P-ISSN : 2598-1277
E-ISSN : 2621-3966

JOURNAL OF GREEN SCIENCE AND TECHNOLOGY

EFFECTIVENESS OF ACTIVATED CARBON OF REED (*IMPERATA CYLINDRICA)* AS METHYL ORANGE BIOADSORBENT WITH BATCH ADSORPTION METHOD

Dian Ratna Suminar[1], Dicky Harianto[1], Najmi Fauziah[1], Dea Tri Febrian[1], Luthfiana Kharisma[1], Tifa Paramitha[1]*

[1]) Chemical Engineering Department, Politeknik Negeri Bandung, Bandung*
Corresponding Author's Email: tifa.paramitha@polban.ac.id

ABSTRACT

The use of synthetic textile dyes causes environmental problems, namely the waste produced is still colored and difficult to degrade. Methyl orange is a synthetic dye in the textile industry which has low solubility in water, is difficult to degrade, has the potential to be carcinogenic and is toxic. To overcome this, adsorption can be carried out using activated carbon from reeds. This research aims to find out how active carbon of reeds is absorbed in methyl orange using the batch method with Langmuir isotherm analysis. Research methods include pretreatment of reeds, manufacture and activation of reeds carbon, testing the characteristics of activated carbon, adsorption process, and analysis of adsorption results using a UV-Vis spectrophotometer. The research was carried out by varying the concentration of methyl orange solution, adsorption time, and mass of activated carbon used. From the research, optimum conditions were obtained for the methyl orange adsorption process using reed activated carbon for 120 minutes with an activated carbon mass of 0.3 grams, a final concentration of 7.328 ppm was obtained with an initial concentration of 10 ppm, and a % adsorption of 26.72%.

Keyword: *Activated carbon, Bioadsorbent, Methyl orange, Reeds.*

1. INTRODUCTION

The use of synthetic textile dyes poses problems, namely the waste generated is still colored and difficult to degrade because about 10% to 15% of the dye used cannot be reused and must be discarded [1]. Methyl orange is a synthetic dye used in fabric dyeing that has low solubility in water [2]. Methyl orange is also an organic pollutant that has carcinogenic, toxic, and mutagenic properties for life [3]. In addition, methyl orange is an indicator of pH and is widely used in acid-base titration because it changes color when pH changes. Methyl orange has the molecular formula $C_{14}H_{14}N_3NaO_3S$ made from sulfanilic acid and N,N-dimethylaniline.

To address the pollution from synthetic waste, adsorption methods can be employed. Adsorption is a phenomenon where materials from components of a fluid, be it liquid or gas, are captured in the interphase area where the substance to be separated (adsorbate) is bound to the surface of a solid material (adsorbent) [4]. The advantage of this method is its high efficiency; moreover, the absorbent can be heated for regeneration. Adsorbents are generally made from materials that have pores, one of which is activated carbon.

Activated carbon is a material in which there are many very small pores. This is because activated carbon has many very small pores, which causes the activated carbon to have a large surface area and high adsorption power so that its utilization can be optimal [5]. The abundant pores on activated carbon are thanks to the activation process. The activation process can be carried out in 2 ways, namely heating with high temperatures and adding chemicals [4].

Reeds is a plant of the Gramineae family. This plant has high adaptability, so it easily grows everywhere and often becomes a weed that is detrimental to farmers. Reeds can reproduce vegetatively and generatively or grow on various soil types [6]. In addition to its detrimental properties, it turns out that reeds can be used as adsorbents because because it has a fairly high cellulose content, including α-cellulose of 40.22%, holocellulose of 59.62%, and lignin of 31.29% [7]. The adsorption process occurs

P-ISSN : 2598-1277
E-ISSN : 2621-3966

due to the interaction between dyes and functional groups of these polymers (such as –OH and –COOH) [8].

Several studies related to the utilization of reeds as a bioadsorbent using batch methods have been conducted, utilizing activated carbon from reeds to adsorb methylene blue, achieving an adsorption capacity of 86.61% [9]. The utilization of cellulose from reeds to adsorb methylene blue achieved an adsorption capacity of 52.89% [10]. Additionally, the utilization of reeds ash to adsorb methylene blue achieved an adsorption capacity of 96.88% [11]. In another study, it was noted that the adsorption capacity of heavy metal Cd (cadmium) reached 65.70% [12]. Meanwhile, in tests with Pb (lead), the adsorption capacity obtained was 96.85% [13]. Through this research, the researcher innovates on the potential of reeds as a source for making activated carbon that can adsorb methyl orange using the batch adsorption method with Langmuir isotherm analysis.

2. RESEARCH METHODOLOGY

2.1 Place and time of Implementation

The research was conducted at the Corrosion Laboratory, Department of Chemical Engineering, Bandung State Polytechnic for four months.

2.2 Tools and Materials

The tools used for the research are furnaces, choppers, porcelain, shieve shakers, analytical balances, buchner funnels, pH papers, desiccant, filter paper, and glassware (chemical beakers, volume pipettes, measuring flasks, measuring cups, and erlenmeyer). The materials needed for this research are reeds, methyl orange, aquadest, and H_2SO_4.

2.3 Pretreatment for the reeds

Pretreatment of reeds, carried out by washing and reducing the size of the reeds, after which they are dried in the oven at 100˚C for 3 hours, after drying they are sifted using a sieve shaker with a size of 40-80 mesh.

2.4 Reeds Activated Carbon Manufacturing

The production of reeds activated carbon is carried out by carbonizing dry reeds in a furnace at 450˚C for 25 minutes, then cooled. Furthermore, the carbonization result was activated by soaking 21 grams of activated carbon in reeds with an activator solution of 180 mL H_2SO_4 2M for 24 hours, the filtered. The activated carbon is then washed with aquadest until the pH of the activated carbon is equal to the pH of the aquadest and dried in the oven at 100˚C for 4 hours. The yield obtained can be calculated by the formula:

$$\%\text{Yield} = \frac{M1}{M2} \times 100$$

Where:

M1 = mass of carbon (g)
M2 = mass of dried reeds

2.5 Bioadsorbent Characteristic Test

Carbon is carried out Characteristic analysis, namely moisture content test, ash content test, volatile matter, and fixed carbon with reference SNI 06-3730-1995. In addition, SEM and FTIR test were also carried out.

2.6 Moisture content

A total of ± 1 gram of activated carbon in a porcelain cup is evaporated using an oven at 115˚C for 3 hours and weighed until a constant weight is obtained.

$$\%\text{Moisture} = \frac{M1}{M2} \times 100$$

Where:

M1 = the lost weight
M2 = mass of sample

2.7 Ash content

A total of ± 2 gram of activated carbon in a porcelain cup are reasted using a furnace at 800-900˚C for 2 hours and weighed until a constant weight is obtained.

$$\%\text{Ash} = \frac{M1}{M2} \times 100$$

Where:

P-ISSN : 2598-1277
E-ISSN : 2621-3966

M1 = mass after the furnace
M2 = mass of sample

2.8 Volatile matter

A total ± 1 gram of activated carbon in the porcelain cup is resealed using a porcelain cup so that the sample is between the cups. Heat in the oven until it reaches a temperature of 950˚C. When it reaches that temperature, cool and weigh it periodically until a constant is obtained.

$$\text{Volatile matter} = \frac{M1}{M2} \times 100$$

Where:
M1 = mass after the oven
M2 = mass of sample

2.9 Fixed carbon

It is determined by a 100% reduction by the total results of the calculation of ash and volatile metter levels.

$$\%\text{fixed carbon} = 100-(A+B)$$

Where:
A = Volatile metter calculation result (%)
B = Ash content calculation result (%)

2.10 SEM

A total ± 1 gram of carbonization and activation results was sent to a 3rd party for furthe analysis.

2.11 FTIR

A total ± 1 gram of carbonization, activation, and adsorption results were sent to a 3rd party for furthe analysis.

2.12 Batch Method Adsorption Test

Adsorption tests are carried out on a laboratory scale with a batch operating system. The test was carried out by mixing a few reeds activated carbon bioadsorbents into a reactor containing a methyl orange solution of 25 mL. Reactions were performed under constant stirring and space conditions with variations in the amount of activated carbon bioadsorbent doses, contact time, and methyl orange concentrations. Then the solution is filtered and the filtrate is measured methyl orange not absorbed using a UV-Vis spectrophotometer. The percentage of adsorbate removal can be calculated using equation (1).

$$\text{Percentage of adsorbate removal } (\%) = \frac{Co - Ct}{Ct} \times 100 \quad \ldots\ldots\ldots\ldots\ldots\ldots (1)$$

Where Co (mg/L) and Ct (mg/L) are the initial concentration and concentration at a given time respectively [9].

2.13 Solution Analysis of Adsorption Test Results with UV-Vis Spectrophotometry

Quantitative sample analysis to determine the methyl orange concentration was carried out on the initial solution (at t=0 minutes) and the adsorption test results with variations in pH, number of bioadsorbents, contact time, and variations in methyl orange concentration. Determination of concentration using the help of UV-Vis spectrophotometer at wavelengths of 400-800 nm.

2.14 Langmuir Isotherm Adsorption

Langmuir adsorption isotherm describes the equilibrium and kinetic relationship between the absorbed particles and their absorption and is also a description of the equilibrium state between the amount of adsorbents at a fixed temperature and the concentration of solutes trapped on the surface of the solids [14]. The adsorption process in the Langmuir isotherm method is based on several main principles, namely:

1. Adsorption occurs in a single layer (monolayer) on a homogeneous surface.
2. Constant adsorption energy for all sites on the surface.
3. No interaction between adsorption molecules.

Adsorption in the Langmuir isotherm method can be calculated using equation (2) as follows.

$$\frac{Ce}{qe} = \frac{1}{qm.KL} + \frac{Ce}{qm} \quad \ldots\ldots\ldots\ldots\ldots\ldots (2)$$

P-ISSN : 2598-1277
E-ISSN : 2621-3966

The correlation of Ce/qe and Ce results in a linear relationship where qm is the maximum adsorption capacity (L/mg), KL is the Langmuir adsorption capacity (L/mg) [15].

3. ANALYSIS AND RESULT

3.1 Reed Pretreatment

Reed pretreatment begins with washing the reed to be free of impurities. Followed by reducing the size of the reeds to make the ovening process easier. Then the reeds are mashed using a chopper and then sifted using a sieve shaker to obtain a uniform size, which is 60-30 mesh.

3.2 Carbonization and Activation of Reeds

The fine reeds are furnaced at 450^{o}C for 25 minutes to obtain carbon. Furthermore, carbon is activated with the aim of enlarging the pores on the carbon surface so that the contact surface area is also larger. Carbon washing after activation aims to bring the pH of activated carbon back to neutral, while ovening aims to reduce the water content of activated carbon. At this stage, a %yield of 38.35% was obtained.

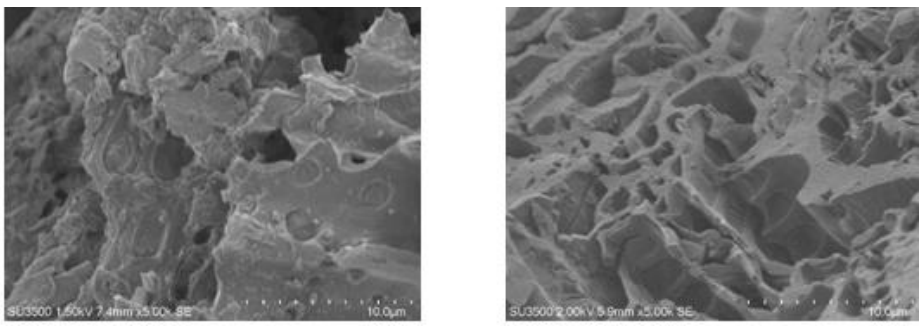
3.3 Reed Activated Carbon Characteristics Test

Table 2. Analysis Results with SNI Parameters 06-3730-1995

Analysis	SNI 06-3730-1995	Result
Moisture content	Max 10%	6.30%
Ash content	Max 15%	6.18%
Volatile matter	Max 25%	50.24%
Fixed carbon	Min 65%	43.46%

Based on the data from the table above, the results of volatile matter and fixed carbon were obtained that were not in accordance with the provisions of SNI 06-3730-1995. This volatile matter yield that is too high is caused by the imperfection of the decomposition of non-carbon compounds during the carbonization process, resulting in small levels of fixed carbon. This can be one of the factors for the lack of effectiveness of reeds.

3.3.1 SEM test results



(A) **(B)**

Figure 2. (A) SEM After Carbonization and (B) SEM After Activation

Table 3. Elemental Levels in Reeds Before and After Activation

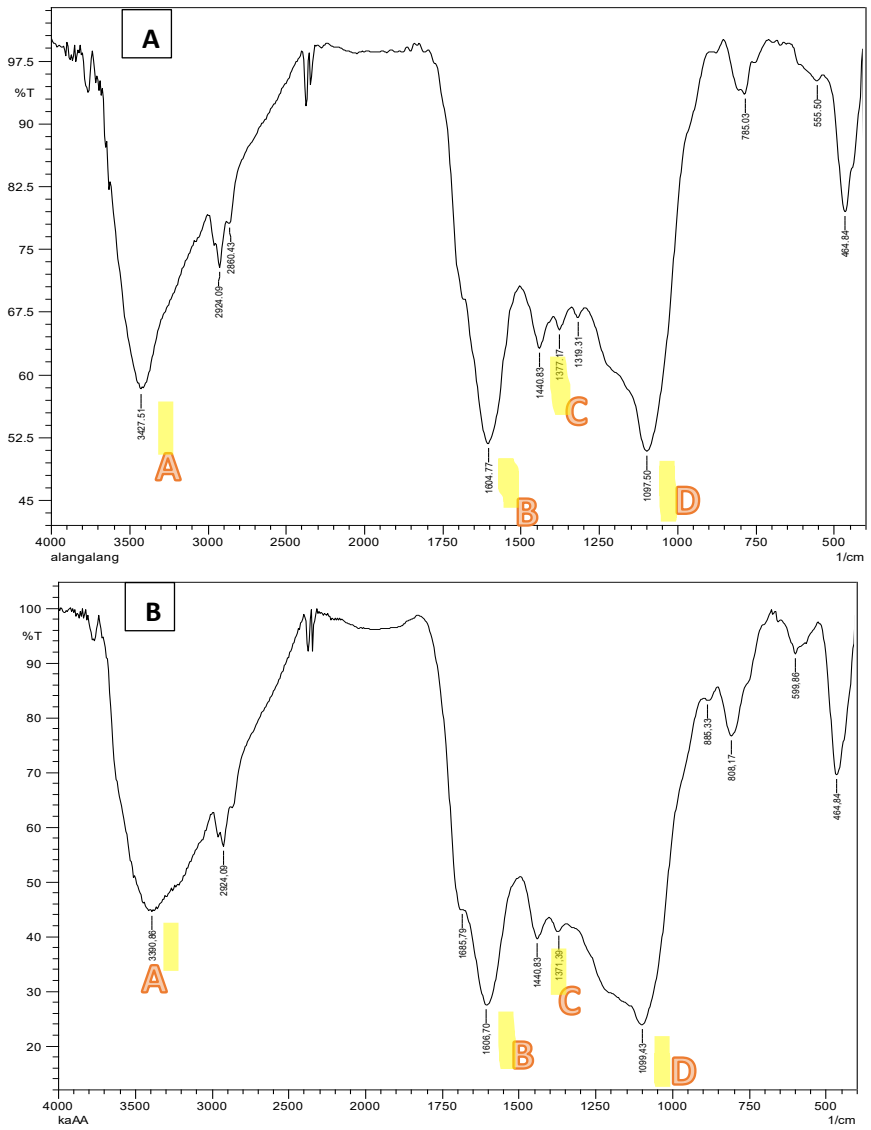
Treatment	*Weight* (%) *Element*							Porosity
	C	O	Al	Si	Au	K	Ca	
Carbonization	65,51	25,18	-	4,95	1,67	1,50	1,20	0,5867447
Activation	59,81	28,87	0,29	11,03	-	-	-	0,571847884

The porosity value in the carbonization treatment was 0.58 and for the activation treatment showed a value of 0.57 with the largest composition in the carbonization treatment being carbon of 65.51% while the activation was 59.81%. This proves that the results of carbon activation are perfect. In the oxygen composition of 25.18% and 28.87%, respectively, the oxygen content (O) contained in activated carbon is caused by the activator H_2SO_4, the high oxygen content and the acidic activator reacts with the oxygen-containing functional group, for the silica composition of 4.95% and 11.03%, respectively, the

P-ISSN : 2598-1277
E-ISSN : 2621-3966

presence of aluminum content in activated carbon, one of which is due to the content of hard water during the process of washing the reed material that participates binded, the content of Al is considered the cause of hardness in water.

3.3.2 FTIR test results



P-ISSN : 2598-1277
E-ISSN : 2621-3966

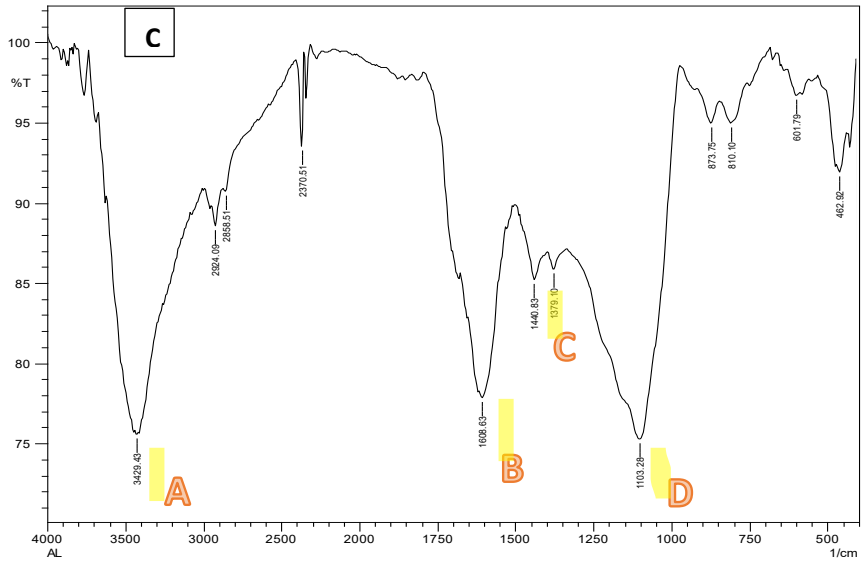


Figure 3. (a) Results of Reeds Carbon FTIR, (B) Reeds Activated Carbon FTIR Results, and (c) Reeds Activated Carbon FTIR Results after Adsorption

The spectrum shows the presence of various functional groups commonly found in carbon, such as hydroxyl, alcohol, aliphatic, and aromatic groups. In the reed carbon, it also shows the presence of cellulose which functions to bind dye substances, namely carbonyl groups C-O and hydroxyl O-H. After the activation of the reed carbon, the FTIR shows an increase in intensity and a change in the position of the peak of the spectrum, signaling the formation or modification of functional groups and changes in chemical structure. In post-adsorption activated carbon samples, the transmittance value changes, indicating adsorbate molecular binding that alters the function group, with the new functional group detected being N-H.

Table 4. Carbon FTIR Results Before and After Activation

Top	Reed Before Activation ($^{cm-1}$)	Reed After Activation ($^{cm-1}$)	Wavelengh ($^{cm-1}$)	Gugus Fungsi
A	3427.51	3390.86	3200-3600	O-H
B	1604.77	1606.70	1500-1600	C=C
C	1440.83	1440.83	1650-1750	C-H
D	1097.50	1099.43	1124-1087	C-O

Table 5. Results of FTIR of Activated Carbon After Adsorption

Top	Reed After Adsorption ($^{cm-1}$)	Wavelength ($^{cm-1}$)	Gugus Fungsi
A	3429,43	3400-3300	N-H
B	1608,63	1500-1600	C=C
C	1440,83	1650-1750	C-H
D	1103,28	1150-1085	C-O

3.4 Adsorption Test of Batch Method and Solution Analysis of Adsorption Test Results with UV-Vis Spectrophotometry

P-ISSN : 2598-1277
E-ISSN : 2621-3966

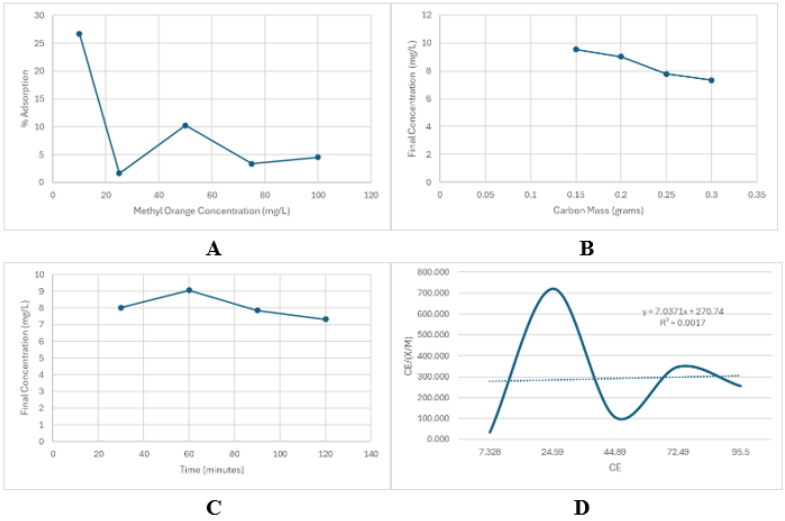


Figure 4. (a) Graph of % Adsorption to Methyl Orange Concentration, (B) Graph of Final Methyl Orange Concentration to Carbon Mass, (C) Graph of Final Concentration of Methyl Orange to Time, (D) Graph of Langmuir Capacity

Based on the graph, the optimal condition was obtained that the initial concentration of methyl orange was at 10 mg/L with an Adsorption % of 26.72%, with an optimal mass of 0.3 grams and an optimal time of 120 minutes with a final concentration of 7,328 mg/L. Low initial concentration can increase adsorption capacity because it lowers concentration faster, this is proportional to the longer contact time with the number of active sites available or the amount of adsorbent mass used. However, based on the Langmuir capacity graph, a very small regression value of 0.0017 was obtained so that the capacity value could not be determined because the value showed inlinearity or low accuracy results, this can be further reviewed from the results of the adsorbent characteristics. The effectiveness value of reed activated carbon is 26.72% which is very small when compared to the effectiveness of reed carbon against methylene blue. This is because methyl orange is a dye that is difficult to degrade and methyl orange is also non-biodegradable. In addition, the difference in properties of methylene blue which is cationic while methyl orange is anioic is also one of the causes of the difference in effectiveness obtained. And judging from the results of the characteristics of volatile matter and fixed carbon levels in carbon that are not in accordance with the parameters of SNI 06-3730-1995, resulting in carbon purity that is below the parameters, and small porosity values after activation result in limited space for the adsorption process.

4. CONCLUCION

The characteristics of activated carbon include moisture content and ash content according to SNI 06-3730-1995 standards. The highest content in the SEM test result was carbon, oxygen, and silica with a porosity value of carbonization treatment of 0.58 and activation treatment of 0.57. The results of the FTIR test on carbonized and activated carbon also show the presence of cellulose which functions to bind dye substances, namely carbonyl groups C-O and hydroxyl O-H, for the results of post-adsorption activated carbon samples, there is a content of a new functional group detected is N-H. The optimum condition of the initial methyl orange concentration was at 10 mg/L with a % adsorption of 26.72%, with an optimum mass of 0.3 grams and an optimal time of 120 minutes with a final concentration of 7.328 mg/L. A review of the capacity using the Langmuir method obtained a very small regression value of 0.0017.

REFERENCE

[1] S. Naimah, S. A. A., B. N. Jati, N. N. Aidha dan A. A. C., "Degradasi Zat Warna pada Limbah Cair Industri Tekstil dengan Metode Fotokatalitik Menggunakan Nanokomposit TiO2-Zeolit," *Jurnal Kimia dan Kemasan,* vol. 36, no. 2, pp. 225-236, 2014.

P-ISSN : 2598-1277
E-ISSN : 2621-3966

[2] A. E. Fitriani, "Penurunan Konsentrasi Methyl Orange dengan Variasi Dosis Koagulan Ekstrak NaCl-Biji Asam Jawa serta pH Larutan dan Konsentrasi Methyl Orange," 2016.

[3] T. Nurlaili, L. Kurniasari dan R. D. Ratnani, "Pemanfaatan Limbah Cangkang Telur Ayam sebagai Adsorben Zat Warna Methyl Orange dalam Larutan," *Jurnal Inovasi Teknik Kimia,* vol. 2, no. 2, pp. 11-14, 2017.

[4] L. Ifa, Nurdjannah, T. Syarif dan Darnengsih, "Bioadsorben dan Aplikasinya," Yayasan Pendidikan Cendikia Muslim, Solok, 2021.

[5] F. D. Permana, "Adsorpsi Senyawa Polisiklik Aromatik Hidrokarbon Fenantrena dengan Menggunakan Adsorben Karbon Aktif dari Sekam Padi (Oryza sativa)," 2017.

[6] T. T. Astuti, "Pemanfaatan Akar Alang-Alang (Imperata Cylindrica) sebagai Adsorben untuk Menurunkan Kadar Magnesium dalam Air Sadah dan Aplikasinya pada Materi Kimia Unsur di Sekolah Menengah Kejuruan Negeri 2 Pekanbaru," 2017.

[7] S. A. Yudistirani, A. M. Sari dan H. Rizawaldi, "Penentuan pH Optimum Adsorbat dalam Pemanfaatan Tanaman Alang-Alang (Imperata cylindrica) sebagai Bioabsorben Logam Berat Cd (II) pada Limbah Industri Cat," *Jurnal Konversi Universitas Muhammadiyah jakarta,* vol. 11, no. 1, pp. 23-30, 2022.

[8] B. Rahadi, A. T. S. H. dan I. Robbaniyah, "Analisis Penurunan Konsentrasi Methyl Orange dengan Biosorben Kulit Pisang Cavendish (Musa Acuminata CV. Cavendish)," *Jurnal Sumberdaya Alam dan Lingkungan,* vol. 6, no. 2, pp. 29-35, 2019.

[9] N. F. M. Salleh, F. F. Asmori, N. M. Shukri dan S. F. M. Hanafiah, "Adsorption of Methylene Blue by Imperata Cylindrica: Reaction Optimization by Response Surface Methodology (RSM)," *Journal of Chemical Engineering and Industrial Biotechnology,* vol. 07, no. 02, pp. 27-33, 2021.

[10] T. Huda dan T. K. Yulitaningtyas, "Kajian Adsorpsi Methylene Blue Menggunakan Selulosa dari Alang-Alang," *Indonesian Journal of Chemical Analysis,* vol. 01, no. 01, pp. 09-19, 2018.

[11] I. Riwayati, N. Fikriyyah dan Suwardiyono, "Adsorpsi Zat Warna Methylene Blue Menggunakan Abu Alang-Alang (Imperata Cylindrica) Teraktivasi Asam Sulfat," *Inovasi Teknik Kimia,* vol. 4, no. 2, pp. 6-11, 2019.

[12] A. Khilya dan A. T. Prasetya, "Optimasi dan Aplikasi Arang Aktif Alang-Alang untuk Menurunkan Kadar Cd (II) dalam Larutan," *Indonesian Journal of Chemical Science,* vol. 5, no. 1, pp. 6-10, 2016.

[13] N. A. Miningsih, D. I. Hidayatin, A. Wijareni, S. I. Isdiyanti dan L. Kurniasari, "Adsorpsi Timbal (Pb) dalam Larutan Menggunakan Adsorben Radix Alang-Alang (Imperata Cylindrica) Termodifikasi Asam Sulfat," *Prosiding Seminar Nasional Sains dan Teknologi,* vol. 1, no. 1, pp. 23-27, 2016.

[14] D. Amanda, F. Arfi dan K. Nisah, "Uji Persamaan Langmuir dan Freundlich pada Penyerapan Ion Logam Kobalt (II) oleh Kitosan dari Kulit Udang Windu (Panaeus Monodon)," *Ar-Raniry Chemistry Journal,* vol. 3, no. 3, pp. 123-135, 2021.

[15] A. Husin dan P. D. R. Sitinjak, "Studi Adsorpsi Furfural Menggunakan Karbon Aktif dari Kulit Durian Termodifikasi," *Jurnal Teknik Kimia USU,* vol. 10, no. 1, pp. 19-24, 2021.