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THE EFFECT OF THE RATIO OF METHACRYLIC ACID AND ETHYLENE GLYCOL DIMETHACRYLATE ON THE ADSORPTION CAPACITY OF ION-IMPRINTED POLYMERS (IIPs) FOR Cr^{3+} IONS

N.D. Sella[1], U. Alizar[1,2,✉], Yulkifli[3], A. Syamsi[1], S.A. Hanifah[4], and R.A. Hassan[5]

[1]Department of Chemistry, Faculty of Mathematics and Natural Sciences, Universitas Negeri Padang, Jl. Prof. Hamka, Padang 25131, Indonesia

[2]Biosensor & Chemical Sensor Research Centre, Department of Chemistry, Faculty of Mathematics and Natural Sciences, Universitas Negeri Padang, Jl. Prof. Hamka, Padang 25131, Indonesia

[3]Department of Physics, Faculty of Mathematics and Science, Universitas Negeri Padang, Jl. Prof. Hamka, Padang 25131, Indonesia

[4]Department of Chemical Sciences, Faculty of Science and Technology, Universiti Kebangsaan Malaysia, 43600 UKM Bangi, Selangor, Malaysia

[5]Department of Chemistry, Faculty of Science, Ibb University, Ibb, Yemen

✉Corresponding author: alizarulianas@fmipa.unp.ac.id

ABSTRACT

Cr^{3+} metal ions are hazardous contaminants commonly found in industrial waste, particularly from the metal plating, leather tanning, and textile industries. One promising method to address this issue is adsorption using Ion-Imprinted Polymers (IIPs), a type of polymer material with high selectivity toward target ions. This study aims to analyse the effect of the ratio of Methacrylic Acid (MAA) and Ethylene Glycol Dimethacrylate (EGDMA) in IIP synthesis on the adsorption capacity for Cr^{3+} ions. The synthesis process was carried out via polymerisation using $CrCl_3$ as the imprinting ion, MAA as the functional monomer, and EGDMA as the crosslinker. Adsorption capacity was evaluated by immersing the polymer in a 20 ppm Cr^{3+} solution and measured using an atomic absorption spectrophotometer (AAS). The results showed that the optimal ratio of MAA and EGDMA, each at 0.015 mol, yielded the highest adsorption capacity, namely 1.95 mg/g. Increasing the amount of MAA and EGDMA beyond this optimal value reduced adsorption efficiency due to the formation of non-specific binding sites and an overly rigid polymer structure. This indicates that the balance between the functional monomer and crosslinker ratios significantly influences the selectivity and effectiveness of IIPs in adsorbing Cr^{3+} ions. Ion Imprinted Polymers (IIPs) can dramatically contribute to addressing environmental pollution caused by heavy metal ion contamination, especially in water and soil. Furthermore, IIPs can also be applied to design electrochemical sensor transducers to detect Cr^{3+} ions.

Keywords: Ion-Imprinted Polymers (IIPs), Photopolymerization, Adsorption Capacity, Template Extraction, Template adsorbing, Clean Water and Sanitation.

INTRODUCTION

Chromium (Cr) is one of the heavy metals widely used in various industries due to its corrosion-resistant, hard, and high-temperature-stable properties. In its compound form, chromium primarily exists in two main oxidation states: Cr^{3+} and Cr^{6+}.[1] Although Cr^{3+} plays a role in human metabolism in small amounts, at high concentrations it can be toxic, causing organ dysfunction, carcinogenic effects, and environmental contamination, particularly of water and soil.[2] Chromium pollution in the environment generally originates from industrial waste, particularly from the metal plating, stainless steel manufacturing, textile, and leather tanning industries.[3] Liquid waste from these processes contains significant amounts of chromium ions, along with other contaminants such as caustic chemicals, hazardous organic compounds,

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and dissolved solids. If not properly treated, this waste can contaminate water and directly impact ecosystems and human health.[4] Various methods have been used to remove heavy metal ions from liquid waste, such as precipitation, ion exchange, coagulation-flocculation, solvent extraction, and adsorption.[5] Adsorption is one of the preferred methods because it is simpler, more efficient, and environmentally friendly. However, conventional adsorbents often have drawbacks such as limited adsorption capacity, a lack of selectivity toward target ions, and high production costs.[6] Ion-imprinted polymers (IIPs) have emerged as a superior alternative adsorbent because they can recognise and selectively bind specific metal ions through ion imprints in their polymer structure.[7] IIPs offer advantages such as high chemical and physical stability, high selectivity toward target ions, and a long polymer shelf life.[8] The synthesis of IIPs can be carried out using various methods, one of which is photopolymerization, which offers the advantages of low operating temperatures, rapid processing, and energy efficiency.[9] In the synthesis of ion-imprinted polymers (IIPs), the selection of the ratio of functional monomers to crosslinkers largely determines the success of specific cavity formation and material stability. The choice of functional monomers is a key factor, as these compounds form pre-polymerised complexes through particular interactions between functional groups and target metal ions. Methacrylic acid (MAA) is one of the commonly used functional monomers because it can form complexes and strong electrostatic bonds with Cr^{3+} ions.[10] On the other hand, crosslinkers such as Ethylene Glycol Dimethacrylate (EGDMA) play an important role in forming a stable three-dimensional polymer structure. The use of EGDMA affects the morphology of the polymer matrix, the stability of the binding sites, and the mechanical strength of the material.[11] Therefore, this study aims to investigate the effect of varying the MAA-to-EGDMA ratio in IIP synthesis on adsorption performance and Cr^{3+} ion selectivity, to obtain materials with optimal adsorption efficiency. This research aligns with the Sustainable Development Goals (SDGs) on Clean Water and Sanitation.

EXPERIMENTAL

Materials and Instruments

The chemicals used in this research were chromium(III) chloride ($CrCl_3$), Methacrylic Acid (MAA), Ethylene Glycol Dimethacrylate (EGDMA), 2,2-Dimethoxy-2-Phenylacetophenone (DMPP), and nitric acid (HNO_3), all supplied by Sigma-Aldrich. The instruments used for polymerisation included a photopolymerization setup with ultraviolet radiation at wavelengths of approximately 250–350 nm, FTIR for functional group analysis, and Atomic Absorption Spectroscopy (AAS) for Cr^{3+} ion quantification.

Synthesis and Characterisation of Ion-Imprinted Polymer Cr^{3+}

Ion-Imprinted Polymer Cr^{3+} (IIP-Cr^{3+}) was prepared according to previously reported methods[12,1314] with slight modifications. A total of 5 mg of chromium chloride ($CrCl_3$) was mixed with various ratios of MAA to EGDMA (Table-1). Then, 0.03 grams of DMPP was added to each mixture. The mixture was sonicated until homogeneous (60 minutes). After homogenization, the mixture was poured into a Petri dish and photopolymerized for 15 minutes under UV light in a nitrogen gas atmosphere.

Table-1: The ratio of MAA and EGDMA

Sample Number	MAA (mol)	EGDMA (mol)
1	0.005	1
2	0.010	1
3	0.015	1
4	0.020	1
5	0.025	1
6	0.015	0.005
7	0.015	0.010
8	0.015	0.015
9	0.015	0.020
10	0.015	0.025

The extraction of Cr^{3+} ions from IIP-Cr^{3+} was carried out by soaking 0.1 g of IIP-Cr^{3+} in 10 mL of 0.1 M HNO_3 for 60 minutes. The adsorption analysis was then conducted by soaking the extracted IIP in a 20

ppm Cr^{3+} solution for 50 minutes. The adsorption capacity of IIP-Cr^{3+}, q (mg/g), was determined using the equation[15]: $q = (C_0 - C_t) / m$, where C_0 and C_t(mg/L) are the concentrations of Cr^{3+} in the solution before and after treatment, respectively, and *m* (g) is the mass of the adsorbent in 1 L of Cr^{3+} solution. The value of C_t was determined using AAS. Figure-1 shows the predicted synthesis reaction mechanism of IIP-Cr^{3+}.

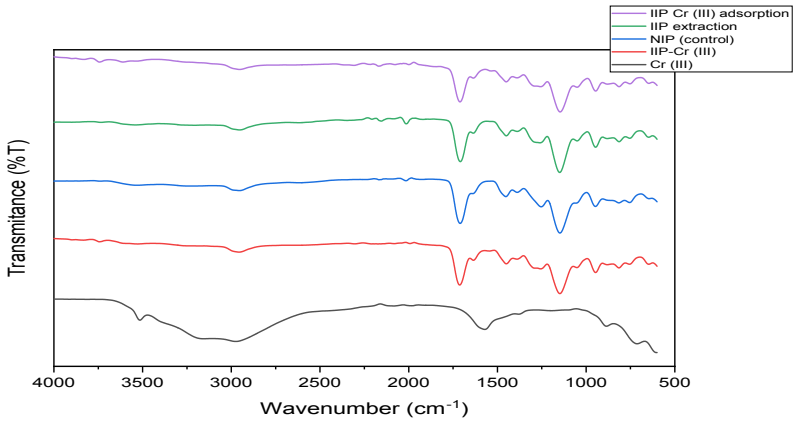

Fig.-1: Spectrum FTIR of Ion-Imprinted Polymers

RESULTS AND DISCUSSION

Ion-Imprinted Polymer Analysis

FTIR characterisation was performed to identify changes in functional groups that occurred during the synthesis of ion-imprinted polymers. Measurements were taken in the wavenumber range between 4000 and 500 cm^{-1}. This analysis was conducted on several samples, namely Cr^{3+} metal ions, IIP-Cr^{3+}, NIP, IIP after extraction, and IIP after adsorption. The FTIR spectra of each sample are shown in the following Figure: The FTIR spectrum of pure Cr^{3+} shows characteristic absorption bands at 600–550 cm^{-1} associated with Cr–O stretching vibrations, indicating interaction between Cr^{3+} ions and oxygen donor groups.[16] In the IIP-Cr^{3+} spectrum, the C=O band at 1725 cm^{-1} shifts and increases in intensity, indicating coordination between the carbonyl group of methacrylic acid and the Cr^{3+} ion. The C–O band at 1250–1145 cm^{-1} also shows a change in intensity, suggesting its involvement in the formation of the metal-polymer complex.[17] After the extraction process, the decreased intensity of the C=O, C–O, and O–H (3440 cm^{-1}) bands indicates the successful release of Cr^{3+} ions and the regeneration of active sites. Conversely, the NIP spectrum shows no significant shift, confirming the absence of specific interactions. After re-adsorption, the Cr–O band reappears with sharper intensity, and the C=O and C–O bands shift back to their original positions, indicating that Cr^{3+} ions were successfully recognized and re-bound into the selective pores. These changes in the position and intensity of the vibrational bands in the C=O (1720–1730 cm^{-1}), C–O (1250–1140 cm^{-1}), and Cr–O (600–550 cm^{-1}) regions confirm the success of the imprinting process and the selective ability of IIP to recognize and re-adsorb Cr^{3+} ions. This interaction is facilitated by oxygen donor groups acting as ligands in the formation of coordination complexes with metal ions

Effect of Molar Variation of Methacrylic Acid (MAA)

The selection of functional monomers and crosslinkers must be carefully considered, as they are crucial components in the interaction between target molecules and polymerisation components.[18] In this study, Methacrylic Acid (MAA) was used as a functional monomer because it contains a carboxyl group (-COOH) that can strongly interact with Cr^{3+} ions through ionic bonds.[10] Additionally, MAA possesses high chemical stability, is easy to polymerise, and enhances the strength and specificity of binding sites within the polymer structure.[19] The graph(Fig.-2) illustrates the effect of the amount of MAA on the adsorption capacity of Cr^{3+} ions.

As shown in Fig.-3, the adsorption capacity increased with increasing moles of MAA from 0.005 to 0.015 mol. The optimum MAA amount was 0.015 mol, with an adsorption capacity of 1.94 mg/g. This suggests that increasing the amount of MAA up to the optimum level enhances the formation of pre-polymerisation complexes with Cr^{3+} ions, resulting in more specific binding sites. However, at 0.020 mol,

the adsorption capacity decreased due to the excess MAA that does not interact optimally with the template ions, leading to non-specific binding sites.[10] This decline may also be attributed to an imbalance between functional monomers and crosslinkers, resulting in a less stable polymer structure.[20]

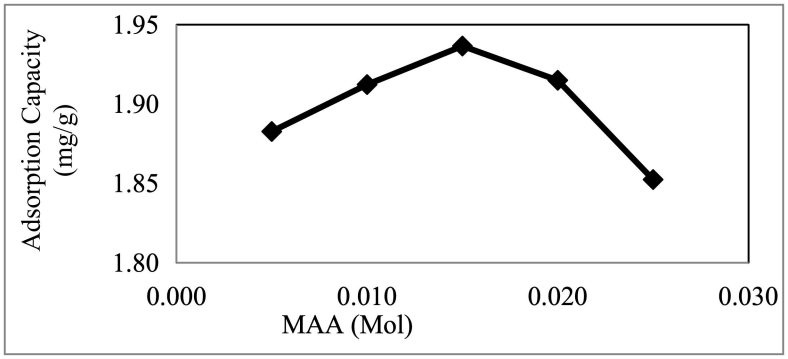

Fig.-2.Effect of MAA Amount on Cr^{3+} Adsorption Capacity

Effect of Ethylene Glycol Dimethacrylate (EGDMA) Composition

Next, variations in the amount of EGDMA were made using the optimum amount of MAA to evaluate the effect of crosslinker concentration on IIP adsorption capacity.[21] EGDMA was chosen as a crosslinker due to its methacrylate groups, which polymerise with MAA to form a robust polymer structure around the metal ion complex.[22] The appropriate amount of crosslinker is essential to maintaining the cavity stability and the polymer matrix structure.[20] Insufficient EGDMA may result in inadequate structural formation and reduced binding specificity, while excessive EGDMA can create a rigid polymer matrix, impeding template ion release and hindering Cr^{3+} ion diffusion to the binding sites.[22]

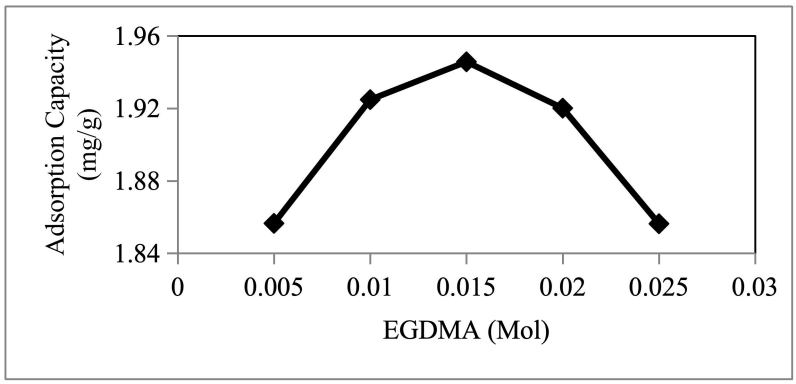

Fig.-3: Effect of EGDMA Amount on Cr^{3+}Adsorption Capacity

As shown in Fig.-4, the optimum amount of EGDMA for Cr^{3+} adsorption was 0.015 mol, yielding an adsorption capacity of 1.95 mg/g. Increasing EGDMA up to this point helped strengthen the polymer structure, maintain cavity shape, and facilitate Cr^{3+} ion diffusion into the binding sites.[23] However, further increases to 0.020 and 0.025 mol reduced the adsorption capacity due to the overly rigid structure, which hindered ion diffusion. These findings demonstrate that excessive crosslinker amounts negatively impact IIP performance.[20] The results indicate that the optimal MAA to EGDMA ratio for synthesising IIPs targeting Cr^{3+} ions is 0.015 mol:0.015 mol. This ratio achieves a balance between specific binding site formation and structural stability, resulting in the highest adsorption efficiency

CONCLUSION

Based on the experimental results, it can be concluded that varying the amounts of Methacrylic Acid (MAA) and Ethylene Glycol Dimethacrylate (EGDMA) significantly affects the adsorption capacity of Ion-Imprinted Polymers (IIPs) for Cr^{3+} ions. The optimal MAA to EGDMA ratio was found to be 0.015:0.015 mol, with adsorption capacities of 1.94 mg/g and 1.95 mg/g, respectively. Exceeding this ratio reduces efficiency due to the formation of non-specific sites and an overly rigid polymer structure.

Therefore, selecting the correct ratio of monomer to crosslinker is key to synthesising selective and efficient IIPs.

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CONFLICT OF INTERESTS

The authors declare that there is no conflict of interest.

AUTHOR CONTRIBUTIONS

The present work is related to *SDG-6: Clean Water and Sanitation*. All the authors contributed significantly to this manuscript, participated in reviewing/editing and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

N.D. Sella https://orcid.org/0009-0002-3170-2032
U. Alizar https://orcid.org/0000-0002-5940-9965
Yulkifli https://orcid.org/0000-0002-0909-6227
A. Syamsi https://orcid.org/0009-0008-5927-378X
S.A. Hanifah https://orcid.org/0000-0003-2123-7704
R.A. Hassan https://orcid.org/0000-0002-8279-6991

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