

Characterization and application of diaion adsorbent (Sepabeds SP 700) for organic compounds removal from waste water of clove oil industry

Anton Restu Prihadi¹, Askal Maimulyanti^{2*}

¹Department of Food Industrial Quality Assurance, Politeknik AKA Bogor, Jl.Pangeran Sogiri No. 283 Tanah Baru, Bogor, Indonesia

²Department of Analytical Chemistry, Politeknik AKA Bogor, Jl.Pangeran Sogiri No. 283 Tanah Baru, Bogor, Indonesia

Abstract : Diaion SP-700 adsorbent (SepabeadsTM SP700) was evaluated as adsorbent to removal of organic compound from wastewater of essential oil industry. This research used waste of clove oil was produced from hydrodistillation process of *Syzigium aromaticum*. Diaion resin was used in the experiment without any chemical treatment. Identification of functional group from diaion SP-700 used infra red (IR) spectroscopy. The potential use of diaion adsorbent for removal organic compounds were investigated as chemical oxygen demand (COD) that indicated the organic matter that found in the wastewater. The result showed the spectrum of IR from diaion adsorbent with wavenumber were 3020 cm⁻¹, 2923 cm⁻¹, 1601 cm⁻¹, 1486 cm⁻¹, 1360 cm⁻¹, 893 cm⁻¹, 827 cm⁻¹, 791 cm⁻¹, 707 cm⁻¹, 568 cm⁻¹. The functional group were found as C-H and N-H (3000 cm⁻¹), group of C-O, C-S, and C-Cl (650-1000 cm⁻¹). The result showed variation of volume from wastewater can decrease of COD content. Variation of volume from wastewater were 10 to 20 ml can decrease of COD content from 5447.12 mg/L to 5248.32 mg/L. Dose of adsorben was 0.5 gram can decrease of COD from 5334,5 to 310,128 mg/L but if dose of resin was increase to 2 gram caused the increase of COD value to 8524.54. The recovery of clove oil from diaion resin resulted of eugenol, eugenol acetate dan methyl ester with composition of 73,31 %, 6,21% , and 0,43 %, respectively.

Keywords : Diaion resin, organic compound, waste water, clove oil.

1. Introduction

Clove (*Syzigium aromaticum*) is an aromatic herb that has many useful purposes. The clove oils have been known to stimulate and desintect body as it travels though the body. The essential oil from clove bud was

extracted by hydrodistillation process. The main compounds of clove oil were eugenol, caryophyllene and eugenyl acetate¹. Hydrodistillation process from clove oil in industry can produced the wastewater that contain of organic matter.

Adsorption technology is currently being used extensively for the removal of organic and inorganic micropollutants from aqueous solution. Activated carbon is the most widely used for the removal of variety of organic from water. Adsorbent using syntetic resins that may facilitate a cheap and effective chemical regeneration process². The wide variation in surface area, functionality and porosity of polymeric absorbents reveal the possibility of the resin for selective removal of specific organics. Diaion resin usually was used in removal of trace metal based on solid phase extraction. Daion HP-2MG resin was used for removal selenium³, diaion WA21J for Rh (III)⁴, Diaion SP-850 for trace metal⁵.

There are several methods suggested for removal organic compounds from aqueous solution. Among those methods, the ion-exchange process is most extensively used. It is well known that chelating resins containing functional group. Solid phase microextraction can be used to recovery of volatile aroma component from aqueous waste using activated carbon⁶. Adsorption process is efficient for the removal of organic matter from waste effluents. Activated carbons are the most widely used adsorbents due to their excellent adsorption abilities for organic pollutants⁷. Limited study about removal organic compounds using diaion adsorbent. In this study, preliminary column test was performed using diaion resin SP 700 for organic compounds removal from waste water of clove bud. The aim of this study were to characterized and removal organic compounds from wastewater clove bud oil using diaion resin SepabedsTM SP 700.

2. Experimental

2.1. Resin Characterization.

Resin characterization was done to identification of functional group from diaion resin. Characterization was use of FTIR (*furier transform infra red*).

2.2. Variation of adsorbent weight

The adsorption process was done by using chromatography column. Variation in resin weight were 0.5 to 2 g. A total of 10 mL of solution was inserted into column that containing of diaion resin. The solution was allowed to contact with diaion resin and was removed from the resin.

2.3. Effect of volume to sorption of organic compounds

Clove oil absorption process was done by using chromatography column. The volume was varied with 10 to 25 ml. The sample was inserted into a column that containing diaion resin weighing of 0.5 g. The solution was allowed to contact and was removed from the resin. After the adsorption process the obtained solution was measured of COD content.

2.4. Determination of COD content

COD (Chemical oxygen demand) is the amount of oxygen needed to break down organic and inorganic compounds in 1 liter of chemical samples. Determination of COD was carried out by oxidation process using oxidator $K_2Cr_2O_7$. This chemical oxidation reaction will oxidize organic substances that can not be oxidized perfectly such as cellulose. The principle is that most of the organic compounds in water can be oxidized by boiling water that has been added chromate and sulfuric acid. After the destruction of the non-reducible potassium dichromate was titrated with a ferrous ammonium sulfate solution to determine the amount of bichromate used to oxidize the organic material.

3. Results and discussion

3.1. Spectrum IR of diaion resin

Analysis of functional group in diaion resin by FTIR analysis can be seen in Figure 1.

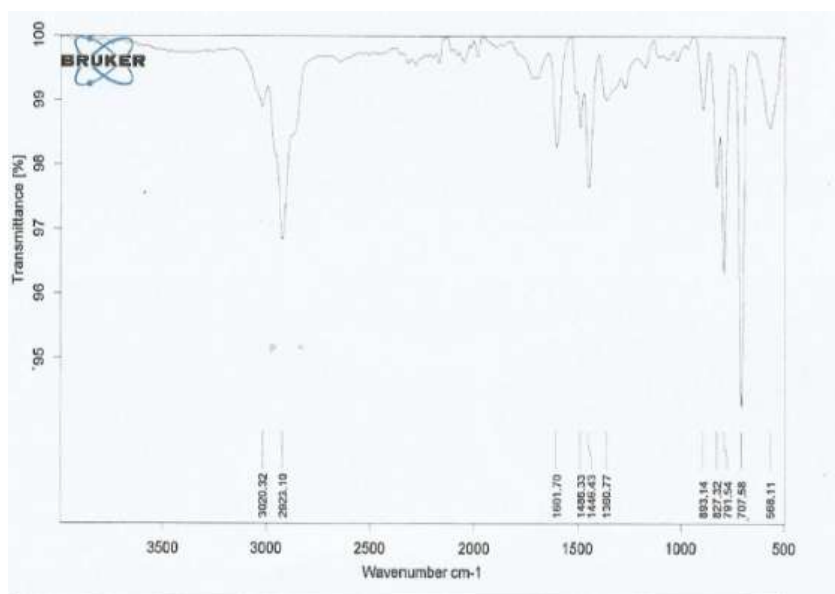


Figure 1. IR Spectrum from diaion resin

Based on the spectrum obtained by the peak on the wave number 3020 cm^{-1} , 2923 cm^{-1} , 1601 cm^{-1} , 1486 cm^{-1} , 1360 cm^{-1} , 893 cm^{-1} , 827 cm^{-1} , 791 cm^{-1} , 707 cm^{-1} and 568 cm^{-1} . The functional group identification of IR spectrum estimated the active group present in the adsorbent which may be involved in the absorption process. Diaion resin that usually appears in the group of 3000 cm^{-1} , C-O, C-S and C-Cl commonly present in $650\text{-}1000\text{ cm}^{-1}$.

3.2. Morphology of diaion resin by SEM

Analysis of morphology from diaion resin adsorbent was identified by SEM. The result can be shown in Figure 2.

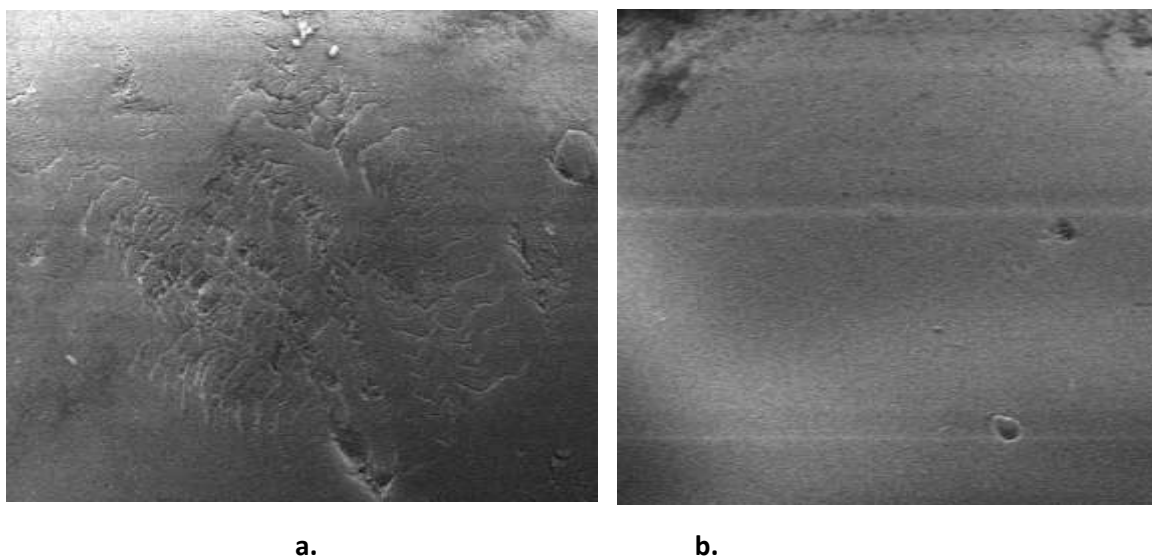


Figure 2. Surface analysis of diaion resin by SEM

a. before adsorption b. after adsorption

Figure 2 shows the surface analysis of diaion resin by SEM analysis. The surface of diaion resin was flat and there was porous to binding of analyte. The surface structure after adsorption process shown the flat surface and the porous has been covered by the organic compounds. It was indicated the organic compound has been adsorbed by the diaion resin.

3.3. Effect of wastewater volume to sorption of organic matter

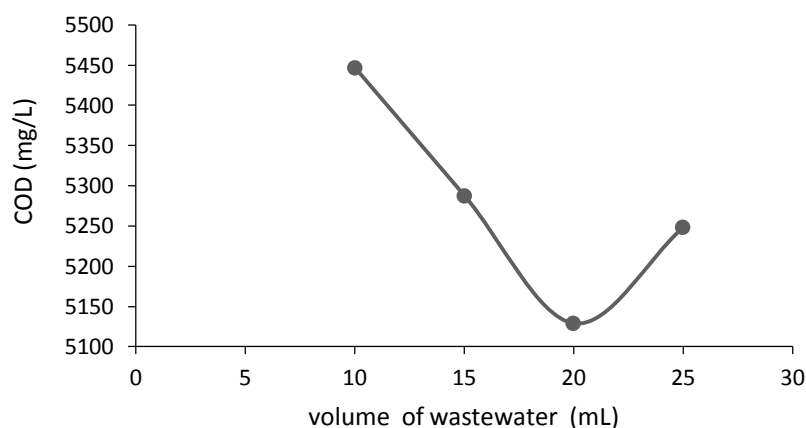


Figure 3. Effect of volume to COD content

Based on Figure 3 we can see a decrease in COD value with increasing waste volume from 10 to 20 mL. But after 20 mL volume COD value increases because after optimum absorption occurs again the process of release of organic compounds into the solution. The optimum absorption was obtained at 20 mL waste volume by decreasing the COD value from 5447.12 to 5129.04 mg / L.

3.4. Effect of resin weight to sorption of organic matter

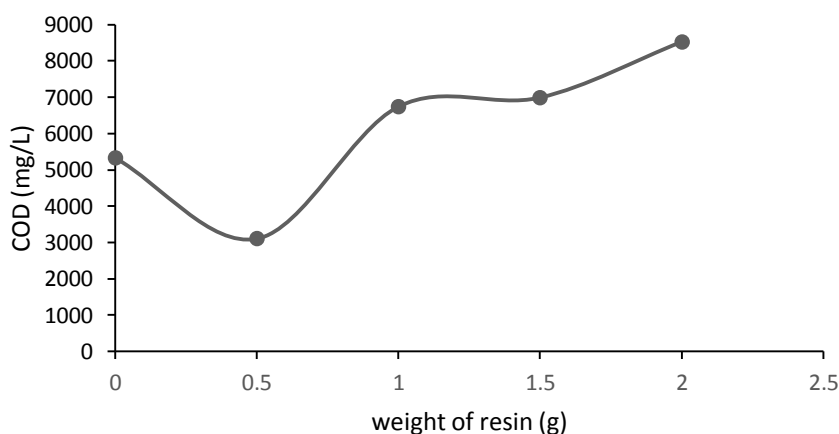


Figure 4. Effect of resin weight to COD content

In the research was done variations of resin weight to absorb 10 ml of waste. Based on Figure 4 it can be seen that COD content was decrease on the addition of resin 0.5 g. This showed the absorption response of organic compounds in the resin. The value of COD increases with increasing of resin weight. This indicated that the resin capability is not effective for absorption due to saturation and pores for absorption to become closed.

3.5. Recovery of clove oil from diaion resin

Recovery of clove oil from diaion resin by eluted with ethanol. The identification of the oil use gas chromatography- massa spectrometry (GCMS) and the result can be shows in Table 1.

Table 1. Chemical composition of clove oil after adsorption with diaion resin

| No | tR(minute) | composition (%) | Compounds |
|----|------------|-----------------|------------------------------|
| 1 | 16,013 | 73,31 | Eugenol |
| 2 | 21.020 | 6,21 | Eugenol acetate |
| 3 | 23,111 | 0,32 | β -Caryophyllene |
| 4 | 24,956 | 0,27 | Patchoulic alcohol |
| 5 | 25,644 | 0,12 | Butanic acid |
| 6 | 26,330 | 0,26 | Callamene |
| 7 | 35,217 | 0,28 | N-ethyl-1,3 dithiosoindoline |
| 8 | 36,319 | 0,43 | Methyl ester |
| 9 | 37,907 | 0,60 | E-1-(4-metoxyphenyl) propene |

Based on Table 1, we can see the main composition of clove oil after adsorption with diaion resin is eugenol, eugenol acetate and methyl ester with composition of 73,31 %, 6,21% and 0,43 %, respectively.

3. Conclusion

The potential use of diaion adsorbent for removal organic compounds were investigated as chemical oxygen demand (COD) that indicated the organic matter that found in the wastewater. The result showed the spectrum of IR from diaion adsorbent with wavenumber were 3020 cm^{-1} , 2923 cm^{-1} , 1601 cm^{-1} , 1486 cm^{-1} , 1360 cm^{-1} , 893 cm^{-1} , 827 cm^{-1} , 791 cm^{-1} , 707 cm^{-1} ; 568 cm^{-1} . The functional group were found as C-H and N-H (3000 cm^{-1}), group of C-O, C-S, and C-Cl (650-1000 cm^{-1}). The result showed variation of volume from wastewater can decrease of COD content. Variation of volume from wastewater were 10 to 20 ml can decrease of COD content from 5447.12 mg/L to 5248.32 mg/L. Dose of adsorbent was 0.5 g can decrease of COD from 5334,5 to 310,128 mg/L but if dose of resin was increase to 2 g caused the increase of COD value to 8524.54. The recovery of clove oil from diaion resin resulted of eugenol, eugenol acetate dan methyl ester with composition of 73,31 %, 6,21% , and 0,43 %, respectively.

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