Comparison of the method used for extraction chloramphenicol from its Molecularly Imprinted Polymer (MIP) using chloroform as porogen

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Abstract—The synthesized of chloramphenicol – MIP (molecularly imprinted polymer) has been developed in many ways and many purposes. The purpose of this research was to compare the methods used for extracting chloramphenicol from its molecularly imprinted polymer (MIP) by calculating the percent of extraction in various ratio of template to monomer using chloroform as porogen. The result showed that the batch methods given a higher percent of extraction compared to the traditional methods.

Keywords—chloramphenicol, molecularly imprinted polymer, chloroform, batch, extraction

I. INTRODUCTION

Molecularly imprinted polymer (MIP) has been developed in many ways. It can be used as absorbent as in this research for separating and pre-concentrating, it also can be used to analyze a target substance used as template or as removal an unwanted target substance from any environment [1]

In this research, chloramphenicol as a target substance, also used as a template and be reacted with a certain monomer (methacrylic acid / MAA) and crosslinker (ethylene glycol dimethacrylate / EGDMA) using chloroform as porogen.

The chloramphenicol should be extracted from the polymer to form cavities in the polymer which is selective only for chloramphenicol. The higher the percent of extraction, the more cavities will be appeared and it will affect the adsorption capacity of the MIP toward the chloramphenicol in analit. According to Lorenzo, et al. the method used in the extraction process will affect the formation of cavities on the polymer and also the quality of the MIP itself [2]

II. MATTER

A. Molecularly Imprinted Polymer (MIP)

Molecularly Imprinted Polymer (MIP) is a specific polymer which can be used as separating, selecting, concentrating, or removing a target substance used as its template. The concept of MIP is based on lock and key concept as described in Figure 2.1. A: a key mixed with its building block; B: the building block and the key interact each other; C: the complex within the building block and rearrangement to set the position of the building block around the key; D: the key release from the building block, left cavities that suitable only for the original key

Fig. 1. The Concept of lock and key [3]

B. Synthesized of the polymer and the formation of MIP

There are many methods for synthesizing the MIP. The most common used was bulking and precipitation method. In this research bulking method was used as it need less solvent than the other method. [4] Chloramphenicol as template was reacted with metacrylic acid in various ratios, i.e: 1:3, 1:4, and 1:5 in chloroform as porogen to form non imprinted polymer (NIP). The chloramphenicol was then be extracted using acetic acid 15% in methanol to form the molecularly imprinted polymer (MIP) [5]
C. Method of extraction

In this research, two methods of extraction were used. The traditional or soxhlet extraction method and the batch method.

III. METHODS

A. Chemical and instrument

Chemicals:
Chloramphenicol (Sigma Aldrich), methacrylic acid (MAA) (Sigma Aldrich), ethylene glycol dimethacrylate (EGDMA) (Sigma Aldrich), chloroform (Merck), methanol for chromatography (Merck), acetic acid (Merck), Nitrogen gas (HP), benzoil peroxide (BPO) (Merck), aquabidest

Instrument:
High performance liquid chromatography (HPLC)

B. Method

- Chloramphenicol (CAP) was mixed with MAA in ratio (1:3, 1:4, and 1:5) in 30 mL of chloroform then left for 10 minutes.
- EGDMA as crosslinker was added so that the amount of EGDMA was 5x the amount of MAA in mole
- The mixture was stirred carefully then the nitrogen gas was added for 10 minutes to remove the oxygen. After that the initiator BPO was added and the vessel was directly closed tightly
- The mixture was heated for one hour until the first suspension was formed. The result was cooled and the precipitated was washed with the porogen, filtered, and dried (powder). This is the NIP (non-imprinted polymer) product.
- The NIP then was weighed 0.05 gram then extracted with 15% acetic acid in 100% of methanol for 5 hours.
- The extraction result was filtered and washed three times until no chloramphenicol was detected by HPLC or until the amount of chloramphenicol on the filtrate remains the same.
- The percent of extraction (leaching process) was calculated using formulas:

\[
\text{Percent of extraction} = \frac{\text{Total mass of CAP extracted}}{\text{Mass of CAP trapped}} \times \frac{\text{Mass of polymer}}{\text{Mass of adsorbent}} \times 100\%
\]

IV. RESULT AND DISCUSSION

Table 1 shows the calculation result of the extraction percentage of both methods for each ratio of template: monomer.

| Ratio of T / M | Extraction (%) |
|---------------|----------------|
| P 1:3 (B)     | 70.03          |
| P 1:4(B)      | 65.93          |
| P 1:5 (B)     | 48.65          |
| P 1:3 (S)     | 20.50          |
| P 1:4 (S)     | 29.54          |
| P 1:5 (S)     | 25.31          |

Table II. Shows the bar diagram of Table I

Fig. 2. The batch extraction method comparing with traditionally method

The result shows that batch method gives better extraction than the traditional method. It is because in batch method the sample and the extractor can be touched directly than in the other method. It is also shown that the ratio of chloramphenicol to MAA 1:3 gives the best result but it needs further research.

V. CONCLUSION

The leaching process of chloramphenicol using batch method gives better result in average compare to the traditional method (Soxhlet).

The best result was reach by chloramphenicol: MAA = 1:3

ACKNOWLEDGEMENT

Mr. Kusairi and Mrs Aniek Setya Budiatin who helped analyzing the sample and gave permission to conduct this research

REFERENCES

[1] B. Collette, "Global Aquaculture Advocate." USFDA. Industry Efforts Reduce Use of Unapproved Drugs., pp. 38-39, 2006.
[2] Lorenzo, R.A, Carro, A.M, Lorenzo, C.A, "To Remove or Not to Remove? The Challenge of Extracting the Template to Make the Cavities Available in Molecularly Imprinted Polymers (MIPs)," International Journal of Molecular Sciences, vol. 4327, p. 12, 2011.
[3] Yan, M. and Ramstrong, O., Molecularly Imprinted Material Science and Technology. :, New York: Marcel Dekker, 2005.
[4] R. Chrisnandari, "Sintesis dan Karakterisasi Molecularly Imprinted Polymer untuk Kloramfenikol Menggunakan Polimerisasi Fasa Ruah.," Journal of Pharmacy and Science, vol. 3, no. 1, pp. 40-46, 2018.
[5] Omidi, F., Behbahani, M., Abandansari, H.S., Sedighi, A., and Jamaledin, Shahatheri, S.J., "Application of Molecular Imprinted Polymer Nanoparticles as A Selective Solid Phase Extraction for Preconcentration and Trace Determination of 2,4-Dichlorophenoxyacetic Acid in the Human Urine and Different Water Samples," Journal of Environmental Health Science & Engineering, vol. 12, p. 137, 2014.