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ORIGINAL ARTICLE

Adsorption of Phenol and Iron in Wastewater Using a Mixture of Active Carbon and Zeolite Modified with Chitosan

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ABSTRACT – Phenol (C_6H_5OH) and iron (Fe^{2+}) contained in electroplating industrial wastewater are toxic even at small concentrations. One of the effective methods to remove these compounds is adsorption because the method is easy and simple. Activated carbon and zeolite are types of adsorbents that are often used in wastewater treatment. In this study, modification of zeolite (Z) and activated carbon (AC) with chitosan (Ch) were carried out to adsorb waste containing phenol and Fe^{2+}. Modified AC-Ch and Z-Ch composites were characterized by FTIR and BET. The variations used were pH (7 and 9) and the adsorbent composition to study its effect on the adsorption of phenol in the presence of Fe^{2+}. Adsorption was carried out in a bed with circulating waste for 2 hours. Feed at pH 9 with adsorbent mixed of zeolite-chitosan composite (Z-Ch) and activated carbon-chitosan composite (AC-Ch) in a ratio of 1:1 gave the optimal results. Under these conditions, 94% and 97% of phenol and Fe^{2+} were adsorbed, respectively.

INTRODUCTION

Nowadays, contamination of water sources is increasing due to industrial activities which are caused by the accumulation of various toxic pollutants. Phenol (C_6H_5OH), a prominent pollutant in wastewater, is an organic compound that can be decomposed in the air, but it takes a longer time to be decomposed in water [1]. Various industries that generate phenol include petroleum refineries, pharmaceutical industries, paper and pulp, petrochemicals, and pesticides [2]. It is very crucial to treat phenol since phenol-contaminated water became toxic to aquatic life, human, and living organism. The presence of phenol in water (even at low concentrations) can cause protein degeneration and damage to organs such as the liver, pancreas, and kidneys [3]. Therefore, the allowable concentration of phenol before discharge to the environment should be less than 0.1 mg/L [2], and the maximum phenol content in wastewater is 0.5 mg/L according to US Environmental Protection Agency (EPA)[3]. Meanwhile, wastewater contaminated with heavy metals such as iron (Fe^{2+}) is also dangerous to human life due to its toxicity. Iron is needed for the body's functions, but will be toxic if the amount is excessive in the body. Some industries which generate metal pollution are mining industries, metallurgy, and metal plating. For drinking water, the Fe concentration should below 0.3 mg/L, according to The World Health Organization (WHO) [3]. Furthermore, phenol is often found alongside heavy metals such as Fe^{2+} or Cr^{6+}, which makes wastewater treatment even more complicated [2]. One example of an industry whose waste contains phenol and heavy metals such as Fe^{2+} and Cr^{6+} is electroplating. Phenol comes from the additives used, while Fe^{2+} or Cr^{6+} are wastes from the electroplating process.

One of the effective methods to treat wastewater containing toxic substances such as phenol and heavy metal (Fe^{2+} or Cr^{6+}) is the adsorption process due to the simple operation, high efficiency, low cost, and many available adsorbents [3]. Some of the adsorbents that have been proven to absorb phenol and heavy metals are activated carbon [4], natural and synthetic zeolite [5], and chitosan [6]. Activated carbon (AC) is a carbon material that is widely used as an adsorbent for filter media and purification processes in various industries [7]. Unfortunately, although activated carbon presents a good adsorption capacity for various pollutants, this adsorbent has drawbacks namely difficulty in regeneration, high disposal costs, and low adsorption capacity of phenol [8]. Meanwhile, zeolite, aluminosilicate (consisting of a three-dimensional network of tetrahedrons SiO_4 and AlO_4^-), is also widely used as adsorbents for purification and catalysts. This adsorbent has unique properties including high absorption capacity/surface area, ion exchange and adsorption abilities, stability and can be regenerated [9,3]. If the Si/Al ratio in the zeolite is high, the adsorption selectivity for phenolic compounds is better, but if the zeolite contains a lot of pure silica, the adsorption capacity will be higher than CAC (Coal Activated Carbon) [10]. Moreover, chitosan (poly(d-glucosamine)) which contains amine, hydroxyl, and acetamide functional groups, is a traditional sorbent that can also be produced by shells of marine animals such as shrimp. It is suitable for the removal of pollutants (phenol, metal ions) from wastewaters via chelation involving the amino and hydroxyl groups in the glucosamine unit [3].

Activated carbon and zeolite are universal adsorbents because they are easy to produce and inexpensive. These adsorbents have high adsorption efficiency, but they are not adsorbents that can selectively separate substances such as

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phenol and iron in wastewater. To increase the selectivity and adsorption capacity, it needs to be modified (to form a composite) with the main adsorbent, which has high selectivity, especially phenol, but it does not reduce its adsorption capacity. Therefore, due to the excellent properties of chitosan, modification of activated carbon (AC) and zeolite (Z) by chitosan (Ch), a biodegradable polymer, is a new challenging alternative to treat wastewater containing organic pollutants such as phenol and heavy metals such Fe^{2+} and Cr^{6+}. Furthermore, the addition of fillers such as activated carbon or zeolite will increase the mechanical properties and chemical stability of chitosan [11]. However, there are actually many factors that affect the efficiency of the adsorption process, including the number and concentration of pollutants and the surface area of adsorbent. To the best of our knowledge, a little study was performed to treat complex pollutant containing organic waste and heavy metals (phenol, Fe^{2+} and Cr^{6+}) using composite of Z-Ch, AC-Ch, and the mixture of Z/AC-Ch with circulating process. Therefore, we claim this process is our academic novelty.

In the present work, we investigate the effect of pH in the removal of complex pollutants using activated carbon and zeolite adsorbents which has been composited with chitosan. To prepare the composite of activated carbon and zeolite modified chitosan, it used the procedure that has been done by previous researchers with modifications. These modifications include chitosan concentrations and sizes of activated carbon/zeolite. In this study, we varied the pH, namely 7 and 9 since pH is a very crucial/influential factor in waste treatment, especially in the adsorption process. The modified composites were then characterized by FTIR and BET. The 3 compositions of the adsorbent in the bed were 150 grams of zeolite-chitosan composite (Z-Ch); 150 grams of activated carbon-chitosan composite (AC-Ch), and the mixture of 75 gr chitosan zeolite composite and 75 grams of chitosan activated carbon composite (Z/AC-Ch). The adsorption processes were performed with a recirculation system of 2 L waste in 2 hours. The initial concentrations of the pollutants were 4.96 ppm of phenol and 8.15 ppm of Fe^{2+}. The initial and final concentrations were measured using a spectrophotometer.

EXPERIMENTAL METHOD
Materials and Instruments

The raw materials, namely granular activated carbon (30 mesh) and zeolite (granular, 6-8 mesh), were washed with demineralization water to clean the dirt and dust attached to the surface of those adsorbents. Then, these materials were dried using heat for several minutes to remove their moisture content. All chemicals namely acetic acid (CH_3COOH), sodium hydroxide (Na(OH)), hydrogen chloride (HCl), phenol (C_6H_5OH), iron(II) chloride ($FeCl_2$), sodium dichromate ($Na_2Cr_2O_7$) used in this study were purchased from Sigma Aldrich.

Figure 1 presents the series of equipment for conducting experiments in this study. The set of equipment consists of an adsorption column with 18 cm long and a diameter of 5.08 cm, a control valve with 0.25 inch, a pH meter, a submersible pump, a basin with a capacity of 5 L, and a pipe and fittings with a diameter of 0.25 inch.

1) Adsoprtion column
2) Control valve
3) pH meter
4) Submersible pump
5) Basin
6) Pipe and fittings
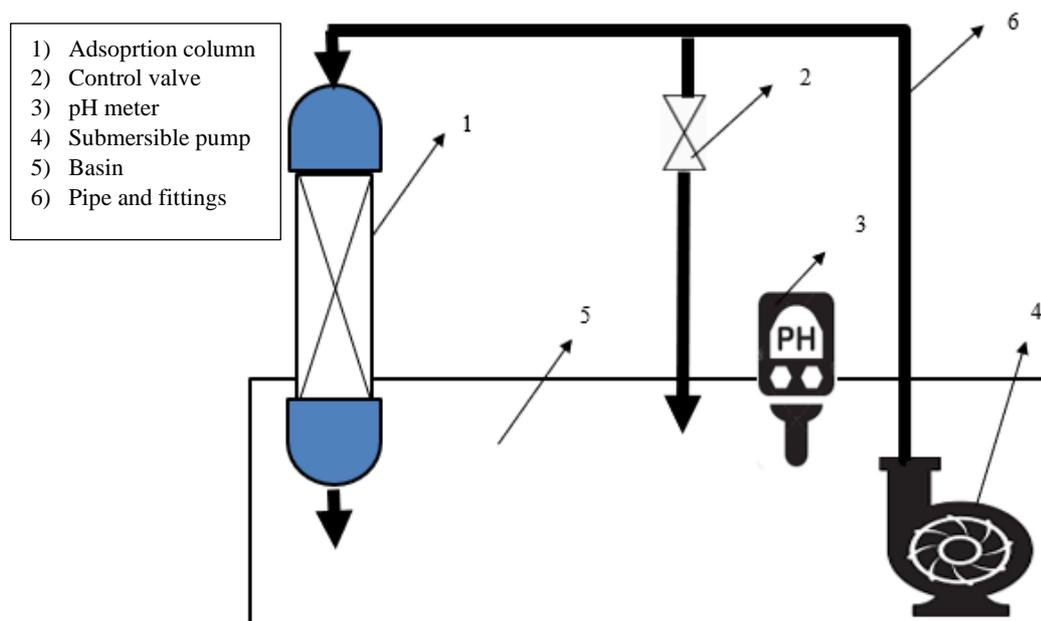
Figure 1. The equipments for conducting adsorsion process

Phenol concentration before and after the adsorption process were measured according to standard procedure of SNI 06-6989.21-2004, (Water and wastewater – Part 21 test method for phenol content spectrophotometrically) [12] with absorbance obtained from a spectrophotometer at the wavelength of 500 nm. Meanwhile, to measure the concentration of Fe, a standard procedure of SNI 6989.4-2009, (Water and wastewater – Part 4 test method for iron (Fe) content by

atomic absorption spectrophotometry) [13] was used at the wavelength of 248,3 nm. The type of the spectrophotometer was Thermofisher Aquamate 8000 Uv-Vis.

Method and Procedure

The zeolite-chitosan composite (Z-Ch) was made based on the research of Fatombi et. al. [14] with slight modifications. As much as 150 grams of zeolite was added to a chitosan-acetic acid solution with a composition of 50 grams of chitosan and 500 ml of acetic acid solution (10v/v). The mixture was stirred continuously using a magnetic stirrer for 2 hours. Then 500 ml of 10% acetic acid solution was added and stirred for 1 hour. The solid particles formed in the suspension were then separated and put into a precipitation bath in which there were 500 ml of NaOH (0.5 M), stirred for 3 hours, and then the solid particles were taken and cleaned using demineralization water until a pH of about 7. Subsequently, this non-crosslink zeolite-chitosan composite was dried in the air for 6 hours. To study the functional groups present in the Z-Ch composite, Fourier transform infrared (FTIR) analysis (Nicolet iS5 Thermo Scientific) was performed over a wavenumber range of 400-4000 cm^{-1}. Moreover, to get information about the specific surface area and pore volume of the composite, BET characterization was performed by adsorption of Nitrogen (Quantachrome Nova, version 11.03).

A composite of activated carbon-chitosan (AC-Ch) was made by mixing 150 grams of activated carbon with 75 grams of chitosan which had been dissolved in 500 ml of acetic acid solution (10v/v). The mixture formed was stirred continuously using a magnetic stirrer for 2 hours. Then 500 ml of 10% acetic acid solution was added and stirred for 1 hour. The solid particles formed were then separated and put into a precipitation bath in which there were 500 ml of NaOH (0.5 M), stirred for 3 hours. Solid particles in suspension were taken and cleaned to a pH of about 7 using demineralization water and finally dried in an oven at 50°C for 6 hours. then a crosslinked activated carbon-chitosan composite is formed. Similar to Z-Ch, the FTIR and BET characterization were also conducted for the AC-Ch composite.

The adsorption process was carried out by pumping 2 L of a sample solution containing phenol (4. 96 ppm) and Fe (8.15 ppm) using a submersible pump at a rate of 0.05 L/sec to the adsorption bed for 120 minutes of circulation. The final concentration of the solution was taken at the sample point to analyze the concentration of phenol and Fe after adsorption. This process was performed at variations in the pH of the solution (7 and 9) by adding 0.25 M HCl or 0.25 M NaOH. The variation of the bed used 3 types, namely the bed filled with 100% zeolite-chitosan composite, 100% activated carbon-chitosan composite, and zeolite-chitosan composite 50% + activated carbon-chitosan composite 50%.

The reduction or elimination of phenol and iron (II) concentration due to the adsorption process was measured after 2 hours process using a UV-Vis spectrophotometer and calculated using the following Eq. (1):

$$\%\text{adsorption} = \frac{C_{initial} - C_{final}}{C_{initial}} \times 100\% \tag{1}$$

where $C_{initial}$ and C_{final} are the initial and final concentrations (after adsorption) of phenol and Fe^{2+}, respectively.

RESULTS AND DISCUSSION

Figure 2 presents the FTIR spectra of AC-Ch and Z-Ch composites. According to the FTIR result for AC-Ch (Figure 2 (a)), a band located at 3674 cm^{-1} indicates the O-H stretching vibration due to intermolecular hydrogen bonding [14]. The peaks at 2970 cm^{-1} and 1670 cm^{-1} represent the C-H vibration of -CH_3 and the stretching vibration of the carbonyl group C=O from amide I of chitosan respectively. The absorption peak at 1527 cm^{-1} attributed to N-H stretching vibration in amide II. Whereas, the peak at 1053 cm^{-1} corresponds to the stretching of C-O or O-H deformation in the carboxylic acids present in AC [14]. These findings confirm that Ch was successfully coated on AC.

FTIR of the Z-Ch spectra is depicted in Figure 2 (b). The wide peak at 3398 cm^{-1} represents the O-H groups in zeolite and chitosan. The peak at 2969 cm^{-1} corresponds to the C-H stretching vibration of O-CH-O in the spectrum of chitosan [15]. The peak at 1639 cm^{-1} attributed to the N-H group in the primary amine, while a band located at 1547 cm^{-1} indicated the NH_3^+ group in the composite. The zeolite major adsorption bands were: 3397, 1639, 1022, and 783 cm^{-1} [15]. The stretching vibration of the Si-O group creates the band at 1022 cm^{-1}, while the peak observed at 783 cm^{-1} corresponds to quarts or amorphous stretching vibration of SiO_2. The peaks at 586 cm^{-1} characterized the stretching vibration Si-C stretching vibration that indicates the presence of bonding between Z-Ch in the synthesized composite. Analysis of the Z-Ch composite spectra in Figure 2 (b) demonstrated Z-Ch composite was produced.

Figure 3 shows AC and Z with sizes of 30 mesh and 6-8 mesh respectively, which have been composited with Ch. Visually, the surface area of AC-Ch is larger than Z-Ch. BET analysis results indicated that AC-Ch has larger surface area of 502.5 m^2/g and Z-Ch has lower surface area of 22.18 m^2/g.

Table 1 shows the concentration of phenol and Fe after the adsorption process with phenol and Fe concentrations before adsorption/initial were 4.96 and 8.15 ppm respectively. Equation 1 is used to calculate the % phenol and Fe^{2+} adsorption.
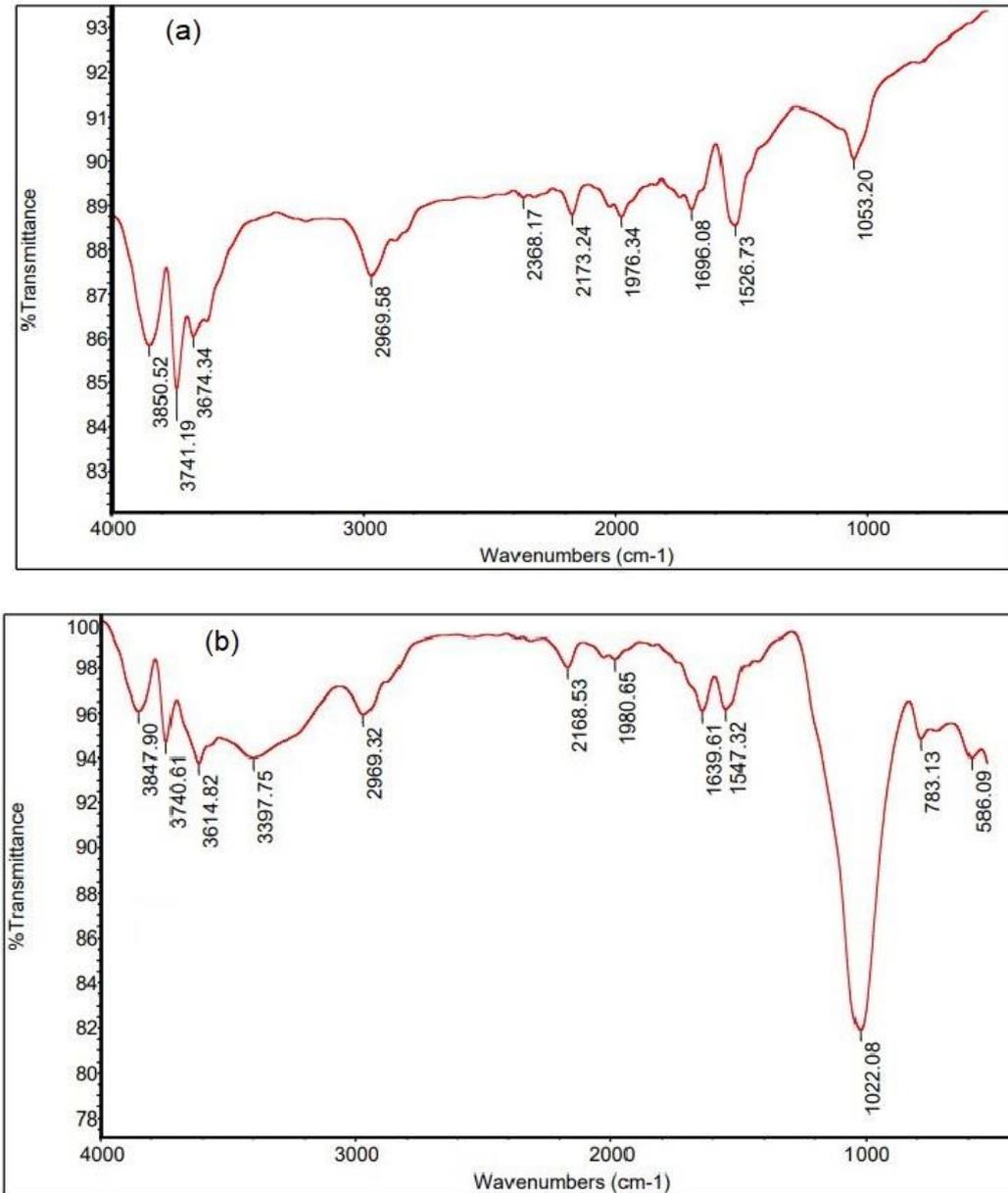
Figure 2. FTIR spectra of (a) AC-Ch (a) and (b) Z-Ch composite
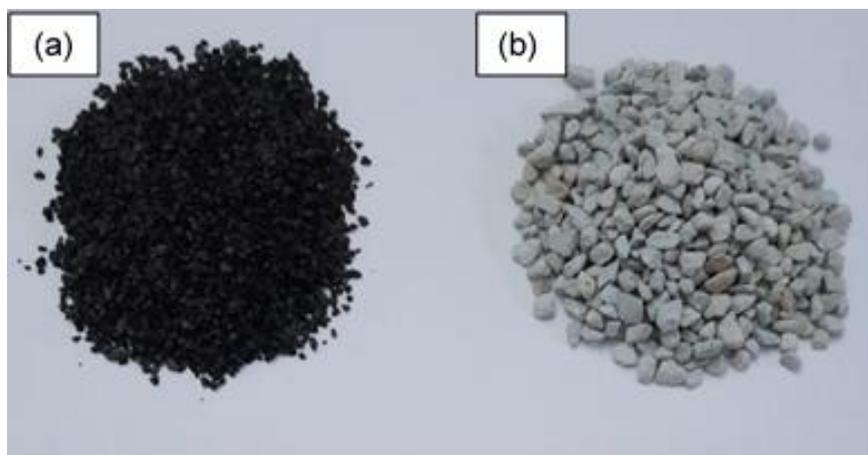
Figure 3. The image of (a) AC-Ch (a) and (b) Z-Ch composites

**Table 1.** The pH and phenol/Fe concentration before and after adsorption at various adsorbents type

Adsorbent type	Initial pH	Phenol concentration after adsorption	Fe concentration after adsorption	pH after adsorption	% phenol adsorption	% Fe adsorption
Zeolite-chitosan	7.0	4.86	5.31	6.0	2.14	34.88
Carbon active-chitosan	7.0	2.55	4.47	5.6	48.60	45.12
Zeolite-Chitosan/Carbon active-chitosan	7.0	0.78	0.26	6.0	84.22	96.82
Zeolite-chitosan	9.0	4.77	5.89	8.0	3.83	27.70
Carbon active-chitosan	9.0	0.75	2.86	7.65	84.97	64.95
Zeolite-Chitosan/Carbon active-chitosan	9.0	0.283	0.245	7.0	94.30	96.99
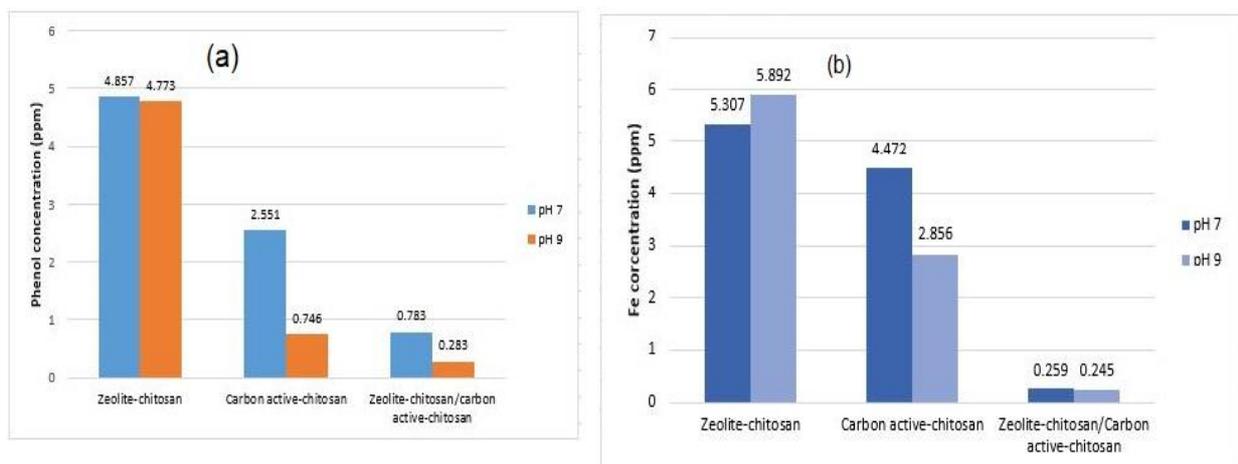
Figure 4. The effect of pH on the final phenol concentration (a) and final Fe concentration (b) at various adsorbent types

Figures 4 and 5 present the effect of pH in the phenol and Fe^{2+} adsorption at various adsorbent types. Based on Figures 4 (a) and 5 (a), pH 9 present % phenol adsorption better than pH 7 for 3 types of adsorbent system. In nature, activated carbon and chitosan have a good adsorption capacity at high pH compared to low pH which tends to be acidic. In general, the adsorption of phenol using chitosan at an acidic pH will reduce while at a pH above 7.8 the adsorption rate will be maximized [16]. Meanwhile, phenol adsorption using zeolite-chitosan composite has a low adsorption capacity. This is because phenol which can react with sodium hydroxide used to raise the pH of the waste forms sodium phenoxide whereas the nature of zeolite which is a positive ion exchanger cannot exchange negatively charged phenoxide ions. Furthermore, the zeolite used is in the form of a large granular so that it has a small surface area which also has an impact on the contact of the waste with the adsorbent.

From Figures 4 (b) dan 5 (b), for the zeolite-chitosan system, pH 7 depicted better % Fe adsorption compared to pH 9. It was reported that in acidic solutions protonation of amine groups of chitosan happens and it reduces the number of binding sites for Fe adsorption, but increasing the pH until 7, the adsorption increases. However, at pH values higher than 7, the Fe ions start to precipitate [17], therefore at pH 9 % Fe adsorption decreases. For the active carbon-chitosan system, at a lower pH (7), the Fe^{2+} ion will compete with H^+ (proton) during the process so that the ability of the adsorbent to absorb Fe^{2+} is reduced. Consequently, the % of the adsorbed substance becomes lesser. On the contrary, at a higher pH (9), the number of protons decreased, thereby increasing the adsorbent's ability to adsorb Fe^{2+}. The same result was

reported by Wulan et al. [18]. Therefore, the adsorption process using a mixture of zeolite-chitosan and activated carbon-chitosan composite will give the same % adsorption at pH 7 and 9.
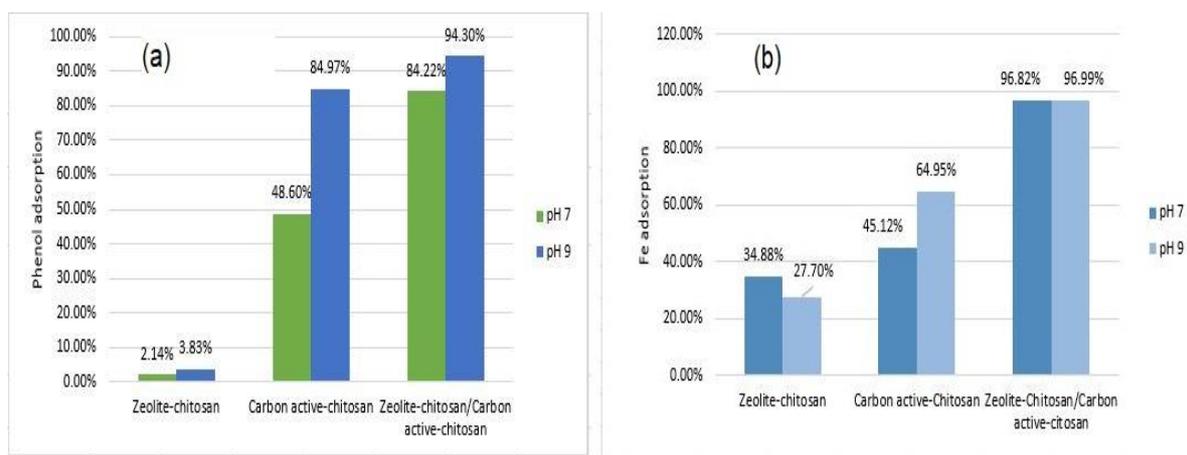
Figure 5. The effect of pH on (a) the % phenol adsorption and (b) the % Fe adsorption at various adsorbent types

From those figures, it can also be concluded that the composite mixture of zeolite-chitosan and activated carbon-chitosan gave the best % adsorption of phenol and Fe. This is because zeolite functions as a pH stabilizer. Due to the zeolite-chitosan (Z-Ch) configuration being placed at the upper of the bed, then when the waste was recirculated, the pH is stable at the optimal pH. In addition, zeolite has positive ion exchange properties [19], therefore Fe ions can be absorbed first in the upper bed which contains zeolite-chitosan. As a result, the activated carbon-chitosan can more easily adsorb phenol and remaining Fe. The AC-Ch system is better % adsorption than Z-Ch since the surface area of the AC adsorbent where the interaction between liquid or gas molecules and solid molecules involved is larger. The more pores or the larger the surface area of the adsorbent, the greater the % adsorption. According to BET results, AC-Ch has a high adsorption capacity due to the higher surface area of 502.5 m^2/g with pore volume of 0.203 cm^3/g. Meanwhile, Z-Ch has lower surface area and pore volume of 22.18 m^2/g and 0.06 cm^3/g respectively. The use of chitosan provides a level of selectivity to phenol compounds due to hydrogen bonds, proton transfer, and London-van der Waals forces between phenol and chitosan due to a large number of the hydroxyl groups and amino groups in chitosan [20]. Activated carbon also has a high adsorption capacity to high dissolved iron in wastewater.

In this study, an adsorption process was also carried out with the addition of Cr^{6+} ions in the waste containing phenol and Fe^{2+}, operating conditions at pH 9 with a mixture of AC-Ch and Z-Ch adsorbents. Cr^{6+} concentrations were measured according to standard procedure SNI 06-6989.71-2009 (Water and wastewater – Part 71 test method for Cr^{6+} content spectrophotometrically) [21] with absorbance obtained at the wavelength of 540 nm. The results are shown in Table 2.

Table 2. The % adsorption of wastewater containing phenol, Fe^{2+} and Cr^{6+} at pH 9

Chemical	Initial Concentration	Final Concentration	% Adsorption
Phenol	4.96	0.97	80.56
Fe^{2+}	8.15	1.26	84.53
Cr^{6+}	0.75	0.36	51.60

Table 3 shows that the addition of chromium (VI) compounds has an impact on decreasing the adsorption of phenol and iron due to the more substances being adsorbed. However, the adsorption value was still above 80% for phenol and Fe^{2+} and 50% for Cr^{6+}. This value is still high even though the waste already contains phenol and iron. Activated carbon can adsorb Cr^{6+} compounds ranging from 27.2 to 62.08 % depending on the operating conditions [22].

CONCLUSION

The pH, type and composition of adsorbent, and the number of chemical substances (adsorbate) in the waste affect the % adsorption. The best condition in this study was achieved at pH 9 using a mixed adsorbent of zeolite-chitosan and activated carbon-chitosan with 93.3 and 96.99 % adsorption for phenol and Fe respectively. If the number and concentration of pollutants in the waste increase, but are treated with the same amount of adsorbent, then the adsorption capacity to each component will decrease because some of the adsorbents will adsorb the added pollutant. This condition occurs due to the competition between the adsorbates to be adsorbed by the adsorbent.

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