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Potential Adsorption of Heavy Metal Ions by Eugenol Compounds and Derivatives through Ion Imprinted Polymer

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Abstract

Research on the potential of Ion Imprinted Polymer (IIP) selective adsorption of heavy metals using eugenol compounds and their derivatives has been carried out. Isolation and synthesis of eugenol derivatives with metal selective active groups and their use as selective metal carriers have been carried out with satisfactory results. Carrier effectiveness can still be improved by methods that focus on the target molecule recognition model. This adsorption method is called Ion Imprinted Polymer (IIP). The main components of IIP are functional monomers, crosslinkers, and target molecules. The use of acrylamide and its derivatives as functional monomers is useful with a lot of success achieved but also invites danger because it includes carcinogenic substances, a nerve poison, and so on. Moreover, the N group, which is an active acrylamide group, and its derivatives are only selective towards borderline metals (HSAB theory). Alternatives that are safe and can increase their selectivity are therefore needed. Eugenol, with its three potential functional groups, is believed to be able to replace the function of acrylamide and its derivatives that can even increase the effectiveness of IIP. The purpose of this study is to determine the potential of eugenol derivatives as selective adsorbents through the IIP method. This synthesis of IIP involved the use of basic ingredients of eugenol and its derivatives (polyeugenol, EOA, polyacetate). Each base material is contacted with a metal template then crosslinked with three kinds of crosslinking agents, namely EGDMA, DVB, and bisphenol. IIP is formed after the metal template is released using acid/HCl. The outcomes obtained demonstrate that the IIP method is able to increase the metal adsorption capacity and that the IIP method for metals is largely determined by the release of metals, which will form a hole for metal entry through adsorption. Poly-Cd-DVB, Eug-Cr-DVB, Poly-Cu-bisphenol, Polyacetate -Cr-DVB are polymer materials that have the potential to make up an IIP.

1. Introduction

Indonesia is rich in natural resources that have not been utilized maximally. As a major essential oil-producing country in the world, one of which is clove leaf oil, Indonesia fulfilled almost half of the world's clove leaf oil needs in the early eighties [1]. Clove leaf oil contains around 80–90% eugenol by weight [2]. Eugenol can be used as a starting material for the synthesis of a compound due to the presence of three functional groups

attached to it, namely the allyl, hydroxy, and methoxy groups. Allyl groups play a role in cross-linking and polymerization into polyeugenol [3, 4]. Through its hydroxyl group, groups with greater activity can be synthesized, such as carboxylates [5, 6] and esters [7]. The hydroxyl group becomes an active group that binds a target metal ion as a mold. Most crosslinkers work with hydroxyl groups on eugenol and their derivatives. As can be found in EGDMA and bisphenol, while other crosslinkers work using their vinyl groups such as DVB.

The eugenol derivatives have been proven to be able to be used as liquid membrane carriers with a selectivity that can be adjusted depending on the functional group incorporated. In addition, eugenol, with its three functional groups, also has the potential to become a functional monomer for selective adsorption processes such as a molecularly imprinted polymer (MIP).

Ion imprinted polymer (IIP) is a synthetic material with synthetic sites (regions) that can bind a target molecule in spite of other very similar molecules. This material is obtained by polymerization and cross-links of functional monomers around the printed molecule, causing the formation of a large three-dimensional network of cross-linked polymers. The functional monomer chosen must be able to interact with the molecular function group of the mold. When polymerization takes place, the mold molecules are extracted, and bonding sites with excellent shape, size, and selectivity to the target analytes are formed. The printed polymers produced are stable, strong, and resistant to ranges of pH, solvent, and temperature. Thus, the nature of IIP exceeds the interactions that occur between natural receptors and selectively restrained target molecules (such as antibodies-antigens) but with no limits instability. In addition, IIP synthesis is also relatively inexpensive and easy, making it an excellent choice compared to using natural receptors [8].

Some of the advantages of MIP that have been reported are the use in glucose sensors that respond to glucose 7 times more selectively than fructose [9], absorbance of only glucose and not fructose [10], absorbance of dinicole (fungicide) four times better than compounds that are structurally very similar (differentiated only by one bound Cl atom), adsorbs 100% hemoglobin [11], separates hemoglobin from vine serum albumin [12], adsorbs pinacolyl methylphosphonate (PMP), a pesticide component far better than similar pesticide compounds, diethyl phosphite [13], determining the levels of simetryn, ametryn and prometryn (triazine herbicide compounds) in river water [14], purifying vanillin from lignin waste [15], matrix of drug release [16] and so on.

Up until now, researchers use acrylamide or its derivatives (methyl acrylamide) as functional monomers [17, 18, 19]. The results obtained are indeed quite good, that is, IIP that is functional for various applications. However, acrylamide and its derivatives have very dangerous potentials, including poisoning the nervous system and causing carcinogenic effects [20]. The toxicity of acrylamide and its derivatives limit wider application that thus needs alternatives. Chitosan is used as a functional monomer by various researchers [12]. Although the results are quite good, there is a need for additional compounds that have vinyl groups for polymerization. With its three functional groups, eugenol is believed to be able to act as a safe and potential functional monomer able to increase IIP selectivity.

Ion Imprinted Polymer (IIP) is a type of sorbent used in the printing of ions based on the formation of complexes of metal ions by ligands and subsequent immobilization in the polymer crosslinking matrix. After the release of metal ions from a spatially bound polymer, it then leaves the site of the sorbent structure [21]. Seeing the potential role of IIP in life and eugenol as a safe and potential functional monomer, it is, therefore, necessary to research the use of eugenol as an IIP functional monomer. This article is a preliminary study to discover polymers made from eugenol derivatives able to be developed in further research.

2. Methodology

The following are the equipment and materials used in this study.

2.1. Equipment and Materials

Equipment used: A set of glassware, Column with 4.6 mm inner diameter and 50 mm length, FTIR (Shimadzu 8201PC), SEM (Shimadzu), AAS (Perkin Elmer). The materials used are Eugenol (Sigma Aldrich), Crosslinker (ethylene dimethacrylate, p-divinylbenzene, and bisphenol) (Sigma Aldrich), Initiator (Azobisisobutyronitril/AIBN) (Sigma Aldrich), solvents (chloroform, toluene), HNO₃, and HCl (Emcrck). Along with metals ions in the form of Cd(NO₃)₂, Cu(NO₃)₂, Cr(NO₃)₂ (Merck)

2.2. Experiment Procedure

1. Synthesis of eugenol derivatives is in followed the procedure [5]

2. Metal extraction with eugenol and its derivatives.

100 ppm of the Cu²⁺ /Cd²⁺ /Cr³⁺ target metals were extracted (stirred, shook, and mixed) with 0.5 grams of eugenol and its derivatives in 15 mL chloroform. It was then separated using a separating funnel. The eugenol and its derivatives are evaporated while the metal solutions were analyzed with AAS.

3. Synthesis of IIP

a. Certain ratios of eugenol and its derivatives – metal complex to crosslinkers were used in the reaction:

b. Eugenol-metal: divinylbenzene (10% and 20%). Polymerization was carried out by adding 0.25 mL BF₃ diethyl ether catalyst for 4 hours, and polymerization was left to occur overnight. Methanol was then added to stop polymerization [22]. The product of polymerization was dissolved in diethyl ether and then neutralized with distilled water.

c. Eugenoxy acetate- metal: ethylene methacrylate (3 mmol: 5 mmol; 3 mmol: 10 mmol; 3 mmol: 20 mmol) [8, 20] was inserted in a glass polymerization tube and mixed with AIBN (50 mg). The tube then flowed with nitrogen gas for 10 minutes under vacuum condition. Polymerization was conducted in an oil

bath with a temperature maintained at 110°C for 6 hours.

- d. Poly Eugenol-metal (0.164 gr): bisphenol (0.17gram): 1 M NaOH (4.0 mL) were dissolved in dioxane (2 mL) solvent. in an oil bath with a temperature maintained at 110°C for 6 hours [23].

4. Synthesis of blank non-imprinted polymer

The procedure for making NIP is the same as IIP, excluding the metal extraction stage.

- 5. Analysis of adsorbents formed using FTIR, SEM, and NMR

6. Release of metal with HCl 6 N

The adsorbent/solid obtained was removed from metals by using HCl 6 N in Erlenmeyer with the agitation of a shaker and stirrer for 24 hours. The metals released were analyzed by AAS.

7. IIP and NIP selectivity analysis

0.05 g IIP and NIP (non-imprinted polymer) polymers were put in separate Erlenmeyer flasks for the adsorption of 15 mL heavy metal mixture (Cu²⁺, Cd²⁺ dan Cr³⁺), each 10 ppm for 24 hours.

3. Results and Discussion

3.1. Metal extraction with Eugenol and its derivatives

The formation of the template on the adsorbent was obtained through solvent extraction between a single metal and functional monomers (eugenol and its derivatives).

Table 1. Metal extraction with various extractants of 0.5 grams eugenol derivatives (except 1 gram eugenol in 15 mL, 15 mL Cu²⁺, Cd²⁺ dan Cr³⁺ 10 ppm each)

| Carrier Compound | Metal | % Extracted | | |
|------------------|------------------|-----------------|------------------|---------------|
| | | 1 Hour Stirring | 24 Hour Stirring | Shaker Mixing |
| Eugenol | Cu ²⁺ | 4.4 | 8.42 | - 68.79 |
| | Cd ²⁺ | 0 | 40.9 | - 9.86 |
| | Cr ³⁺ | 0.97 | 12.5 | - 81.64 |
| EOA | Cu ²⁺ | 98.9 | 25.68 | 1.6 75.32 |
| | Cd ²⁺ | 0 | 38.22 | 14.94 11.73 |
| | Cr ³⁺ | 0 | 30.54 | 68.75 88.5 |
| Polyeugenol | Cu ²⁺ | - | 10.37 | 0 76.4 |
| | Cd ²⁺ | - | 0 | 17.17 12.29 |
| | Cr ³⁺ | - | 12.99 | 17.5 90.62 |
| Polyacetate | Cr ³⁺ | - | - | - 89.45 |

It can be seen from Table 1 that, in general, the mixing technique (stirring from above with a stirring rod) achieved better results than the technique using stirrer or shaker. This is possible because with mixing, maximal contact between the extractant and the metal occurred and could not easily be released again (leaching). Furthermore, the organic phase (eugenol and its derivatives – metal complex) was separated from the

aqueous phase (containing remaining metals not involved in the reaction/complexation/extraction) was then evaporated and used as a template.

3.2. Crosslinking of eugenol and its derivatives – metal complex with a crosslinker

The eugenol and its derivatives – metal complex obtained from the extraction was then evaporated to remove its organic solvents to be used as a powder as an initial material that is also in the form of powder, such as polyeugenol and polyacetate. The product obtained was then weighed and crosslinked using various crosslinkers. The obtained masses of powders are shown in Table 2. NIP blank sample was also made using the same procedure but without the complexation stage with metals

Table 2. Weight of various eugenol and their derivatives.

| No | Adsorbent | Mass (gram) |
|----|--------------------------------------|-------------|
| 1 | Poly-Cd extraction product (mixing) | 0.29 |
| 2 | Poly- Cd extraction product (mixing) | 0.11 |
| 3 | Poly- Cd extraction product (mixing) | 0.21 |
| 4 | Poly-Cu-shaker | 0.17 |
| 5 | Polyeug-dvb 20% | 0.75 |
| 6 | Polyacetate DVB 20% | 0.81 |
| 7 | Eug-DVB 10% | 1.62 |
| 8 | Poly-bisphenol | 0.38 |
| 9 | EOA-EGDMA 0,8 mL | 1.01 |
| 10 | EOA-EGDMA 0,4 mL | 0.64 |
| 11 | EOA-Cr-EGDMA (mixing) | 0.75 |
| 12 | EOA-Cu-EGDMA (mixing) | 1.39 |
| 13 | Mixed poly-Cd-bisphenol | 0.42 |
| 14 | Mixed poly-Cr-bisphenol | 0.19 |
| 15 | Eug-Cr-DVB-20% (media) | 0.25 |
| 16 | Eug-Cd-DVB 20% (media) | 0.55 |
| 17 | Poly-Cr-DVB 20% | 0.14 |
| 18 | Poly-Cd-DVB 20% | 0.23 |
| 19 | Poli-Cu-DVB 20% | 0.16 |

FTIR and SEM results of adsorbent complexes of eugenol derivatives can be seen in Figures 1 and 2 below. The FTIR spectra of EOA-Cr-EGDMA compared to the spectra without metal is presented.

As shown in Figure 1 (a), the FTIR spectra of one of the eugenol derivatives (eugenoxo acetate (EOA) bound to the Cr ion template and crosslinked with EGDMA when compared to Figure 1 (b) (without the Cr template) do not show a significant difference. Seen from the FTIR spectra, absorption at 1728 cm⁻¹ indicates the presence of EGDMA carbonyl ester (C = O) groups [24] crosslinked with monomers. Binding with template ions is expected to give a change in the fingerprint region. The absence of significant change is possibly caused by the low concentration of Cr template (50 ppm). However, SEM images differ notably between NIP and Eugenol-Metal-Crosslinker derivatives, as shown in Figure 2. SEM images of Eugenol-Metal-Crosslinker derivatives when

compared to the adsorbent without metal show more open pores, especially in pictures e and f.

3.3. Metal Removal

The metal that was printed on the adsorbent was released with an acid (HCl 6 N) by stirring for 24 hours. It was then filtered and analyzed with an Atomic Absorption Spectrophotometer (AAS). The results obtained are

presented in Table 3. As shown in the table, most metals are rather difficult to be released again with acid (HCl solution), which is likely to affect the performance of IIP. Other studies use HNO₃ for the removal of metals [25]. Djunaidi *et al.* [25] has compared the Fe metal releasing eluents in various concentrations. The eluents used include HNO₃, HCl, and Na₂EDTA-HCl.

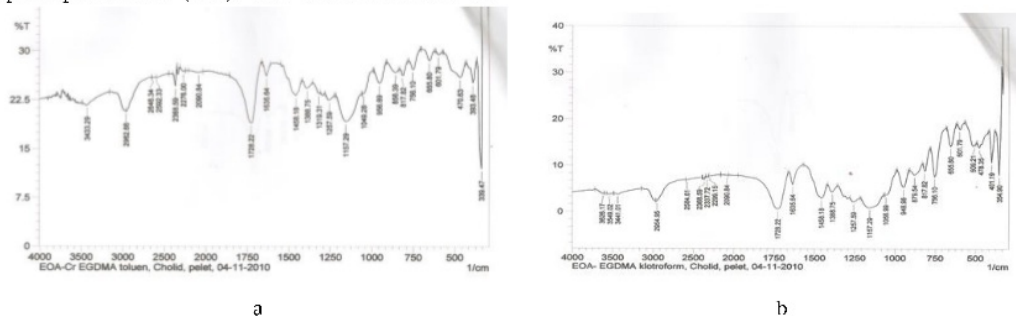


Figure 1. FTIR spectra of (a) EOA-Cr-EGDMA and (b) EOA-EGDMA

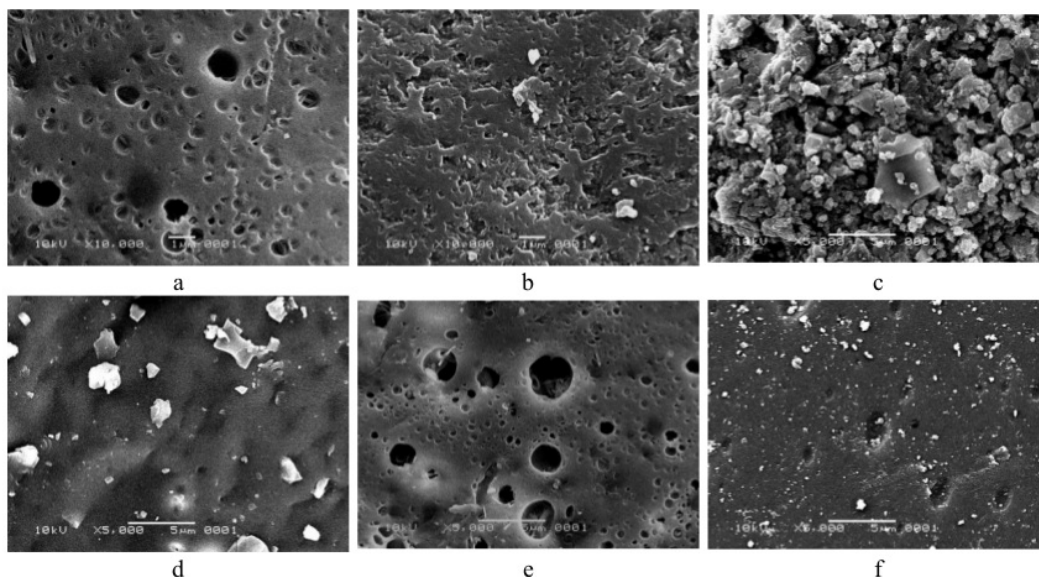


Figure 2. SEM images of (a) EOA-Cr-EGDMA, (b) EOA-EGDMA, (c) Eug-Cd-DVB 10%, (d) Eug-DVB 10%, (e) Poly-Cr-DVB, (f) Poly-DVB

Table 3. Removal of metal from adsorbents

| No | Adsorbent | Cd ²⁺ /Cu ²⁺ /Cr ³⁺ Metal (mg/gram) | Extractant-metal complex (mg/gram) |
|----|--------------------------------------|--|------------------------------------|
| 1 | Poly-Cd-Bis (shaker; 24 hours) | 0.021 | 0.495 |
| 2 | EOA-Cu-EGDMA (shaker; 24 hours) | 0.103 | 1.557 |
| 3 | Poly-Cu-bisphenol (shaker; 24 hours) | 0.017 | 1.579 |
| 4 | EOA-Cr-EGDMA (shaker; 24 hours) | 0.052 | 1.943 |
| 5 | Poly-Cr-bisphenol (shaker; 24 hours) | 0.087 | 3.480 |
| 6 | Poly-Cd-bis (stirrer; 24 hours) | 0.013 | 0.495 |
| 7 | Poly-Cd-DVB | 0.009 | 0.495 |
| 8 | Eug-Cd-DVB 20% | 0.009 | 0.398 |
| 9 | Poly-Cr-DVB | 1.003 | 3.480 |
| 10 | EOA-Cr-EGDMA | 0.038 | 1.943 |
| 11 | Eug-Cr-DVB | 0.243 | 3.135 |
| 12 | Poly-Cu-DVB | 0.024 | 1.579 |
| 13 | Poly-Cu-bisphenol | 0.007 | 1.579 |
| 14 | EOA-Cu-EGDMA | 0.088 | 1.557 |
| 15 | Eug-Cr-DVB | 0.284 | 3.135 |
| 16 | Polyacet-Cr-DVB | 0.143 | 3.435 |
| 17 | Eug-Cu-DVB | 0 | 1.422 |
| 18 | Eug-Cd-DVB | 0.016 | 0.398 |

3.4. Ion Imprinted Polymer

After removal of the metal, the adsorbent was filtered and dried to be used to adsorb other metal. Ideally, if the adsorbent is still “full” of metal, it will be difficult to adsorb metal from the solution. The results obtained can be seen in Table 4.

As shown in Table 4, in general, after the metal is released, the adsorption for metal becomes greater, as shown by adsorbents no. 4, 7, 9, and 10. This is largely influenced by the amount of metal that can be released from the mold that is expected to leave a “hole”. A concrete example of this is the Poly-Cr-DVB adsorbent, where the Cr³⁺ metal successfully released amounts to 1,003 mg/gram. The hole left behind when used to adsorb metal alloys can increase the adsorption capacity for Cr³⁺ metal. This is the result of the influence of the template that prompts a memory effect on the -OH groups that would instigate the occurrence of coordination interactions with the metal. In addition, the corresponding geometrical factors and the affinity and size of the metal ions and polymers have the potential for these polymers to be developed as IIP material for the metals observed.

Table 4. Various kinds of adsorbents for the adsorption of metals by the IIP method

| No | Adsorbent | IIP metal (mg/g) | | |
|----|-------------------------------------|------------------------|------------------|------------------|
| | | NIP blank metal (mg/g) | | |
| | | Cd ²⁺ | Cu ²⁺ | Cr ³⁺ |
| 1 | Poly-Cd-Bis (shaker; 24 hours) | 0.230 | 0.246 | 0 |
| | | 0.621 | 0.559 | 0.530 |
| 2 | EOA-Cu-EGDMA (shaker; 24 hours) | 0.039 | 0.065 | 0 |
| | | 0 | 0.117 | 0.227333 |
| 3 | EtOA-Cr-EGDMA (shaker; 24 hours) | 0.03 | 0.105 | 0 |
| | | 0 | 0.117 | 0.227 |
| 4 | Poly-Cd-DVB mixing | 0.273 | 0.064 | 0 |
| | | 0 | 0.039 | 0.174 |
| 5 | Eug-Cd-DVB 20% | 0 | 0.028 | 0.018 |
| | | 0.002 | 0.047 | 0 |
| 6 | Poly-Cr-DVB | 1.026 | 0.925 | 1.054 |
| | | 0 | 0.039 | 0.174 |
| 7 | Eug-Cr-DVB | 0 | 0.009 | 0.013 |
| | | 0.002 | 0.047 | 0 |
| 8 | Poly-Cu-DVB | 0.004 | 0.005 | 0.218 |
| | | 0 | 0.038667 | 0.174 |
| 9 | Poly-Cu-bisphenol | 0.112 | 0.151 | 0 |
| | | 0.621 | 0.559 | 0.530 |
| 10 | Polyacet-Cr-DVB | 0 | 0.096 | 0.611 |
| | | 0.089 | 0.103 | 0 |
| 11 | Eug-Cu-DVB | 0 | 0.029 | 0.019 |
| | | 0.002 | 0.047 | 0 |
| 12 | Eug-Cd-DVB 10% | 0 | 0.028 | 0.018 |
| | | 0 | 0.028 | 0.053 |

4. Conclusion

The IIP method is able to increase metal adsorption capacity. This method for metal adsorption is largely determined by the release of metal that will leave holes for other metal to enter through adsorption. Poly-Cd-DVB, Eug-Cr-DVB, Poly-Cu-bisphenol, Polyacet-Cr-DVB are potential polymer materials for IIP.

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