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Adsorption of Isopropyl Alcohol (IPA) in Water Using **Activated Bentonite**

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Abstract. It has been done separation isopropyl alcohol (IPA) in water using activated bentonite as adsorbent. Preparation of activated bentonite is done using H_2SO_4 1 M. Characterization of adsorbent includes structural analysis with XRD, surface area and pore size using the BET method. The adsorption test is carried out in a batch system with variations in stirring time, adsorbent mass, pH and temperature. Analysis of the amount of IPA adsorbed is determined by a picnometer. The results of the adsorption test showed that the adsorption capacity optimum of IPA by activated bentonite was 116.62 mg/g at optimum conditions, i.e. 40 minutes stirring time, 1 g of active bentonite mass, at 30 °C and acidity level 7. Model of isotherm adsorption of IPA in activated bentonite are isotherm Langmuir model with corelation coefficient (R²) of 0,9283. The thermodynamic parameters (ΔH° , ΔS° and ΔG°) are 189.37 J·mol⁻¹,-1.08 J·mol⁻¹·K⁻¹ and 132.63 J·mol⁻¹respectively.

Keyword: bentonite, isopropyl alcohol, separation

1. Introduction

Isopropyl alcohol (IPA) is a popular name for chemical compounds with the molecular formula C_3H_8O or C_3H_7OH . This compound is a colorless, flammable compound with a strong odor. This compound is the simplest secondary alcohol IPA is a tertiary alcohol that can be used as an antiseptic, cleaning, dispersant pigment and wet wipes. But the biggest use of IPA is as a solvent [1]. IPA is one of the hazardous and toxic water wastes (B3) produced by a pharmaceutical industry, hospital, car salon and others. IPA if it enters the body can cause poisoning, respiratory problems, affecting the digestive tract and nervous system. IPA can be easily oxidized in the liver to acetone [2].

IPA waste can be separated from water using air stripping and activated carbon fiber columns. This method is relatively easy, but requires pH control and cannot be carried out at low temperatures [3]. The process of removing high concentrations of IPA in waste is done by pervaporation using crosslink membrane IPN (interpenetrating network) such as PVAH (polyvinyl acrylic acid and hydroxyethyl methacrylate) and modification of PVOH (polyvinyl alcohol) [4]. Pervaporation process requires low energy but the process is complex and not easy to operate [3]. In addition to these methods, Hsu et al., 2011 used carbon nanotubes (CNTs) as well as adsorbents in the process of removing IPA [5]. Other materials that have the potential to be used as IPA adsorbents replace carbon nanotubes are bentonite. Pratiwi (2015) reported that bentonite can be used as an adsorbent in removing heavy metals contained in wastewater [6]. Hartanti et al., 2012 reported that bentonite-chitosan composites can be used to adsorb organic pesticides, namely diazinon [7]. Adsorption is considered a powerful technique and is widely used to eliminate IPA from domestic and industrial wastes [8].

2. Experimental Method

2.1. Materials

Natural bentonite was obtained from the Badan Geologi Bandung, IPA, H₃PO₄, H₂SO₄ and AgNO₃, CH₃COONa, NH₄Cl, NaH₂PO₄, Na₂HPO₄, NH₃, CH₃COOH, BaCl₂ and aquades.

2.2. Preparation and characterization of adsorbent

Natural Turen's bentonite was purified by pouring in water with ratio bentonite:water 1:4 (wt./wt.), stirring for 15 minutes, and letting the mixture stand a day. This process was repeated seven times. Finally, one third of solids at the top was taken and dried at 110 °C for 24 h [9]. A total of 5 g of purified

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natural bentonite was placed into a glass beaker containing 200 mL of 1.2 M H₂SO₄ and stirred with a magnetic stirrer for 24 h. The mixture was then centrifuged to separate the activated bentonite with a solution of H₂SO₄. The activated bentonite was then washed with distilled water until free of SO₄⁻ ions. Test of SO₄⁻ ions were performed by adding 2 drops of BaCl₂ 0.1 M into the former washing water until no more white precipitate formed. Characterization to determine the surface area and pore size of the catalyst was performed by N₂ adsorption-desorption method. The structural analysis were monitored using a XRD spectrophotometer with CuK α radiation ($\lambda = 1.5405$ Å).

2.3. Study of adsorption isotherms

The adsorption study was carried out at optimum conditions. Optimization of the adsorption time was carried out through an adsorption test on the a variation of stirring times of 40, 60, 80, and 100 minutes. For adsorption test a total of 1 g of actived bentonite was palced into Beaker glass containing 50 mL of 30% (3930 ppm) IPA and stirred with a magnetic stirrer at room temperature and neutral pH. The mixture was centrifuged to separate the actived bentonite with a solution of IPA which is not adsorbed. The concentrations of IPA was determined using picnometers. The adsorbed IPA (%) was determined by equation 1, with C_o and C_e were the concentration before and after the adsorption process. The optimum time is determined from the curve of adsorption (%) versus time.

Adsorption (%) =
$$\frac{C_o - C_e}{C_o} \times 100\%$$
 (1)

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For optimization of pH, it was prepared solution of buffer acetate to provide a solution of IPA at pH 5, solution of phosphate buffer to provide solution of IPA at pH 6, 7, 8 and solution of ammonia buffer to provide a solution IPA at pH 9. The adsorption test was carried out in the same procedure at room temperature and optimum stirring time. The optimum pH was determined from the curve of adsorption (%) versus pH. The optimization of temperature was carried out in the same procedure in the variation of temperature (30, 40, 50, and 60 °C), optimum stirring time and optimum pH. The optimization of the mass of adsorbent was carried out in the same procedure (1, 2, 3 and 4 g of activated bentonite), optimum pH, optimum temperature and optimum stirring time. The optimum of mass of adsorbent was determined from the curve of adsorbent.

To determination of the adsorption model, the adsorption capacity (q_e) was determined with the adsorption test at optimum conditions of the solution IPA in variation of concentrations (10% (1310 ppm), 20% (2620 ppm), 30% (3930 ppm), 40% (5240 ppm) and 50% (6550 ppm) The adsorption capacity is calculated according to equation 2, with m is the mass of the adsorbent.

$$q_e = \frac{C_o - C_e}{m} \tag{2}$$

The model of Langmuir or Freundlich isotherm adsorption was determined from the curve of adsorption capacity versus the concentration of the IPA which gives a high correlation coefficient (R^2).

As for thermodynamic studies, it was determined q_e with adsorption test at optimum conditions (time, pH and mass of adsorbent) in the variation of temperatures (303, 313, 323 and 323 K) to the IPA solution in variation of concentrations (10, 20, 30 and 40%). From the q_e and C_o values obtained, it was used to determine the equilibrium constant (*K*), i.e. the intercept of curve of Co versus (C_o/q_e). Then the thermodynamic units was determined from the curve of ln *K* vs 1/T according to equations 3 and 4.

$$\ln K = \frac{\Delta S^o}{R} - \frac{\Delta H^o}{RT}$$
(3)

$$\Delta G^{o} = \Delta H^{o} - T \Delta S^{o} \tag{4}$$

3. Results and Discussion

3.1 Characteristics of the adsorbent

The characteristic test with XRD on sample of natural bentonite was conducted to prove that the sample used contained the main mineral bentonite constituent, namely montmorillonite. Based on the analysis of the diffractogram, sample of natural bentonite has a peak with a relatively large intensity at the

diffraction angle of 20, i.e. 5.71, 17.20, 19,82, 20,89, 28,10, 31,98 and 34,95°. Based on the simulation results by X'pert Highscore program, the diffraction angle of 20 and the relative intensity of sample of natural bentonite has conformity with the PDF data of montmorillonite (PDF-00-003-0010, molecular formula $(Na,Ca)_{0.3}(A1,Mg)_2Si_4O_{10}(OH)_2\cdot xH_2O)$ and impurities, i.e. clinoptilolite (PDF-00-025-1349, molecular formulas $(Na,K,Ca)_6(Si,A1)_{36}O_{72}\cdot 20H_2O)$, quartz (PDF-01-085-0794, the molecular formula SiO₂) and calcined albit (PDF-00-020-0548, formula molecule $(Na,Ca)_6(Si,A1)_4O_8$), shown in Figure 1.



Figure 1. X'pert Highcore program simulation results from the diffractogram of sampel natural bentonite of Turen Malang

In addition to the structure, the surface area of the adsorbent has a very important role in the adsorption process, because on the surface of active bentonite there are siloxane groups that have the ability to interact with organic compounds so that molecules of organic compounds can be adsorbed. From the results of the analysis of surface area using the BET method, it was found that activated bentonite had a surface area of $379.24 \text{ m}^2/\text{g}$ and an average pore diameter of 126.50 Å. Based on the pore size, activated bentonite is a mesoporous material, because it has a pore diameter size of 20\AA to 500 Å. From the results of the analysis of surface area using the BET method, it was found that active bentonite had a surface area of $379.24 \text{ m}^2/\text{g}$ and an average pore diameter of 126.50 Å. Based on the pore size, activated bentonite is a mesoporous material, because it has a pore diameter size of 20\AA to 500 Å. From the results of the analysis of surface area using the BET method, it was found that active bentonite had a surface area of $379.24 \text{ m}^2/\text{g}$ and an average pore diameter of 126.50 Å. Based on the pore size, active bentonite is a mesoporous material, because it has a pore diameter size of 20\AA to 500 Å (Granite et al., 2015). This conclusion is also supported by the Langmuir isotherm adsorption curves (Figure 2), i.e. type IV, which is the type of adsorption-desorption that occurs in mesoporous material [10].



Figure 2. Curve of isoterm adsorption (-o-)-desorpsi (-o-) Langmuir of activated bentonite

3.2 IPA adsorption studies

The adsorption study of IPA solution in bentonite was carried out to describe the phenomenon of molecular mobility of IPA from the aquatic environment to the solid phase at optimum conditions. Optimization of the adsorption conditions of IPA solutions in bentonite adsorbents was carried out to obtain optimum adsorption capacity. From the optimization results show that the IPA adsorption process in active bentonite produces the optimum amount of IPA adsorbed at stirring time of 40 minutes, using 1 gram of active bentonite mass, at 30 °C and pH 7. The amount of IPA adsorbed after the optimum stirring time is almost the same (Figure 3a). This is because after 40 minutes the adsorption-desorption equilibrium has been reached (Kamel, 2013). Mass optimization shows that adsorption-desorption equilibrium is not affected by mass (Figure 3b). The increase in temperature causes a little increase in the amount of IPA adsorbed but then decreases (Figure 3c), indicating that the adsorption that occurs is physical adsorption (Madan, 2015). The influence of the acidity (pH) on the IPA adsorption process on the surface of the bentonite is very small (Figure 3d). In these conditions an optimum capacity of 116.62 mg/g is obtained.

Furthermore, based on the results of the optimum adsorption capacity on the variation of the concentration of the IPA solution, the isotem adsorption model of the IPA solution was determined in the activated bentonite, that is the adsorption of Freundlich isotherm and the Langmuir isotherm. The isotherm adsorption study was carried out to describe the adsorption that occurred, monolayer, multilayer, non-ideal, reversible, irreversible, homogeneous or heterogeneous. In the isotherm adsorption model of Freundlich (equation 4) and Langmuir (equation 5), x/m is the amount of substance adsorbed per mass adsorbent (in this study defined as adsorption capacity, q_e) and P is the pressure of the adsorbate molecule in the gas phase (in this study is the initial concentration of molecular adsorbate, C_o). Based on this, equations 4 and 5 turn out to be equations 6 and 7, so that the Freundlich isotherm adsorption curve is obtained (Figure 4a) and Langmuir (figure 4b). The curve of q_e/C_o versus C_o curve has a correlation coefficient (\mathbb{R}^2) greater than the curve of log q_e vs. log C_o . This shows that the adsorption of IPA in activated bentonite is more appropriate in the Langmuir isotherm model, which occurs in a monolayer, homogeneous and limited to certain active sites.



Figure 3 a) Curve of time optimization, b) curve of mass of adsorbent optimazation, c) curve of temperature optimazation and d) curve of acidity optimazation





$$\log \frac{x}{m} = \log k + \frac{1}{n} \log P \tag{5}$$

$$\frac{x/m}{P} = \frac{1}{a} + \frac{b}{a}P \tag{6}$$

$$\log q_e = \log k + \frac{1}{n} \log C_o \tag{7}$$

$$\frac{q_e}{C_o} = \frac{1}{a} + \frac{b}{a}C_o \tag{8}$$

From the curve of q_e/C_o versus C_o at temperatures of 303, 313, 323 and 333 obtained the equilibrium constant at each temperature (Table 1). From the curve of ln *K* versus 1/T, thermodynamic parameters (ΔH^o , ΔS^o and ΔG^o) are obtained 189.37 J·mol⁻¹,-1.08 J·mol⁻¹·K⁻¹ and 132.63 J·mol⁻¹respectively. This shows that the adsorption process is physically carried out exotherm and not spontaneously when the temperature is raised.

| Table 1. Equilibrium adsorption constant | | | |
|--|--------|--------|---------|
| Т | Κ | 1/T | ln K |
| 303 | 0.0186 | 0.0033 | -3.9829 |
| 313 | 0.0180 | 0.0032 | -4.0171 |
| 323 | 0.0168 | 0.0031 | -4.0853 |
| 333 | 0.0154 | 0.0030 | -4.1730 |



Figure 5. Curve of ln K versus 1/T

4. Conclusion

Activated bentonite can be used as an adsorbent for IPA in water. The optimum conditions of adsorption are stirring time of 40 minutes, mass of adsorbent 1 g, acidity 7, temperature of 30 °C and optimum adsorption capacity of 116.62 mg/g. The Langmuir isotherm model takes place physically, endotherm and not spontaneously if the temperature is raised.

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