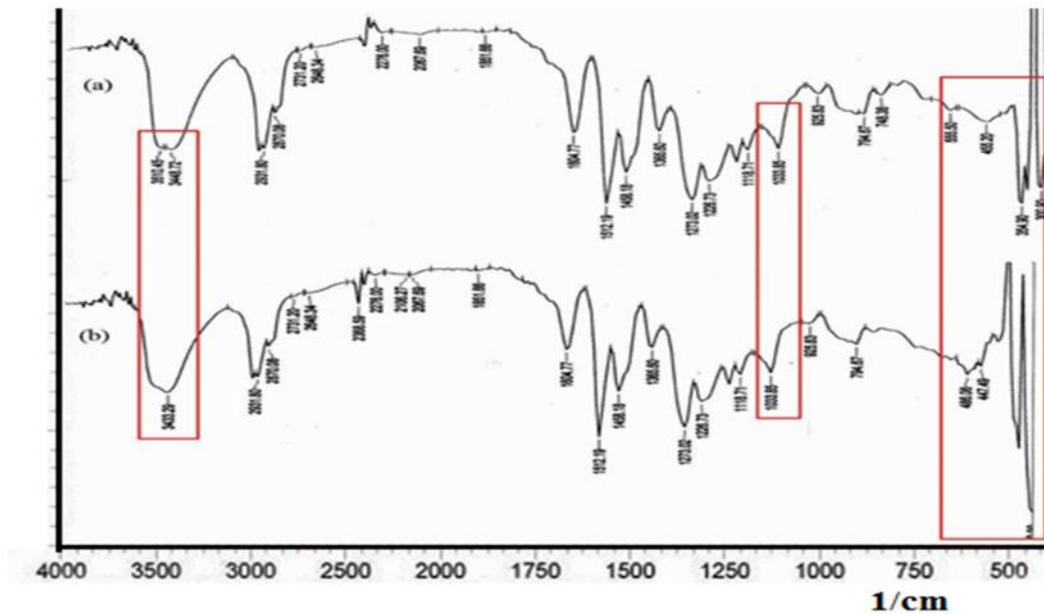


Fig. 7 FT-IR spectra (a) EDVB before adsorption and (b) EDVB after adsorption

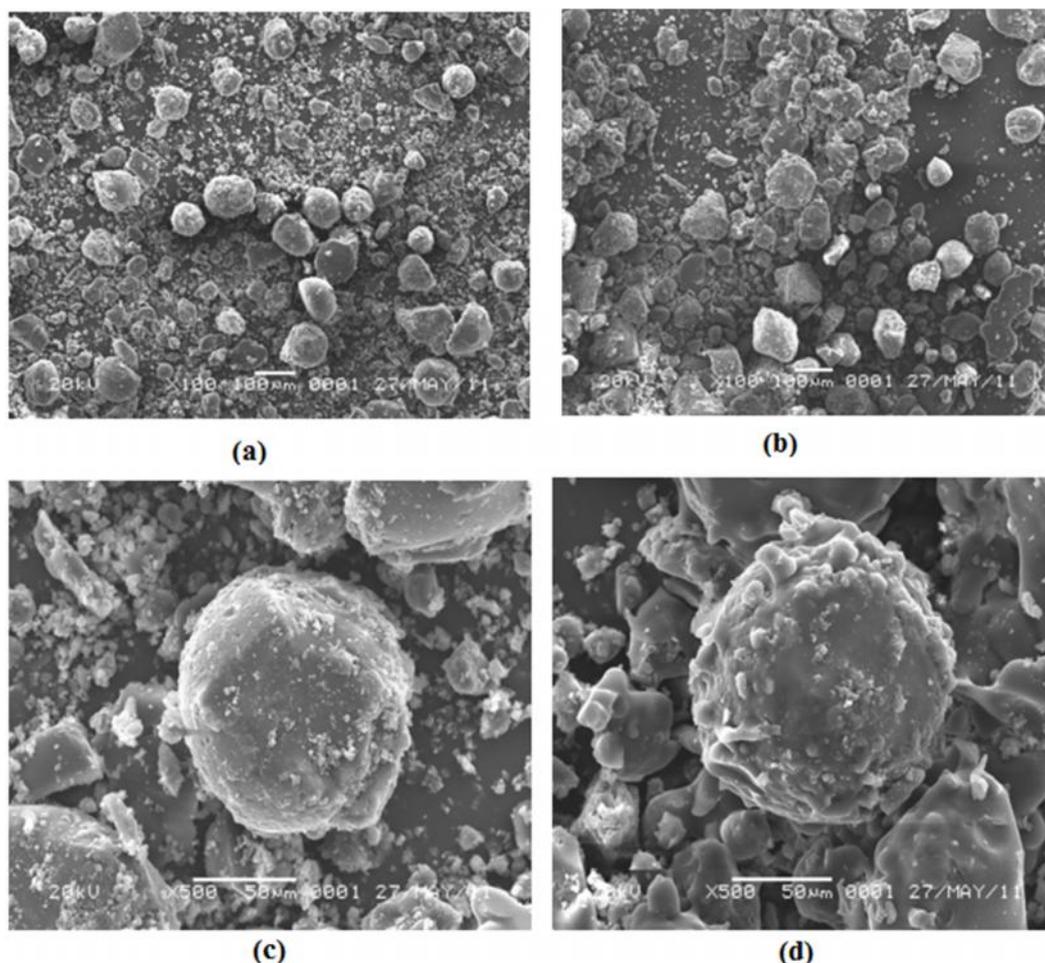


Fig. 8 SEM of surface EDVB (a) before interaction, magnification 100 $\times$ ; (b) after interaction, magnification 100 $\times$ ; (c) before interaction, magnification 500 $\times$ ; and (d) after interaction, magnification 500 $\times$

Based on the comparison of IR spectra, it can be concluded that the adsorbent-adsorbate interaction is possible to occur in the active -OH groups. It is in accordance to the reactivity of -OH group is stronger than  $-OCH_3$  group. Ref. [28,29] found that the interaction of metal with carboxymethyl-chitosan adsorbent whose an active group are O and N showed the change in IR absorption band below  $1000\text{ cm}^{-1}$ . In this study, IR spectra also indicated a change in the form of absorption below  $1000\text{ cm}^{-1}$  around  $750\text{-}400\text{ cm}^{-1}$ . Therefore, it can be concluded about the interaction between the adsorbent EDVB with Iron ions occurs.

The morphological features and surface characteristics of EDVB before and after the adsorption experiment were obtained from the scanning electron microscopy using JSM-6360LA at the accelerating voltage of 20 kV (Fig. 8). It was taken at two different magnification, at 100 $\times$  magnification the granules of EDVB before and after adsorption looks the same, yet at 500 $\times$  magnification the surface of EDVB granules is clearer then

shows the different surface between EDVB before and after adsorption. The figure shows that the surface area of EDVB before interaction with iron looked smooth while after interaction it appeared bumpier with some protruding parts. These pictures verify that after interaction the surface of EDVB has some protruding parts that expected as the adsorbed metal ion of iron.

## CONCLUSIONS

In this study, the potential of EDVB has been assessed for the adsorption of iron. EDVB can be used as an adsorbent to adsorb iron through the formation of hydrogen bonds and chelate formation. The adsorption kinetics model experiments fitted with pseudo second-order that showed a correlation coefficient  $R^2$  0,999 with the value of reaction rate constant  $0,144\text{ min}^{-1}$ . The maximum adsorption capacity had the high value at  $250\text{ mg}\cdot\text{g}^{-1}$ , it value is better compared to some other Iron adsorbent. EDVB also had a selectivity adsorption to iron compared to the chromium, coppers, and

cadmium. Based on the result, it can be conclude that EDVB, a modified eugenol polymer adsorbent, is a potential alternative material for iron adsorption from aqueous solution or water.

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