

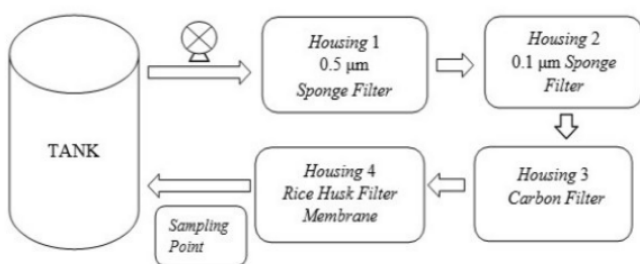
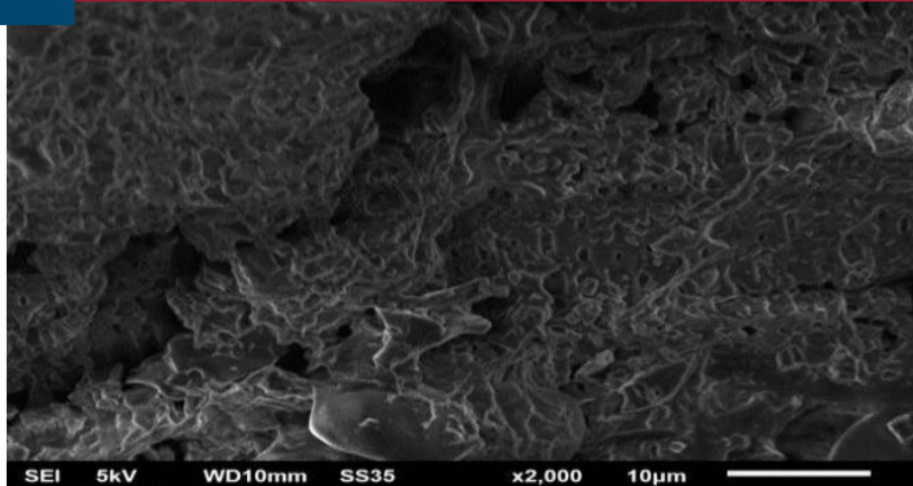


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Membrane Filter
From Rice Husk



Filtration System

Time (Minute)	1.5 Bar		2 Bar	
	With Rice Husk Additive (ppb)	Without Rice Husk Additives (ppb)	With Rice Husk Additive (ppb)	Without Rice Husk Additives (ppb)
0	1250	1250	1250	1250
15	1120	1241	1100	1220
30	1050	1233	950	1210
45	980	1190	895	1130
60	920	1150	800	1000
75	900	1170	760	993
90	890	1123	710	954

Silica (SiO_2) Content
Decreased to 710 ppb

Reduced Silica (SiO_2)
Content 43.2%

Reducing Silica Levels using Rice Husk Filter Membranes (Setiorini, et al)

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Evaluation of Heat Loss Effect on Package Boiler Performance (5007-U) in the Utility Unit of Urea Fertilizer Industry

*Evaluasi Pengaruh Kehilangan Panas Terhadap Performa Package Boiler (5007-U)
Pada Unit Utilitas Industri Pupuk Urea*

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Abstract

Package boiler (PB) is a utility unit essential for producing steam by heating Boiler Feed Water (BFW) through the combustion of natural gas with air. Therefore, this study aimed to investigate the effect of heat loss on PB efficiency. To achieve this, direct and indirect methods were adopted, with data collected between 4 July and 29 August 2023. The results showed that PB efficiency in the fertilizer industry during this period ranged from 76.06% to 80.71%. On August 29, 2023, under optimal conditions, an efficiency of 80.71% was achieved, while a significant drop to 65.44% occurred during the 6th week on August 8, 2023, due to low oxygen (O₂) levels. Flue gas analysis on August 29, 2023, obtained 3.64% excess O₂, 11.17% carbon dioxide (CO₂), and 0.04% carbon monoxide (CO). PB performance was influenced by heat loss from the dry flue gas, hydrogen (H₂) content on flue gas, moisture in air and fuel, incomplete combustion, as well as radiation and convection phenomena.

Keywords: excess O₂; flue gas; heat loss; natural gas flow; package boiler

Abstrak

Package boiler merupakan salah satu unit utilitas yang berperan dalam menghasilkan steam dengan memanaskan boiler feed water dari panas hasil pembakaran natural gas dengan udara. Tujuan dari penelitian ini adalah untuk menyelidiki kehilangan panas pada package boiler, yang cenderung dapat menurunkan efisiensinya. Penelitian ini menggunakan dua metode untuk mengevaluasi efisiensi package boiler, yaitu metode langsung dan tidak langsung yang datanya dikumpulkan dari minggu 1- 9 (4 Juli – 29 Agustus 2023). Berdasarkan data yang didapatkan dari bulan Juli hingga Agustus, efisiensi package boiler di industri pupuk berada dalam rentang 76,06% hingga 80,71%. Package boiler industri pupuk urea mengalami kondisi optimal di pekan ke 9 pada tanggal 29 Agustus 2023 dengan efisiensi sebesar 80,71% dan penurunan efisiensi secara drastis di pekan ke 6 pada tanggal 08 Agustus 2023 dengan efisiensi sebesar 65,44%. Hasil analisa flue gas pada tanggal 29 Agustus 2023 didapatkan O₂ excess 3,64%, CO₂ content 11,17%, dan CO content 0,04%. Kinerja package boiler dipengaruhi oleh panas yang hilang karena flue gas kering, kadar H₂, kelembaban di udara dan bahan bakar, pembakaran tidak sempurna, radiasi dan konveksi.

Kata kunci: excess O₂; flue gas; heat loss; natural gas flow; package boiler

Evaluation of Heat Loss Effect on Package Boiler Performance (5007-U) in the Utility Unit of Urea Fertilizer Industry

1. Introduction

The utility unit is responsible for producing steam, which serves as a driving force for turbines and pumps. Meanwhile, an ammonia plant operates its steam generator for production purposes. The utility unit produces steam using Waste Heat Boiler (WHB) and Package Boiler (PB) [1]. WHB utilizes exhaust gases from the combustion of natural gas and air from the Gas Turbine Generator (GTG), while PB uses heat from the combustion of natural gas with air, and is designed with excellent heat transfer capabilities through radiation, convection, and high conduction.

PB is a key component of the utility unit and the primary steam supply unit in the Urea Fertilizer Industry (UFI). It functions by raising the water temperature until reaching the steam point through direct contact with the flame generated by natural gas combustion. PB is fed with Boiler Feed Water (BFW) [2] and heat transfer occurs from the combustion of natural gas to water, resulting in the generation of steam used in the production process. However, its operation leads to heat loss due to various factors, including incomplete combustion [3]. Significant heat loss can result in reduced efficiency, thereby impacting performance [4].

Boiler efficiency represents the performance that is obtained by comparing the energy produced with the chemical energy from the fuel. The systems and components used in PB at the UFI utility unit have been operational for an extended period, necessitating an efficiency analysis. This analysis ensures that the steam produced reaches the appropriate temperature for use in the production process, requiring calculations of overall component flow rates [5] and heat absorbed in PB [6]. The efficiency of this

boiler is below 84%, signifying the need for further evaluation. This study aims to investigate the effect of heat loss on PB efficiency.

2. Research Methods

Direct and indirect methods were adopted to evaluate PB performance (5007-U) in the utility unit of UFI. The evaluation of the direct method utilized actual data of heat input and output, calculated using equation 1.

$$\text{Efficiency} = Q_{\text{input}} / Q_{\text{output}} \dots\dots\dots(1)$$

$$\text{Efficiency} = 100\% - (L1 + L2 + L3 + L4 + L5 + L6) \dots\dots\dots(2)$$

$$L1 = \frac{m \times C_p \times (T_f - T_a)}{\text{GCV Fuel}} \times 100\% \dots\dots(3)$$

$$L2 = \frac{9 \times H_2 \times [584 + C_p (T_f - T_a)]}{\text{GCV Fuel}} \times 100\% \dots\dots(4)$$

$$L3 = \frac{M \times [584 + C_p (T_f - T_a)]}{\text{GCV Fuel}} \times 100\% \dots\dots(5)$$

$$L4 = \frac{\text{AAS} \times \text{humidity factor} \times C_p \times (T_f - T_a)}{\text{GCV Fuel}} \times 100\% \dots\dots(6)$$

$$L5 = \frac{\%CO \times C}{\%CO + \%CO_2} \times \frac{5744}{\text{GCV Fuel}} \times 100\% \dots\dots(7)$$

$$L6 = 0,548 \times [(T_s / 55,55)^4 - (T_a / 55,55)^4] + 1,957 \times (T_s - T_a)^{1,25} \times \text{sq.rt of } [(196,85 \times V_m + 68,9) / (68,9)] \dots\dots(8)$$

Descriptions:

Q_{input} = heat input (kJ)

Q_{output} = heat output (kJ)

L1: Heat loss due to dry flue gas (%)

L2: Heat loss due to hydrogen in fuel (%)

L3: Heat loss due to moisture in fuel (%)

L4: Heat loss due to moisture in air (%)

L5: Heat loss due to carbon monoxide (%)

L6: Heat loss due to surface radiation and convection

m: Mass flow (kg/h)

C_p : Specific heat capacity (J/kg.K)

T_f : Final Temperature (°C)

T_a : Ambient Temperature (°C)

T_s : Entropy Temperature (°C)

GCV: Gross calorific value (KJ/kg)

AAS: Actual air supply (kg)

Evaluation of Heat Loss Effect on Package Boiler Performance (5007-U) in the Utility Unit of Urea Fertilizer Industry

The indirect method used actual data, including sample quantities of independent variables such as flue gas components, flue gas temperature, natural gas components, steam flow, steam temperature, steam pressure, BFW pressure, BFW temperature, and airflow in PB at the utility unit. The evaluation comprised data collection, processing, boiler efficiency calculation, and analysis of the effect of heat loss on boiler performances [7]. PB efficiency was calculated using equation 2, while the effect of heat loss was analyzed by calculating losses from flue gas, hydrogen (H₂) content, moisture content on fuel, moisture on air, incomplete combustion, as well as radiation and convection using equations 3-8. The required data for this specific task, including actual input and output values of PB in UFI, were collected from the Utility Unit every Tuesday from 4 July to 29 August 2023 and symbolized with weeks 1st to 9th.

3. Results and Discussion**3.1 The Effect of Total Heat Loss on PB Efficiency**

The evaluation of boiler efficiency is conducted using an indirect method by calculating heat loss during the process. The direct method showed an average PB

efficiency of 71,33% and did not present potential causes of inefficiency. Therefore, it requires actual calculations with the indirect method to accurately assess factors such as heat loss [8].

The results of the performance efficiency calculation for PB using an indirect method are presented in Figure 1. The average efficiency was in the range of 70-80%, except on August 8, 2023, where a significant decrease was observed. In week 6th, specifically on August 8, 2023, PB efficiency experienced a drastic decrease, reaching 65.44%. This phenomenon occurred due to heat loss caused by a relatively high level of incomplete combustion. In the same week, the flue gas analysis conducted showed that approximately 5.89% of carbon monoxide (CO) gas was contributing significantly to the sharp efficiency decrease. During the last week of July and the middle of August, UFI was shut down, leading to suboptimal operation of PB in the facility and contributing to an efficiency decrease. Various factors can contribute to heat loss, including high flue gas temperatures, hydrogen content in natural gas, moisture content in air as combustion feedstock, incomplete combustion of natural gas, and heat loss due to radiation.

Table 1. Collected data from PB (5007-U)

Data	Unit	Weeks								
		1 st	2 nd	3 rd	4 th	5 th	6 th	7 th	8 th	9 th
Steam flow	ton/h	40.02	40.99	32.79	32.25	37.92	26.88	44.50	46.17	33.50
Steam pressure	kg/cm ³	42.11	43.07	42.68	42.48	42.37	42.90	43.28	43.64	43.17
Steam temperature	°C	409.00	410.42	410.00	409.42	409.33	407.40	408.42	409.50	408.50
BFW pressure	kg/cm ³	57.99	58.29	59.67	58.40	37.92	59.53	58.75	59.09	56.47
BFW temperature	°C	84.74	72.41	77.76	83.92	76.17	86.80	54.83	60.00	97.33
Natural gas flow	m ³ /h	4,445	4,173	3,532	3,463	4,430	3,094	4,947	5,198	3,028
Airflow	kg/h	59,312	56,259	47,278	46,913	58,753	40,541	66,374	81,779	46,131

Evaluation of Heat Loss Effect on Package Boiler Performance (5007-U) in the Utility Unit of Urea Fertilizer Industry

3.2 The Effect of Heat Loss Due to Dry Flue Gas on PB Efficiency

Heat loss due to dry flue gas represent a significant inefficiency, as the gas exiting boiler system still contains heat. This loss can be measured by the flue gas temperature, as recorded in UFI laboratory. The exiting gas carries away heat that should have been utilized for steam production or heating, thereby reducing efficiency.

Figure 2 shows a consistent loss of heat caused by dry flue gas each week. In week 6th, a significant loss was observed compared to other periods, as evidenced by the flue gas temperature, which reached 219°C. The flue gas temperature is directly proportional to heat released by PB. This case presents the effect of heat loss on efficiency reduction. An increase in temperature can be attributed to insufficient heat transfer and fouling caused by deposits from fuel or BFW [9]. However, the flue gas temperature should not be very low to prevent water vapor from condensing on the chimney walls [10].

3.3 The Effect of Heat Loss Due to H₂ Content in Fuel on PB Efficiency

H₂ content in fuel is typically in the form of hydrocarbons or pure H₂ gas and can impact boiler efficiency. A significant factor contributing to decreased efficiency is the evaporation of water due to H₂ content in the fuel. In the case of PB, water evaporation can occur due to the presence of H₂ in the natural gas used. Water is formed from the reaction between hydrogen and oxygen in the air during combustion [11]. It will turn into steam by absorbing heat from combustion, leading to a reduction in heat absorption for

evaporating BFW and reduced efficiency [12].

Heat loss due to H₂ content in the fuel represents the largest, particularly because PB relies on natural gas, predominantly composed of methane. Figure 3 shows that the average heat loss due to H₂ in natural gas was approximately 11.8%. This loss is influenced by H₂ content in the mass component of natural gas fuel, leading to relatively stable heat loss over time. However, an increase was observed on week 6th (August 8, 2023), rising to 12.17%. The increase was traced to an imbalance in the air-fuel ratio, as evidenced by an excess small oxygen (O₂) level of 1.21%. This can lead to incomplete combustion, affecting heat generated [13].

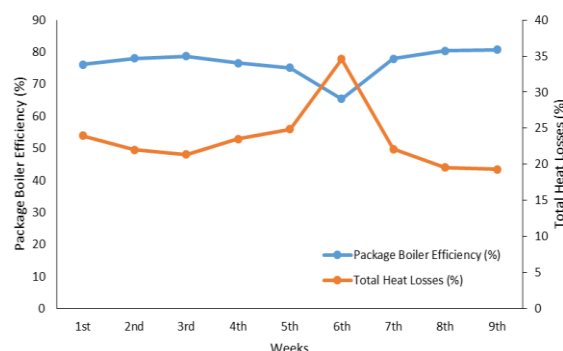


Figure 1. Effect of total heat loss on PB

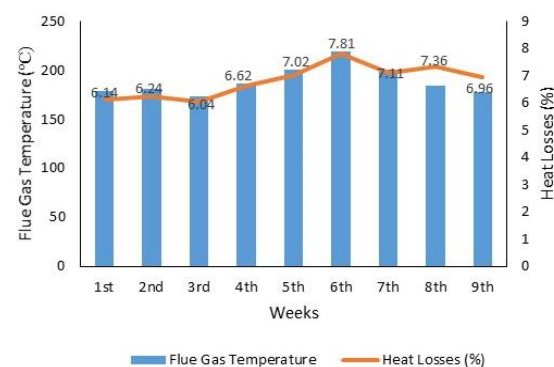


Figure 2. Effect of dry flue gas on heat loss

Evaluation of Heat Loss Effect on Package Boiler Performance (5007-U) in the Utility Unit of Urea Fertilizer Industry

3.4 The Effect of Heat Loss Due to Moisture in Air and Fuel on PB Efficiency

Heat losses can be caused by the presence of moisture in both natural gas feed and air used in the combustion process. Moisture in the natural gas feed and air enter the boiler and leave as steam. Heat needed to warm BFW is consumed by moisture, leading to heat loss and a reduction in PB efficiency. The higher the moisture content in the fuel, the greater the heat loss, due to the reduction in the effective calorific value of the fuel [10]. Heat loss was attributed to sensible heat used to raise moisture to boiling point, latent heat for moisture evaporation, and excess heat to reach the flue gas temperature.

Figure 4 shows heat loss caused by the presence of moisture content in natural gas fuel. The analysis results from July-August present minimal moisture in the natural gas feed, leading to heat loss consistently measured at 0% for each week. However, the combustion air drawn from the ambient environment also contains moisture. During the combustion process, this water vapor did not participate in any reaction but mixed with the smoke gases produced. As a result, some of the heat generated during combustion is absorbed by moisture in the air, which reduces the energy available for evaporating moisture content [12].

Figure 4 shows heat loss caused by moisture content in the combustion air. A constant heat loss was observed until the 1st week of August, averaging approximately 0.32%, while in the 2nd week, it increased to 0.37%. This was attributed to an imbalance in the air and natural gas flow. In the 4th week of August, a significant difference was observed. On

week 8th, exactly on August 22, 2023, the air and natural gas flows were 81,779.9 kg/h and 4,348.74 kg/h, respectively, which led to excess air and increased water vapor entering the boiler. On August 29th, 2023, the air supplied was at an average of 46,131.3 kg/h, but the natural gas flow decreased to 2,510.85 kg/h. Therefore, the excess air and water vapor entering the boiler was increased.

3.5 The Effect of Heat Loss Due to Imperfect Combustion on PB Efficiency

Incomplete combustion occurs as signified by the presence of CO content in the flue gas analysis. Furthermore, it results from insufficient O₂ to burn the fuel completely into carbon dioxide (CO₂) and water [13].

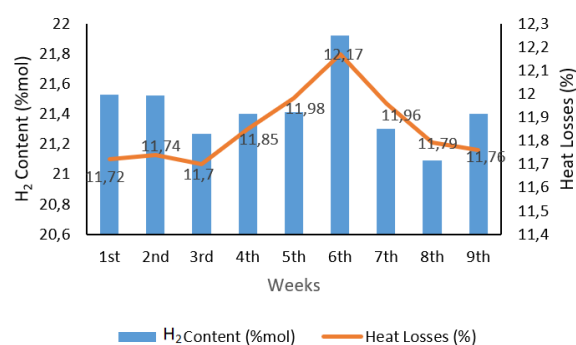


Figure 3. Effect of H₂ in fuel on heat loss

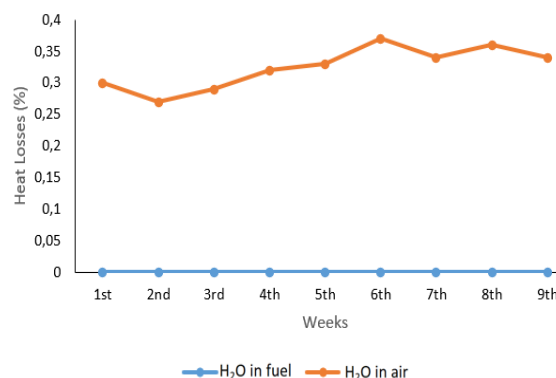


Figure 4. Graph of heat loss due to moisture in air and fuel

Evaluation of Heat Loss Effect on Package Boiler Performance (5007-U) in the Utility Unit of Urea Fertilizer Industry

Figure 5 shows the weekly fluctuations in heat loss due to incomplete combustion, which is signified by the high CO component. On week 6th, exactly on August 8, 2023, the calculated losses were quite high, with CO content in boiler flue gas reaching approximately 5.89%. This percentage is relatively high when compared to other weeks. The increased levels were attributed to a lower combustion air rate of approximately 40,541.72 kg/hour. The reduced airflow resulted in uneven air distribution in the boiler, leading to incomplete combustion and higher CO production in the flue gas. Incomplete combustion occurs when there is not enough O₂ to burn the fuel completely into CO₂ and water [14].

On weeks 7th and 9th, heat loss decreased drastically because CO output in the flue gas analysis was small. During weeks 8th and 9th, CO content was 0, implying that all natural gas was completely burned. This was attributed to a change in the setting of combustion air operating conditions.

3.6 The Effect of Heat Loss Due to Radiation and Convection on PB Efficiency

Heat loss due to radiation and convection occurs when heat energy from PB is transferred to the environment instead of being utilized for steam production. The influencing factors include ambient temperature, natural gas flow, and the Gross Heating Value (GHV) of natural gas. The amount of heat loss depends on the temperature of the hot surface which is affected by the insulation (thickness, thermal conductivity, and condition) [15].

Figure 6 shows heat loss caused by convection and radiation phenomena. Heat loss due to radiation fluctuates weekly, and

the amount depends on the surface area of PB. These losses become significant when PB operates at low loads [16]. Additionally, variations in natural gas flow during the combustion process can affect the air temperature in the system. On weeks 6th and 9th, exactly on August 8 and 29, the natural gas flow had the lowest value. This affects the thermal conditions and air humidity in the room or system, thereby influencing the temperature of the environment surrounding boiler.

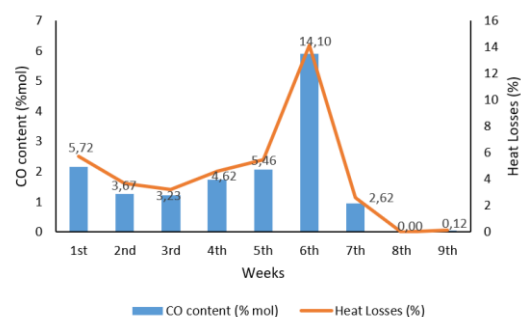


Figure 5. Heat loss due to imperfect combustion

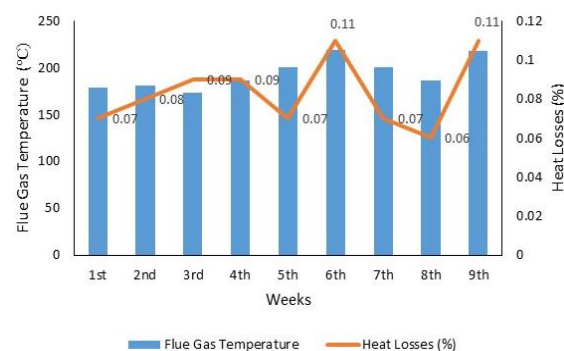


Figure 6. Heat loss due to radiation and convection

4. Conclusion

The best PB performance efficiency was obtained on week 9th (August 29, 2023), reaching 80.71%. This was attributed to optimal airflow settings and increased excess O₂ content, which facilitated complete combustion and minimized CO content. The value obtained approached the design data of 84%.

Evaluation of Heat Loss Effect on Package Boiler Performance (5007-U) in the Utility Unit of Urea Fertilizer Industry

Meanwhile, the lowest efficiency of 65.44% was achieved on week 6th (August 8, 2023). The decrease in PB performance was caused by several factors, including heat loss carried by flue gas, H₂ content in fuel, moisture in the air, incomplete combustion, as well as radiation and convection from the boiler. The highest efficiency was achieved when excess O₂ in

the flue gas reached an optimal limit. An excess O₂ of 3% was sufficient to ensure optimal heat production without losses due to incomplete combustion. The highest heat loss, which impacted efficiency, was caused by H₂ content in the natural gas. The highest total amount of heat loss signified the lowest PB efficiency.

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Silica Adsorption from Boiler Effluent Using Activated Charcoal Derived from Palm Oil Fibre Waste with H_3PO_4 Activator

Adsorpsi Silika dari Limbah Boiler Menggunakan Arang Aktif yang Berasal Dari Limbah Serat Kelapa Sawit Dengan Aktivator H_3PO_4

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Abstract

Most waste from palm oil processing can be repurposed, such as using palm fibre waste to create activated charcoal. This study aimed to assess the effectiveness of activated charcoal, produced by activating palm fibre with H_3PO_4 , in removing silica from boiler output water. The production process involved pyrolysis at 200°C for 2.5 hours. A completely randomized design was employed to test the effects of different H_3PO_4 concentration (with 1 to 2 M) and reaction times (30, 60, and 90 min). Results revealed that the moisture content of the activated charcoal ranged from 1.96% to 2.42%, ash content from 9.82% to 21.63%, and iodine adsorption from 366.43 to 457.87 mg/g. The highest silica adsorption capacity (3.5 g/g) was achieved with 2 M H_3PO_4 for 90 min. This indicates that palm fibre-derived activated charcoal is effective for silica removal, highlighting it is potential for enhancing environmental sustainability in industrial sector.

Keywords: activated charcoal; adsorption; fibre waste; phosphoric acid; pyrolysis

Abstrak

Sebagian besar limbah dari pengolahan minyak kelapa sawit dapat dimanfaatkan, seperti menggunakan limbah serat kelapa sawit untuk membuat arang aktif. Penelitian ini bertujuan untuk menilai efektivitas arang aktif yang dihasilkan dari aktivasi serat kelapa dengan H_3PO_4 dalam menghilangkan silika dari air limbah boiler. Proses produksi melibatkan pirolisis pada suhu 200°C selama 2,5 jam. Desain acak lengkap digunakan untuk menguji efek konsentrasi H_3PO_4 (1 hingga 2 M) dan waktu reaksi (30, 60, dan 90 menit). Hasilnya menunjukkan bahwa kadar air arang aktif berkisar antara 1,96% hingga 2,42%, kadar abu antara 9,82% hingga 21,63%, dan daya serap yodium antara 366,43 hingga 457,87 mg/g. Daya serap silika tertinggi (3,5 g/g) dicapai dengan 2 M H_3PO_4 selama 90 menit. Ini menunjukkan bahwa arang aktif dari serat kelapa sawit efektif dalam menghilangkan silika, menyoroti potensinya untuk meningkatkan keberlanjutan lingkungan di sektor industri.

Kata kunci: adsorpsi; arang aktif; asam fosfat; limbah serat; pirolisis

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1. Introduction

Along with the rapid development of the palm oil processing industry in Indonesia, the volume of waste produced is also increasing every year. During each processing stage, palm oil mills produce significant amounts of solid waste, including empty palm fruit bunches, kernel waste, sludge decanter, fibre, and liquid by-products, which make up 23%, 6.5%, 4%, 13%, and 50% of the total waste, respectively. Although these materials are commonly seen as waste, they are more accurately described as by-products due to their potential for use in various applications [1]. For example, palm fibre is obtained from the pulp of oil palm fruits after processes like kernel crushing and mechanical pressing in digesters and screw press machines [2]. This fibre is rich in nutrients such as phosphorus (P), calcium (Ca), magnesium (Mg), and carbon (C). Its composition mainly consists of lignocellulosic compounds, which are complexes of lignin, cellulose, and hemicellulose. Hemicellulose, with its hydrophilic properties, aids in water absorption, contributing to the fibre irregular structure [3]. In contrast, cellulose, when dry, shows hygroscopic tendencies, readily absorbing water while maintaining hardness and brittleness [4].

Given its nutrient-rich and fibrous composition, palm fibre can be transformed into activated charcoal, a solid substance characterized by its porous structure, which excels in adsorbing particles from liquids through surface adsorption. Adsorption, defined as the adhesion of molecules from a fluid onto a surface, occurs within these pores, enhancing the material's effectiveness in removing contaminants like colorants, odors, and dissolved metals from

water [3, 5]. A recent study conducted by Herlambang et al. [6] has reported that palm kernel shell activated with 4.78% phosphoric acid (H_3PO_4) and 24.67 hours of reaction time can produce fixed carbon of 75.3008%. Although these studies primarily focus on enhancing carbon yield rather than metal adsorption, they underscore the potential of biomass sources for producing effective activated charcoals. In the context of palm fibre, activated charcoal production involves chemical activation with H_3PO_4 , which enlarges carbon pores compared to base activators, thereby boosting its adsorption capacity [7-9]. This process typically includes dehydration, exposure to sunlight for preconditioning, carbonation at high temperatures ranging from 300 to 900°C, and a final chemical activation step to maximize pore accessibility [6, 10].

One of the critical applications of activated charcoal in the palm oil industry is in the treatment of boiler water. Boilers play a crucial role in palm oil mills, functioning as pressurized vessels that transform water from H_3PO_4 plants into steam for various industrial processes, such as electricity generation and heating [11, 12]. After use, boiler water containing minerals like phosphate, sulfite, iron, alkali, and notably silica, is discharged. Silica, in particular, presents significant challenges as it tends to precipitate and create scale deposits on boiler surfaces when heated and then cooled. Its inherent stability and hydrophobic properties worsen these issues, requiring effective strategies to preserve boiler efficiency and lifespan [13, 14]. It is vital to reduce the silica content in boiler output water to prevent scale formation and deposition within the boiler system. Elevated silica levels can result in serious operational problems, including decreased

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heat transfer efficiency, higher energy consumption, and potential equipment damage, putting overall boiler performance and longevity at risk.

This research aims to explore the feasibility of using activated charcoal made from palm fibre waste to reduce silica levels in the water discharged from boilers in palm oil mills. By adsorbing silica, the study seeks to encourage water recycling efforts, which can help conserve important boiler feed water resources and improve overall operational sustainability. Utilizing palm fibre waste for activated charcoal not only helps address environmental issues related to waste disposal but also enhances resource efficiency in the palm oil processing sector. The study intends to establish a practical framework that combines waste utilization with water management strategies to support sustainable development objectives in industrial settings. By demonstrating the efficacy of palm fibre-derived activated charcoal in silica removal, this research aims to offer practical insights for enhancing water reuse efficiency and reducing the environmental impact of palm oil production.

2. Research Methods

2.1 Tools and Materials

The raw materials used in this research were palm fibre and boiler output water obtained from PT. Anaktuha Sawit Mandiri, located in Bumi Ratu Nuban Village, Bumi Ratu District, Central Lampung Regency. Materials for analysis included 0.1 N iodine solution, 1 M H_3PO_4 (Merck, Germany), 2 M 1% starch indicator, distilled water, and Na_2SO_3 (Merck, Germany). The tools used in this research include a silica meter (Hach, USA), a 100 mesh sieve (Retsch,

Germany), a pyrolysis unit (Carbolite, UK), a pH meter (Hanna Instruments, USA), a thermometer (Fisherbrand, USA), an oven (Mettler, Germany).

2.2 Experimental Procedures

This research was carried out in two stages. The first stage involved the production of activated charcoal from palm fibre, followed by the second stage which tested the performance of activated charcoal on the adsorption capacity of silica content in boiler output water. The study focused on the impact of the activator H_3PO_4 and reaction time. The research design utilized the Completely Randomized Design method with two factors, as shown in Table 1.

Table 1. Experimental procedure to reduce silica content in the boiler water

H_3PO_4 concentration (M)	Boiler feed (min)	Repetitions	
		1	2
1	30	X1Y1	X1Y1
	60	X1Y2	X1Y2
	90	X1Y3	X1Y3
2	30	X2Y1	X2Y1
	60	X2Y2	X2Y2
	90	X2Y3	X2Y3

2.3 Activated Charcoal Production

Fibre waste was cleaned and removed from impurities such as remaining kernels and shells, then dried in the sun for 2 days. Next, the palm oil fibres are charred or carbonized using pyrolysis at an operating temperature of 200 °C for 2.5 hours, and a proses slightly modified by Herlambang et al. [6] and Elisa et al. [15]. The resulting charcoal was reduced in size using a 60-mesh disk mill and sieved using a 100-mesh sieve.

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The weight of the resulting charcoal powder was measured to determine the yield of charcoal formed. The dried charcoal was then soaked at a temperature of 200 °C using an activator in the form of a 1 M phosphoric acid solution for half of the sample and with 2 M phosphoric acid for the other half for 24 hours. The ratio of activated charcoal to phosphoric acid activator is 1:4, meaning that every 1 g of activated charcoal is activated with 4 mL of phosphoric acid. The soaked materials are filtered, and the residue is washed with distilled water until the pH is neutral. After washing, the activated carbon is dried in an oven at 105 °C for 1 hour, then placed in a desiccator to cool.

2.4 Analyzing the properties of activated charcoal

2.4.1 Moisture Content

Procedure for determining the amount of water refers to the Indonesian National Standard (SNI) 06–3730-1995, which outlines quality requirements and testing procedures for activated charcoal by Laresha et al. [16]. The moisture content was determined by weighing 1 g of activated charcoal and placing it in a porcelain cup that had been dried and weighed. The sample was then heated in an oven at 105°C for 1 hour, cooled in a desiccator for 15 min, and reweighed. Equation 1 can be utilized to calculate the moisture content. Where m_1 represents the mass of the cup plus sample before being placed in the oven, and m_2 represents the mass of the cup plus sample after being in the oven.

2.4.2 Ash content

A Pyrex porcelain cup with a known mass was used to weigh two grams of the

substance [6]. The samples were placed in a furnace (B-One, China) and exposed to ash for three hours at a maximum temperature of 550°C. The sample was weighed until a consistent weight was reached after cooling it for 15 to 30 min in a desiccator. The amount of ash contained was determined using equation 2. Where W is the mass of the sample after the furnace, m_1 is the mass of the empty crucible, and m_2 is the mass of the sample itself. This calculation provides the ash content as a percentage of the sample mass.

$$\text{Moisture (\%)} = \frac{m_1 - m_2}{m_1} \times 100\% \quad \dots\dots\dots(1)$$

$$\text{Ash (\%)} = \frac{m - m_1}{m_2} \times 100\% \quad \dots\dots\dots(2)$$

$$A = \frac{B \times N_1}{N_2} \quad \dots\dots\dots(3)$$

$$C = \frac{A \times 126.93 \times fp}{a} \quad \dots\dots\dots(4)$$

2.4.3 Iodine Absorption Capacity

The iodine absorption test was conducted by weighing 0.5 g of activated carbon and mixing it with 5 mL of 0.1 N iodine solution. The mixture was then shaken with a shaker for 15 min and filtered. The filtered solution was adjusted back to its original volume by adding distilled water up to 25 mL. The filtrate solution was shaken, 5 mL was extracted, and titrated with 0.1 N sodium thiosulfate solution. If the yellow color of the solution starts to fade, 1% starch solution was added as an indicator [6, 15]. The dark blue color was titrated again until it became clear. Iodine absorption capacity was determined using equation 3 and 4. Where A is the volume of the iodine solution (mL), B is the volume of Na₂S₂O₃ used (mL), C is iodine absorption capacity, fp is the dilution factor, a is the weight of the activated carbon (g), N_1 is the

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concentration of iodine, N₂ is the concentration of iodine, and 126.93 is the amount of iodine corresponding to 1 mL of Na₂S₂O₃ solution.

2.4.4 Performance of activated charcoal for reducing silica in boiler water

Activated charcoal resulting from combustion at a temperature of 200 °C, which had been activated with 1 M and 2 M H₃PO₄, was weighed at 2.5 grams each. The charcoal was then placed into a glass beaker containing a 100 mL sample of boiler output water. Subsequently, dissolution was conducted on a hot plate using a magnetic stirrer set at a stirring speed of 200 rpm. This process was repeated for 30 min, 60 min, and 90 min, each carried out twice, and then analyzed using a silica meter. This procedure was slightly modified from Fadilah et al. [17] for phosphorus removal. Silica removal was determined using equation 5. Where C_o and C_e represent initial and final concentrations, respectively.

$$\text{Silica removal(\%)} = \frac{C_o - C_e}{C_o} \times 100\% \quad \dots(5)$$

3. Results and Discussion

3.1 Moisture content

Based on the test results in Figure 1, the moisture content of activated charcoal from palm fibre is relatively low and meets the quality requirements of SNI 06-3730-1995. The highest moisture content value was $2.42 \pm 0.13\%$ in the 2 M H₃PO₄ solution. Meanwhile, the lowest moisture content value was $1.96 \pm 0.01\%$ without activation. A study by Herlambang et al. [6] has reported that the moisture content of the adsorbent derived from palm kernel shells activated with H₃PO₄ was reported to be 1.0007%. This indicates that the moisture

content increases with the acidity of phosphoric acid. High moisture content can negatively impact the absorption capacity of activated charcoal for liquids and gases. Low moisture content is desirable as it can lead to a higher absorption capacity due to the pores remaining unobstructed by moisture. A lower moisture content provides more available spaces in the activated charcoal pores for adsorbate molecules, optimizing the adsorption process. Moisture molecules in activated charcoal are absorbed by the activator, enlarging the pores in the carbon ash. Larger pores result in increased surface area of activated charcoal, enhancing its adsorption ability [18].

3.2 Ash content

Based on the test results in Figure 2, the ash content of palm fibre activated charcoal meets the quality requirements of SNI 06-3730-1995. The highest ash content value was $21.63 \pm 0.17\%$ without activation. Meanwhile, the lowest ash content was $9.82 \pm 0.23\%$ in the 2 M H₃PO₄. The high ash content is due to the activation process where the activator can react with the metals covering the surface of the activated charcoal to form salts that can dissolve in the activator solution. When heated at 900°C, these salts also burn. The high and low levels of activated charcoal ash are influenced by minerals. Inorganic substances present in activated charcoal can be dissolved by the activator. Ash may develop due to the mineral elements like calcium, potassium, sodium, and magnesium found in activated charcoal. These elements disperse across the activated charcoal grid, clogging the pores. The residual minerals block the pores of the activated charcoal, hindering the adsorption process [19].

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3.3 Iodine absorption

Based on the quality requirements for activated charcoal, specifically SNI 06-3730-95, the minimum iodine absorption capacity should be 750 mg/g. However, the iodine absorption capacity of activated charcoal derived from palm fibre falls short of this standard due to impurities that obstruct the pores of the activated charcoal, resulting in insufficient micropores to adsorb the iodine solution. The highest iodine absorption value recorded was 457.87 ± 32.01 mg/g in the 1 M H_3PO_4 , while the lowest iodine absorption capacity was 366.43 ± 3.52 mg/g without activation.

Figure 3 illustrates the iodine adsorption capacity of activated charcoal activated with 1 M and 2 M H_3PO_4 . It is

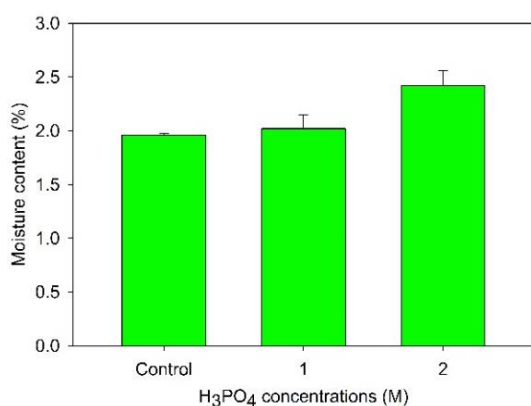


Figure 1. Moisture content results with various concentrations of H_3PO_4

evident that as the molarity value of the activator increases, there is a decrease in iodine adsorption level, indicating a significant impact on the quality of activated charcoal in terms of pore formation and impurity absorption. The surface area of activated charcoal pores is a crucial parameter that influences its quality by affecting its adsorption capacity. The variance in iodine absorption capacity is attributed to the lack of mesopores and macropores in the activated charcoal for iodine absorption. The high iodine absorption capacity is linked to the formation of a micropore structure pattern, signifying the large pore diameter of activated charcoal that can only accommodate molecules with a diameter of less than 10 Angstroms [20].

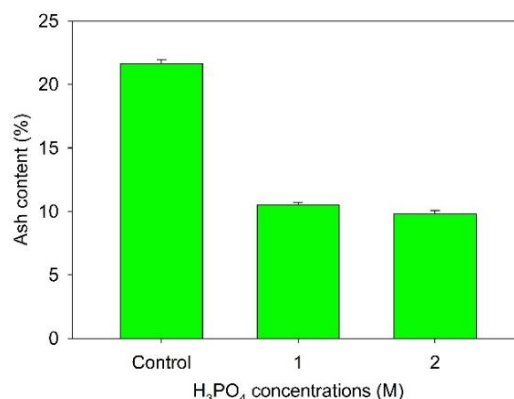


Figure 2. Ash content result with different H_3PO_4 concentrations

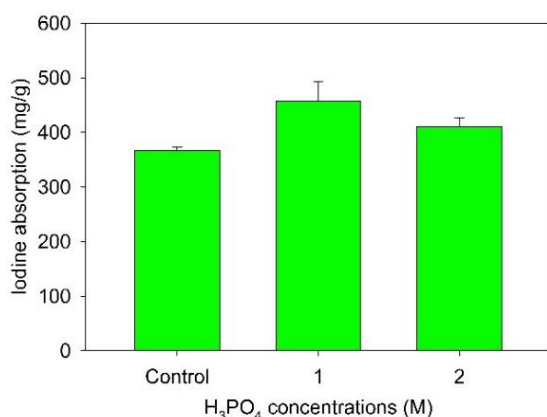


Figure 3. Iodine absorption content result with different H_3PO_4 concentrations

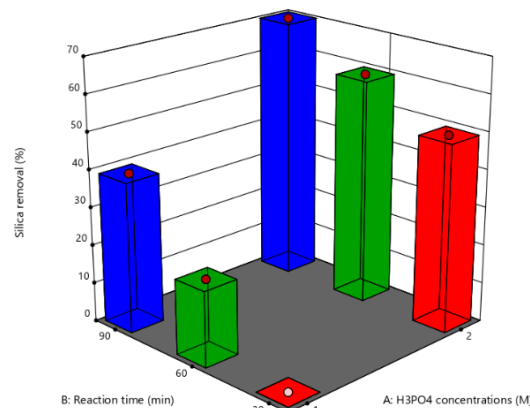


Figure 4. Silica removal result with different H_3PO_4 concentrations and reaction time

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3.4 Activated charcoal performance

Figure 4 illustrates the effect of H_3PO_4 concentrations (1 M and 2 M) and reaction times (30, 60, and 90 min) on silica removal percentages. At the shortest reaction time of 30 min, silica removal was minimal with 1 M H_3PO_4 (0%), moderate with 1.5 M (20%), and highest with 2 M (30%). As the reaction time increased to 60 min, silica removal improved significantly across all concentrations: 1 M reached about 40%, 1.5 M achieved around 50%, and 2 M approached 60%. At the longest reaction time of 90 min, silica removal continued to increase, with 1 M reaching approximately 50%, 1.5 M achieving about 60%, and 2 M peaking around 70%. This trend indicates that both higher concentrations of H_3PO_4 and longer reaction times contribute positively to the efficiency of silica removal. In addition, a higher activator concentration causes the driving force of the adsorbate molecules to be higher so that the number of silica molecules absorbed becomes greater [21].

Adsorption of silica from boiler water using activated charcoal derived from palm fibre waste involves a detailed understanding of the physicochemical interactions between silica and the activated charcoal surface. Silica, a common impurity in boiler water, can precipitate and form scale under high-temperature conditions, reducing boiler efficiency. The adsorption process relies on the ability of activated charcoal to attract and retain silica molecules on its surface and within its pores, mitigating scale formation and improving boiler performance. Factors influencing adsorption efficiency include surface area, pore size distribution, pH of the water, temperature, and the presence of competing ions [12]. The application of

activated charcoal from palm fibre waste offers a sustainable solution by utilizing agricultural residues, contributing to waste valorization and environmental sustainability in boiler water H_3PO_4 applications.

3.5 Future prospect and challenges

Looking ahead, the future prospects for utilizing activated charcoal derived from palm fibre waste for silica removal in boiler water H_3PO_4 are promising yet face several challenges. Firstly, the sustainability aspect of using agricultural residues aligns with global initiatives towards circular economy practices and sustainable development goals. This approach not only reduces environmental impact by repurposing waste materials but also supports cost-effective and environmentally friendly solutions in industrial water H_3PO_4 . Advances in activation techniques and material science can further optimize the surface area and pore structure of activated charcoal, enhancing its adsorption capacity and efficiency in removing silica and other contaminants from boiler water.

However, several challenges must be addressed to fully realize the potential of this technology. The variability in feedstock quality and composition of palm fibre waste can impact the consistency and performance of activated charcoal. This necessitates rigorous quality control measures and standardization in production processes to ensure reliable and effective adsorption properties. Moreover, while activated charcoal offers sustainable advantages, the cost-effectiveness and scalability of production remain critical considerations. Balancing production costs with performance requirements and market competitiveness is essential for widespread adoption in diverse industrial applications.

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In addition to technological challenges, regulatory compliance, and competition from alternative water H₃PO₄ technologies pose significant hurdles. Meeting stringent regulatory standards for water quality and discharge limits requires continuous innovation and adaptation of H₃PO₄ technologies. Furthermore, competing technologies such as chemical precipitation and membrane filtration offer alternative approaches to silica removal, prompting comparative studies and technological advancements to demonstrate the unique advantages of activated charcoal-based adsorption systems. Overcoming these challenges will require collaborative efforts across academia, industry, and regulatory bodies to drive innovation, optimize operational efficiencies, and promote sustainable practices in boiler water H₃PO₄ using activated charcoal from palm fibre waste.

4. Conclusion

Activated charcoal produced from palm fibre waste via pyrolysis and H₃PO₄ activation exhibits promising characteristics for water treatment

applications, particularly in silica removal from boiler water. The results demonstrated that moisture content, ash content, and iodine absorption capacity were significantly influenced by the concentration of H₃PO₄ used during the activation process. The study found that higher concentrations of H₃PO₄ led to increased moisture content and lower ash content, which positively influenced the adsorption capacity of the activated charcoal. Specifically, the activated charcoal produced using a 2 M concentration of H₃PO₄ was most effective, achieving up to 70% silica removal after 90 min of reaction time. This concentration not only optimized the pore structure of the charcoal, leading to better adsorption efficiency but also minimized ash content, enhancing the overall quality of the activated charcoal. This finding highlights the potential of utilizing agricultural waste products like palm fibre in creating sustainable solutions for industrial water treatment, contributing to both resource efficiency and environmental sustainability.

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Synthesis of $KAl(SO_4)_2$ Solid Coagulants from Used Pots and Beverage Cans

Sintesis Koagulan Padat $KAl(SO_4)_2$ dari Panci Bekas dan Kaleng Minuman Bekas

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Abstract

Used pots and beverage cans are good sources of aluminum-rich raw materials for the synthesis of potassium aluminum sulfate ($KAl(SO_4)_2$), a solid coagulant. The synthesis process includes preparation, dissolution, extraction, sedimentation, and drying. Therefore, this research aimed to determine the characteristics of $KAl(SO_4)_2$ synthesized from aluminum pots and beverage cans waste, adjusted to the quality requirements of commercial $KAl(SO_4)_2$ according to SNI 06-2102-1991 standard. The materials used were aluminum pots, as well as a mixture of pots and beverage cans, with varying concentrations of 20%, 30%, and 40% KOH solvent. The synthesis results, characterized by XRF (X-ray fluorescence), showed an Al content of 0.001-3%. In addition, the results of the data analysis, adapted to SNI 06-2102-1991 standard for potassium aluminum sulfate, indicated that the synthesis met the required parameters for water-insoluble parts, Fe, Pb, and As, and Al_2SO_3 , which was close to the quality requirements.

Keywords: aluminum pots; beverage cans; coagulant; extraction; potassium aluminum sulfate

Abstrak

Koagulan padat kalium aluminium sulfat ($KAl(SO_4)_2$) dapat disintesis menggunakan bahan baku dengan kandungan aluminium tinggi, seperti panci dan kaleng minuman bekas. Proses sintesis meliputi preparasi, pelarutan, ekstraksi, sedimentasi, dan pengeringan. Oleh karena itu, penelitian ini bertujuan untuk mengetahui karakteristik $KAl(SO_4)_2$ hasil sintesis dari limbah panci dan kaleng minuman aluminium, yang disesuaikan dengan persyaratan mutu $KAl(SO_4)_2$ komersial menurut standar SNI 06-2102-1991. Bahan yang digunakan adalah panci aluminium, serta campuran panci dan kaleng minuman, dengan variasi konsentrasi pelarut KOH 20%, 30%, dan 40%. Hasil sintesis yang dikarakterisasi dengan XRF (X-ray fluorescence) menunjukkan kadar Al sebesar 0,001-3%. Selain itu, hasil analisis data yang disesuaikan dengan standar SNI 06-2102-1991 untuk kalium aluminium sulfat menunjukkan bahwa sintesis tersebut memenuhi parameter yang disyaratkan untuk bagian yang tidak larut dalam air, Fe, Pb, dan As, serta Al_2SO_3 , yang mendekati persyaratan mutu.

Kata kunci: ekstraksi, kaleng minuman, Kalium Aluminium Sulfat, koagulan, panci aluminium

Synthesis of $KAl(SO_4)_2$ Solid Coagulants from Used Pots and Beverage Cans**1. Introduction**

The increasing population in Indonesia has caused the production of both organic and inorganic waste. One type of inorganic waste that is widely produced is aluminum waste, which requires approximately 400 years to decompose [1]. In this regard, household activities generate about 38.13% of Indonesia's waste, with 3,520 tons of aluminum waste produced daily in 2022 [2]. The current waste management is still limited to collecting and selling without prior processing, even though the aluminum content in pots and beverage cans can be reused by recycling [3].

The percentage of aluminum content in used pots ranges from 97.93% to 99.05% [4], while beverage cans percentage ranges from 92.5% to 97.5% [3]. The high aluminum content in used pots and beverage cans can be synthesized into coagulants, which form flocs by coagulating suspended solid particles, dyes, and colloids [5]. The coagulant formed from this synthesis include Potassium Aluminum Sulfate ($KAl(SO_4)_2$), which is commonly used in wastewater treatment, purification, and fire extinguishers [6].

$KAl(SO_4)_2$ coagulant can be formed from aluminum materials, and several brands of pots in the community have a high percentage of aluminum. Furthermore, the Eagle brand pots contain 99.05% aluminum, Djawa contains 99.26%, and Orchid contains 97.93% [4]. The aluminum content of used beverage cans of the Pocari Sweat brand is 96.38%, Cap Kaki Tiga is 89.74%, Greensand is 90.87%, and Coca-Cola is 93.28% [7],[8].

Solid-liquid extraction is used in the synthesis of $KAl(SO_4)_2$ because it can dissolve certain substances in solid

materials. Factors affecting the formation of $KAl(SO_4)_2$ include the concentration of solid base solvents (KOH) and solid acids (H_2SO_4), the precipitation process, and drying [9]. The amount of aluminum content synthesized can be affected by the solvent used, such as KOH. In addition, H_2SO_4 plays a role in the precipitation of dissolved aluminum, and drying affects the water content of the final product [10].

Aluminum is extracted from used beverage cans to produce coagulants with a high yield percentage of 97.95% and a water turbidity removal efficiency of 70% [11]. Moreover, reacting 1 g of used beverage cans in 30% KOH and 8 M H_2SO_4 and drying the mixture at 50°C, can produce 14.8 g of $KAl(SO_4)_2$ [9]. Similar research had produced coagulants with physical form, odor, pH, and quality characteristics almost identical to commercial $KAl(SO_4)_2$ [12]. It can also be synthesized from aluminum spoons and food containers and applied in raw water treatment [13]. Furthermore, its synthesis with aluminum foils as the base materials can produce coagulants with an Al_2SO_3 content of 15.18% [14]. Therefore, this research aimed to determine the characteristics of $KAl(SO_4)_2$ synthesized from aluminum pots and beverage cans waste, adjusted to the quality requirements of commercial $KAl(SO_4)_2$ according to SNI 06-2102-1991 standard.

2. Research Methods**2.1. Tools and Materials**

The raw materials used were aluminum pots and Pocari Sweat brand beverage cans. The materials used in the synthesis process included KOH, H_2SO_4 , and distilled water. In this research, the equipment consisted of an analytical balance, drying oven, desiccator, and XRF

Synthesis of $\text{KAl}(\text{SO}_4)_2$ Solid Coagulants from Used Pots and Beverage Cans

(X-Ray Fluorescence) Spectrometer AMETEK, which aimed to determine the composition of metal content with quantitative data.

2.2. Sample Preparation

The initial stage to prepare the raw materials, namely used pots and beverage cans, included cleaning with coarse sandpaper, cutting into approximately 1 cm in size, and soaking in hot water for a few moments. Therefore, this stage removed visible impurities, and the preparation process aimed to adjust the size and shape of the raw materials before the next process.

2.3. Sample Dissolution Process

The dissolution process aimed to dissolve the raw materials using KOH to produce potassium aluminate. In this research, the concentration of KOH solvent was varied by 20%, 30%, and 40% in a volume of 50 mL. At each concentration variation, 1 g of pots as variation A and 0.5 g of pots + 0.5 g of beverage cans as variation B were added into the KOH solvent. The dissolution process was carried out on a hot plate at 50°C.

2.4. Extraction Process of $\text{KAl}(\text{SO}_4)_2$

Potassium aluminate filtrated from the sample dissolution process (45 mL) was reacted with sulfuric acid reagent (H_2SO_4) in a volume of 30 mL, maintaining a ratio of 3:2. In this process, H_2SO_4 was gently dripped into the filtrate and stirred continuously. Occasionally, a few drops of distilled water were added to prevent clumping.

2.5. Precipitation and Drying Process

The precipitation process was carried out by placing the extracted solution inside

the freezer for 1 hour at a temperature of 7°C. Subsequently, the resultant $\text{KAl}(\text{SO}_4)_2$ was washed using 50% ethanol. Drying was conducted using a drying oven at a temperature of 50°C for 1 hour and continued until a product with a constant weight was produced.

3. Results and Discussion

3.1. Synthesis Process

Dissolving aluminum using KOH is a reduction-oxidation (redox) reaction. This reaction released H_2 gas, indicated by the appearance of bubbles and smoke during the process. Furthermore, the bubbles and smoke were produced by the reaction between aluminum pieces from pots or cans and aluminum cations [15]. This process was exothermic, and released heat into the environment as well as produced bubbles. The reaction was concluded when no more bubbles were formed, and filtration was carried out to separate the potassium aluminate filtrate from other impurities. The reaction in the extraction process using H_2SO_4 was denoted as follows:

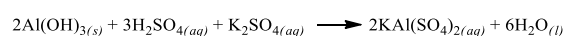
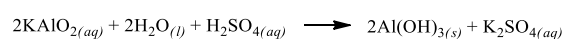
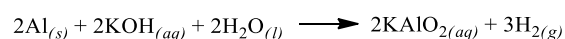


Figure 1. Physical form of $\text{KAl}(\text{SO}_4)_2$

Synthesis of $\text{KAl}(\text{SO}_4)_2$ Solid Coagulants from Used Pots and Beverage Cans

Adding H_2SO_4 affects the formation of the $\text{KAl}(\text{SO}_4)_2$ coagulant as it precipitates the aluminum bound by the KOH solution. Therefore, this reaction produced an $\text{Al}(\text{OH})_3$ white residue, excess H_2SO_4 was added to dissolve $\text{Al}(\text{OH})_3$, and distilled water was occasionally added while stirring continuously [13]. Figure 1 shows the physical form of $\text{KAl}(\text{SO}_4)_2$ from the research results.

3.2. Characterization Results

The synthesis of $\text{KAl}(\text{SO}_4)_2$ coagulant used materials with high aluminum content as the basic element. Testing of the coagulant product was needed to determine the amount of extracted aluminum content. Moreover, testing the characteristics using XRF aimed to determine the metal content with data presented quantitatively. The XRF testing used the AMETEK XRF Spectrometer instrument to determine a material's

chemical composition. Also, testing the characteristics of $\text{KAl}(\text{SO}_4)_2$ was conducted using the SNI 06-2102-1991 standard to compare the quality requirements of the same product. The results of the product characterization showed that the elements with the largest percentage were dominated by Potassium (K), Aluminum (Al), and Sulfur (S). This was because the elements forming $\text{KAl}(\text{SO}_4)_2$ consisted of Potassium, which originated from a strong base as a solvent, Aluminum from pots and cans, as well as Sulfur from H_2SO_4 in fairly high amounts. In this research, the concentration of KOH did not affect the percentage of extracted K because KOH only acted as a solvent. Testing the characteristics of $\text{KAl}(\text{SO}_4)_2$ was conducted using SNI 06-2102-1991 standard to compare the quality requirements of the same product that had been traded.

Table 1. Characterization Results

Parameter	Sample						Standard SNI 06- 2102- 1991
	A1	A2	A3	B1	B2	B3	
Water Insoluble Part (%)	0.00238	0.012	0.0246	0.0112	0.0114	0.0212	Max 0.5
Al_2O_3 (%b/b)	0.1016	6.266	0.4193	2.533	0.00255	1.773	Min. 8
Fe (%b/b)	<0.00013	0.00277	<0.00019	0.00259	<0.00015	<0.00015	Max. 0.01
Pb (mg/kg)	<0.3	<0.3	<0.3	<0.3	<0.3	<0.3	Max. 50
As (mg/kg)	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	Max. 50
K (%b/b)	0.00009	6.845	1.925	1.986	0.00125	2.698	-
S (%b/b)	1.529	1.300	5.645	4.554	9.49	4.593	-

Description:

A1: Pots and 20% KOH solvent

A2: Pots and 30% KOH solvent

A3: Pots and 40% KOH solvent

B1: Pots + Cans and 20% KOH solvent

B2: Pots + Cans and 30% KOH solvent

B3: Pots + Cans and 40% KOH solvent

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Table 1 shows that the parameter of the water insoluble part met the quality requirements, which was below 0.5%. The water-insoluble part is a non-polar substance that does not dissolve when reacting with water [16]. The $KAl(SO_4)_2$ product synthesized from used beverage cans met the parameter requirements for the water-insoluble part, which was below 0.5%. This parameter indicated the amount of impurity in the product. The greater the impurity in alum, the greater the percentage of the water-insoluble part [9].

The Al_2O_3 content value from the synthesis of the $KAl(SO_4)_2$ coagulant did not meet the standard requirements based on SNI 06-2102-1991 because it ranged from 0.00255% to 6.266%. Moreover, the Al_2O_3 content produced did not meet the standard requirements contained in SNI 06-2102-1991 because the raw materials of $KAl(SO_4)_2$ were different from commercial $KAl(SO_4)_2$, namely bauxite [1]. The synthesis of $KAl(SO_4)_2$ based on aluminum foil packaging produced a product with an Al_2O_3 content of 15.18% [16]. Although the synthesis materials were also derived from the packaging materials, the type of raw materials and the amount of aluminum content from previous analyses differed from that of this research. Therefore, the resulting product differed, especially for the Al_2O_3 content.

Chemical parameters in the form of Fe content were in the range below 0.00013% to 0.002%. This value was still below the SNI standard, which was <0.01%. The low Fe content characterization results can be attributed to very low Fe content in the raw materials, leading to very small extracted content. Meanwhile, Pb (lead) met the SNI 06-2102-1991 standard with a value below 0.3 mg/kg. The As (arsenic) parameter also

met the standards required in SNI 06-2102-1991, being below 0.2 mg/kg. Arsenic metal content exceeded the threshold and can cause arsenic pollution [17]. In addition, analysis of metal components in the $KAl(SO_4)_2$ product showed the values were safe, and confirmed that used pots and beverage cans were safe to be synthesized into $KAl(SO_4)_2$ coagulant.

3.3. The Effect of KOH on the Weight of $KAl(SO_4)_2$

KOH is a strong base characterized by a white solid form with high solubility in water and forms salt when reacted with acid [9]. The weight of $KAl(SO_4)_2$ produced from this research is presented in Figure 2. The results showed a relationship between the concentration of KOH solvent and the weight of $KAl(SO_4)_2$ produced.

The relationship was directly proportional, meaning that the higher the concentration of KOH solvent used, the higher the weight of $KAl(SO_4)_2$ produced. Previous research on alum formation stated that a constant volume with varying concentrations of KOH base solvent affected the amount of $KAl(SO_4)_2$ produced. Specifically, the higher the concentration of KOH, the more alum was formed [18]. However, in this research, the complex content of pots and cans caused the amount of Al extracted to be indirectly proportional to the increase in KOH concentration.

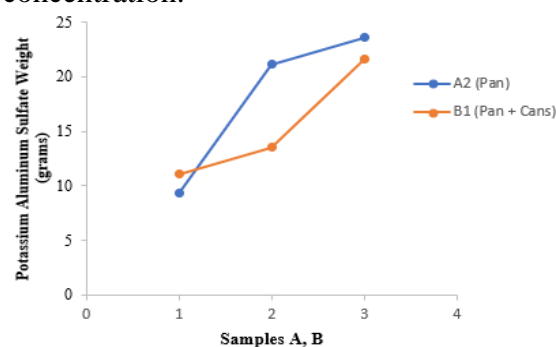


Figure 2. Graph of the Effect of KOH on $KAl(SO_4)_2$

Synthesis of $KAl(SO_4)_2$ Solid Coagulants from Used Pots and Beverage Cans

4. Conclusion

In conclusion, based on the XRF characterization results and data analysis, the Al_2O_3 parameter was still below the standard requirements. Compared to other variations, used aluminum pots materials with a KOH concentration of 30% and aluminum pots + beverage cans with a KOH concentration of 20% produced

Al_2O_3 content close to the quality standard requirements according to SNI 06-2102-1991. Also, when viewed from the comparison of raw materials used, aluminum pots materials produced coagulants with characteristics closest to the reference according to the quality requirements of $KAl(SO_4)_2$.

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Treatment of Textile Industrial Wastewater using Membrane Technology: A Review

Pengolahan Limbah Cair Tekstil dengan Teknologi Membran: Review

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Abstract

Textile industry wastewater is a very complex pollutant-containing waste with high dye intensity, requiring proper and appropriate treatment. Membrane technology is one of the appropriate methods for treating textile wastewater due to several advantages such as environmentally friendly and biopolymer-based processing. Therefore, this review aimed to determine the effectiveness of membrane technology and provide information regarding the appropriate treatment of textile wastewater. The articles subjected to review were obtained from several journal sources such as ScienceDirect, Elsevier, Springer, Google Scholar, and national journals. The results showed that several membranes had been used in textile wastewater treatment, such as PTFE (Polytetrafluoroethylene), PES (Polyethersulfone), Polysulfone-Polyvinyl Pyrrolidone Blend Polymer Composite Membrane, CA (Cellulose Acetate), Cellulose Membrane of Sargassum Sp., polysulfone (PSF), Bacterial Cellulose Membrane, and cellulose acetate propionate (CAP). Furthermore, membrane technology was found to reduce dye pollutants in textile wastewater with the highest coefficient value of approximately 97%.

Keywords: membrane; membrane technology; textile industry; treatment methods; wastewater

Abstrak

Limbah cair industri tekstil merupakan limbah mengandung polutan yang sangat kompleks dengan intensitas pewarna yang tinggi. Karena itu, pengolahan limbah cair tekstil ini harus ditangani dengan baik dan tepat. Teknologi membran merupakan salah satu metode yang tepat dalam mengolah limbah cair tekstil. Pengolahan berbasis ramah lingkungan dan bersifat biopolimer merupakan keunggulan dari teknologi membran. Review ini bertujuan untuk mengetahui efektivitas teknologi membran dan memberikan informasi mengenai teknologi membran dalam mengolah limbah cair tekstil yang tepat. Metode yang digunakan dalam review ini adalah meninjau artikel dari beberapa sumber jurnal seperti sciencedirect, elsevier, springer, google scholar, dan jurnal nasional. Terdapat banyak membran yang digunakan dalam pengolahan limbah cair tekstil, seperti: PTFE (Polytetrafluoroethylene), PES (Polyethersulfone), Polysulfone-Polyvinyl Pyrrolidone Blend Polymer Composite Membrane, CA (Cellulose Acetate), Membran Selulosa Sargassum Sp., polisulfon (PSF), Bacterial Cellulose Membrane, cellulose acetate propionate. Metode teknologi membran dapat mengurangi polutan pewarna dalam limbah tekstil dengan nilai koefisien tertinggi hingga 97%.

Kata kunci: industri tekstil; limbah cair; membran; metode pengolahan; teknologi membran

Treatment of Textile Industrial Wastewater using Membrane Technology: A Review

1. Introduction

Indonesia is one of the largest countries in the sector industry, particularly fabric or textile. This shows that textile industry plays a significant role in the national economy, as indicated by many workers who live and work in the sector. However, waste is often generated as pollution from industrial activities that produce hazardous and toxic materials (B3) [1],[2],[3].

Textile industry is currently dominated by 63% synthetic fibers, most of which are derived from petrochemicals, causing CO₂ emissions. This is followed by cotton, whose production can cause water scarcity and toxic pollution due to its widespread use [4]. Textile wastewater contains very complex pollutants with high dye intensity. Some dyes are toxic and have carcinogenic and mutagenic effects on aquatic life and humans [5],[6]. Remazol red (C₁₈H₁₄N₂Na₂O₁₀S₃), remazol

black ($C_{26}H_{21}N_5Na_4O_{19}S_6$), naphthol yellow ($C_{10}H_4Na_2O_8S$) are examples of dyes commonly used in textile industry. Dyes have high synthetic color content, chemical oxygen demand (COD), and metal values [5],[6],[7],[8],[9]. In the process, 95% of dyes will be discarded and only approximately 5% is effective in binding fabric. Therefore, there is a need for appropriate and environmentally friendly processing methods. Previous analysis of textile wastewater processing is shown in Figure 1.

Current waste industry processing technology uses many chemical and biological processes that require large operational costs and areas of land. One method of processing textile wastewater is using membrane technology. This membrane technology in the separation process has developed rapidly, particularly for processing wastewater [10].

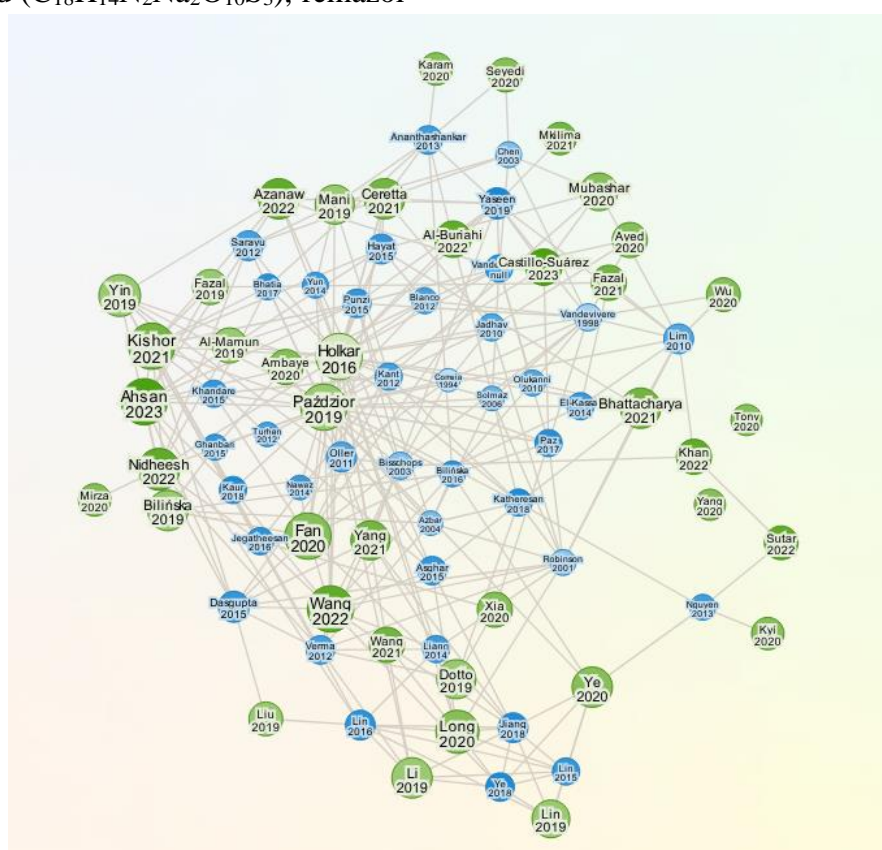


Figure 1. Study on textile wastewater treatment

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Membrane technology can be used in the processing of wastewater, such as liquid tofu waste and domestic wastewater [11],[12]. It is a reliable separation method, specifically in water separation process [13],[14]. In the membrane manufacturing process, the phase inversion method is generally used[12],[15],[16],[17],[18],[19].

2. Research Method

In recent years, textile wastewater treatment has been continuously developed. There are several methods commonly used in textile wastewater treatment taken from several journal sources such as ScienceDirect, Elsevier, Springer, Google Scholar, and several national journals. Literature searches using several keywords such as textile wastewater treatment methods, textile waste, and membrane.

Based on the previous analysis, the methods commonly used in textile wastewater treatment include electrocoagulation [17],[20],[21], electrooxidation-electrocoagulation [20], phytoremediation [22], adsorption [14], [23],[24],[25]–[32],[33], filtration [23],[34],[35],[36], and membrane technology [14],[37]. Therefore, this review was conducted based on research development regarding textile wastewater treatment. The results are expected to provide information to readers regarding textile wastewater treatment and appropriate treatment methods.

3. Textile Wastewater

Wastewater treatment system in textile industry usually include primary, secondary, and tertiary treatments when the quality exceeds the applicable standard values [38]. In this review, the properties of wastewater in textile industry will be

discussed. First, physical properties include density, odor, color, and temperature, the amount of dissolved oxygen (DO). Second, chemical properties include pH, DO, and COD [20]. Third, biological properties where most wastewater contains various types of microorganisms with concentrations ranging from 105 to 108 organisms/mL. In assessing water quality, bacteria also play a significant role. When waste is discharged into the environment, it will have a negative impact on the environment and become a serious problem in the industry era. The properties and composition of textile wastewater are shown in Table 1.

Textile wastewater contains large total suspended solids, bright colors, with high acidity fluctuations, temperatures, as well as concentrations of chromium and phenol. The main contaminants come from the finishing or dyeing process which includes synthetic and natural dyes to produce permanent colors and have good resistance to physicochemical treatment [16],[39].

Table 1. Properties and composition of textile wastewater parameters

Parameter	Value
pH	7,00-9,00
BOD (mg/L)	80-6.000
TTS (mg/L)	15-8.000
COD (mg/L)	150-12.000
Chloride (mg/L)	1.000-1.600
Total Kjeldahl nitrogen (mg/L)	70-80
Color (Pt-Co)	50-2.500
TDS (mg/L)	2.900-3.100

Source: [20]

4. Textile Wastewater Treatment

Several methods that have been applied in the processing of textile wastewater are shown in Table 2. Electrocoagulation can be used in waste

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processing by applying electrochemistry. Electrooxidation-electrocoagulation is a process to reduce organic content in textile wastewater using variations in voltage and time [20]. The phytoremediation method uses plants to reduce pollutants in waste, including water hyacinth plants as biological agents [40]. Adsorption is a method of absorbing liquids or gases where the adsorbate is bound to the adsorbent [14],[23],[25]–[29],[31]–[34]. Furthermore, the filtration method uses filtering media in separation which produces filtrate and residue. This method is included in the membrane technology method but for filtration with macro waste [23],[34],[35],[36].

Membrane technology is a water treatment method based on biopolymers and is environmentally friendly. The advantages of membrane technology include relatively low energy, maintenance of structure-separated substances, and easy operation at room temperature. Furthermore, it is clean and non-toxic due to the absence of additional chemicals. The processing of textile wastewater has a standard quality value that is a reference to avoid polluting the environment. This is regulated in Appendix II of the Regulation of the Indonesia Minister of Environment Number 5 of 2014 concerning Wastewater Quality Standards [41], as presented in Table 3.

Based on Table 2, electrocoagulation method shows COD reduction value of 112 mg/L or 50.98% of the initial COD value of the waste. However, these results do not meet the quality standards for textile wastewater.

Electrooxidation-electrocoagulation method can reduce COD by 397.40 mg/L or 83% of the initial value but does not meet the quality standards. The phytoremediation method

reduces biochemical oxygen demand (BOD) by 165.33 mg/L or 47.2% of the initial value of the waste. This value also does not meet the quality standards for textile wastewater. The adsorption process is only able to reduce the color concentration by 26% of the initial concentration value, which is 95% of the dye produced from liquid textile waste. The filtration method reduces the COD value by 38.81% or 211 mg/L. These results have COD value above 350 mg/L and are not included in the quality standards for textile wastewater. Furthermore, the hybrid filtration system method can reduce COD levels by 72.14% or 1066.25 mg/L from the initial total waste of 3826.6688 mg/L.

Membrane technology method reduces COD value from 1542.36 mg/L to 149.76 mg/L with a removal rate of 90.08%. These results are included in textile wastewater quality standards [13]. Additionally, the Sono-Fenton method provides results that are in environmental quality standards, namely reducing the COD value from 1532.16 mg/L to 143.36 mg/L with a removal rate of 90.64%. The sono-fenton method is effective, but it is not environmentally friendly due to the use of chemicals with a negative impact on the environment.

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Table 2. Textile wastewater treatment method

Treatment Method	Waste Types	Reaction Conditions	Results	Ref.
Electrocoagulation	Wastewater from the batik industry	Reaction time: 20, 60, 100, 140 and 180 minutes Voltage: 12 volts	Decrease in COD Concentration: 50.98%, TSS: 50%, Color: 30%	[42]
Electrooxidation-Electrocoagulation	Wastewater from textile industry	Voltage: 8,12,16, and 20 volts Duration: 15 minutes and 45 minutes	TSS: - BOD: 83% COD: 75%	[20]
Phytoremediation Method	Wastewater from hand-drawn batik industry	Treatment for: 12 days Number of water hyacinths used: 7 pieces	Decrease in Concentration COD: >40% BOD: 47,2% TTS:33%	[40]
Adsorption	Wastewater from the woven fabric industry	Using a photocatalytic reactor and sunlight assistance in the process	Decrease in Color Concentration: 26%	[43]
Filtration (Macro Waste Filtration)	Wastewater from the batik industry	Using coagulant KAl(SO ₄) ₂ ·12H ₂ O (alum)	Decrease in Concentration COD: 38.81% BOD:40.88%	[38]
Hybrid filtration system method	Wastewater from textile industry	Raw material: Bottom Ash Duration: 4 days Temperature: 30-40 °C	Decrease in Concentration: COD: 72.14% Color: 94.78 %	
Membrane Technology	Wastewater from the batik industry	The membrane is heated at temperatures: 80 °C and 90 °C Duration: 1 hour Membrane drying: 24 hours at room temperature Drying membrane: 3 days	Decrease in Concentration of Rhodamine B Dye: B: 80.04%; Methylene Blue: 77.83%; Methyl Orange: 75.84%.	[18]
Membrane Technology	Wastewater from the eco-print textile industry		Reduce COD by 90.08%, BOD by 85.92%, and TOC by 92.34%	[13]
Sono-Fenton Method	Wastewater from textile industry	Sonication process: 10,30,60, and 90 minutes Variation of H ₂ O ₂ and FeSO ₄ : concentration 1:1, 2:1, 4:1, 8:2	Color Concentration Reduction: 99.61%, Ph: 68.94%, COD: 90.64%, BOD: 97.48%, TSS: 91.28%.	[44]

Table 3. Textile wastewater quality standards

Parameter	Highest Level (mg/L)	Highest Pollution Load (kg/ton)
BOD	60	6
COD	150	15
TSS	50	5
Total phenol	0.5	0.05
Total chromium	1	0.1
Total ammonia	8	0.8
Sulfide	0.3	0.03
Oil and fat	3	0.3
pH	6.0-9.0	
Highest waste discharge	100 m3/ton textile products	

Source: [41]

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5. Advantages and Disadvantages of Textile Wastewater Processing Methods

5.1. Electrocoagulation

The advantages of electrocoagulation method are ease of operation, absence of odor, and flocs formation like chemical coagulation. However, the method has disadvantages including the inability to treat wastewater with high electrolyte content due to short circuits between electrodes. It is also not effective in reducing heavy metal content, requires electricity consumption for processing, and small contact area of wastewater. Electrodes used for electrocoagulation should be replaced regularly because the formation of a layer on electrodes can affect the processing efficiency [17].

5.2. Electrooxidation-Electrocoagulation

Electrooxidation-electrocoagulation method is pollutant-free because electrooxidation process produces water and carbon dioxide. However, this process uses chemicals and electricity which generate new waste and consume a significant amount of energy during processing [20].

5.3. Filtration

Filtration method filters suspended solid pollutants/ types of waste with macro particles conventionally. However, the process uses chemicals and is less environmentally friendly [45].

5.4. Hybrid Method of Filtration System

Hybrid method uses bottom ash raw materials containing minerals in the form of silica (SiO_2) and alumina (Al_2O_3) which can adsorb pollutant waste. However, this method has disadvantages, namely, long contact time to obtain effective pollutant

reduction and high waste concentrations can affect performance [46].

5.5. Adsorption

Adsorption uses TiO_2 -volcanic ash photocatalyst absorbent but is not effective in removing pollutant parameters in complex wastewater conditions. After use, the adsorbent needs to be regenerated or washed. This method requires quite expensive costs. The adsorbed pollutants are still accumulated in the adsorbent which can cause a new problem [47].

5.6. Sono-fenton

Sono-Fenton is an effective method to remove many pollutants in textile wastewater. However, this method uses chemicals, reagent consumption costs, high process costs, and elevated amounts of iron sludge produced at the neutralization stage of the treated solution before being discharged [48].

5.7. Membrane Technology

Membrane technology is a method of processing textile wastewater that is a biopolymer. Some of the advantages include absence of large equipment, low energy, simple membrane module design, and ease of operation. This method does not require additional chemicals such as coagulants or flocculants compared to conventional water treatment processes. Membrane technology does not use high temperatures and is a non-destructive, sterile process, making it environmentally friendly. The membrane materials vary, which allows easy adaptation according to needs, hybrid processing, and simple scale-up [49],[50]. In the processing of textile wastewater, membrane technology causes a reduction in COD, BOD, and color levels at environmental quality standards [13]. The disadvantage of this method is that the

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flux and selectivity are inversely proportional [51].

6. Membrane

Membrane is a biopolymer derived from membrane technology processes. It is thin and semi-permeable in the form of a thin layer that can separate two phases by holding certain components and transporting others through the pores. Additionally, membrane can be widely used in various separation processes, such as in leather, pulp and paper, food (one of which is the dairy industry), seawater desalination, and drinking water, particularly in textile industry.

6.1. Types of Membrane

In the manufacture of membrane, several processes with filtration principles are widely developed. The processes in membrane technology can be classified based on the pressure difference, namely:

1. Microfiltration (MF) is filtration process that uses a porous membrane to separate airborne particles with a

diameter between 0.1 and 10 μm [52, 53]

2. Ultrafiltration (UF) is a variation of membrane filtration where the fluid is forced through a semipermeable membrane by hydrostatic pressure [13, 54].

3. Nanofiltration (NF) is a relatively new membrane filtration process commonly used in waters with low total dissolved solids. This type of membrane can be used for surface and groundwater processes, as well as to soften and remove natural and synthetic organics. [65].

The application of membrane in the processing of wastewater in textile industry will continue to develop. Based on the material, membrane are divided into organic or natural such as cellulose, synthetic, and inorganic [55]. Table 4 shows the raw materials and type of membrane produced with membrane technology.

Table 4. Raw materials for membrane production and type of membrane produced

Raw material	Type of membrane produced	Ref
Polytetrafluoroethylene	PTFE (polytetrafluoroethylene) Membrane	[56]
Polyethersulfone	PES (polyethersulfone) Membrane	[57]
Polyvinylidene Fluoride	PVDF (polyvinylidene fluoride) Membrane	[13]
PVDF (polyvinylidene fluoride)-Zeolite Composite	PVDF (polyvinylidene fluoride) Membrane	[4]
Oil palm empty fruit bunches	Cellulose Acetate Membrane	[58]
Pineapple crown fiber	Cellulose Acetate Membrane	[59]
Polysulfone	PSF (polysulfone) Membrane	[60]
Fermentation of <i>Acetobacter xylinum</i> bacteria	Bacterial cellulose membrane	[18]
Synthesis of bacterial cellulose from coconut and sugar using <i>Acetobacter xylinum</i> bacteria with ZnO nanoparticles	Bacterial cellulose membrane	[61]
Cellulose triacetate	Cellulose triacetate membrane	[15]
Cellulose acetate propionate	Cellulose acetate propionate membrane	[62]

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The application of membrane technology is continuously increasing, such as the production of high-quality water and the removal or recovery of toxic/useful substances from various industrial wastes, particularly in the processing of textile wastewater [63]. This has also led to the development and application of membrane in the processing of textile wastewater, such as PTFE, PVDF (polyvinylidene fluoride), CA (cellulose acetate), PES, CAP (cellulose acetate propionate).

6.2. Analysis of Membrane

In testing or analyzing membrane, there are several examples of tests or analyses that are commonly carried out, namely:

1. Morphological and functional group tests are carried out on the membrane including SEM and FTIR analysis [14],[64],[65].
2. Membrane performance tests include pure water flux, rejection, and porosity [51].
3. Mechanical test is in the form of membrane tensile strength test [66],[67].

7. Textile Wastewater Treatment Using Membrane Technology

The processing method using membrane technology applies a biopolymer-based membrane. This technology is considered efficient for wastewater treatment, particularly in textile industry [17]. Wastewater that enters through the membrane with contaminants larger than the membrane pore size will be retained, hence, the water that passes through is cleaner. Common terms used in this process include feed, permeate, and retained water [12].

The process of textile wastewater treatment using membrane technology depends on the physical properties of membrane. These include hydraulic permeability and thickness, pressure through the membrane, filtration time, as well as feed concentration, along with the various polymer materials. Table 5 shows the types of membrane and their effectiveness in textile dye wastewater treatment.

Based on Table 5, PTFE membrane can reduce dye pollutants by more than 97% in textile wastewater. PES is capable of removing dye pollutants in textile wastewater by 82%. Polysulfone-Polyvinyl Pyrrolidone Blend Polymer Composite Membrane can reduce 85.73% of dye pollutants in textile industry washing wastewater. CA membrane reduces dye pollutant levels by 81.77% in remazol red textile wastewater. Cellulose Membrane of *Sargassum Sp.* shows the potential to remove dyes with rhodamine B, methylene blue, and methylene orange types by 80.04%, 77.83%, and 75.84%, respectively. PSF membrane can reduce color pollutants in textile wastewater by 85.7%. Bacterial cellulose membrane can reduce the pollutant dye type black remazol in textile wastewater by 32.10%. Furthermore, CAP membrane reduces the pollutant dye type blue remazol in textile wastewater by 43%.

8. Conclusion

In conclusion, this review showed that membrane technology reduced pollutants by approximately 97% in textile wastewater. Compared to other methods, membrane technology offered numerous advantages such as being biopolymer, requiring less equipment, low energy, and easy to operate. Furthermore, it did not

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require chemicals during processing and could be operated at temperature, serving as non-destructive, sterile technology. Several types of membrane that were identified included PTFE, PES,

Polysulfone-Polyvinyl Pyrrolidone Blend Polymer Composite Membrane, CA, Cellulose Membrane of *Sargassum Sp.*, PSF, Bacterial Cellulose Membrane, and CAP.

Table 5. Types of membrane and their effectiveness in treating textile dye wastewater

Membrane Type	Types of Dye Waste	Color reduction results	Ref
PTFE (Polytetrafluoroethylene)	Textile Wastewater	Color loss >97%	[56]
PES (Polyethersulfone)	Textile Wastewater	Color loss 82%	[57]
Polysulfone-Polyvinyl Pyrrolidone Blend Polymer Composite Membrane	Textile Industry Washing Waste	Color loss 85.73%	[60]
CA (Cellulose Acetate)	Textile Wastewater (Remazol Red)	Color loss 81.77%	[58]
Cellulose Membrane of <i>Sargassum Sp.</i>	Textile Wastewater (Rhodamine B)	Color loss 80.04%	[18]
	Textile Wastewater (Methylene Blue)	Color loss 77.83%	[18]
	Textile Wastewater (Methylene Orange)	Color loss 75.84%.	[18]
	Textile Wastewater	Color loss 85.7%	[60]
Polysulfone (PSF)	Textile Wastewater (Black Remazol)	Color loss 32.10%.	[61]
Bacterial Cellulose Membrane	Textile Wastewater (Blue Remazol)	Color loss 43%	[14]

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Reducing Silica Levels in WTP PLTGU X Wastewater using Rice Husk Filter Membranes

Penurunan Kadar Silika pada Air Limbah WTP PLTGU X Menggunakan Membran Filter Sekam Padi

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Abstract

During the water demineralization process, silica content is increased due to the failure of the chemical reagent. This research aimed to determine the effectiveness of silica reduction in the Water Treatment Plant (WTP) of PLTGU X by observing the permeate flux values relative to time and operating pressure variables, using ceramic membrane made from rice husk. To achieve the objective, ceramic membrane was made from rice husk additives, with a pore diameter of 365 nm and a surface area of 25 cm². The results showed that the composition ratio of clay, rice husk, and iron powder was 82.5%, 15%, and 2.5%, respectively. Furthermore, ceramic membrane with rice husk additives successfully reduced silica content from 1250 ppb to 890 ppb at a pressure of 1.5 bar and 90 minutes of operation and from 1250 ppb to 710 ppb at 2 bar and 90 minutes of operation. This suggested that wastewater could be processed again in the demineralization plant to produce demineralized water. The best membrane performance in the filtration process was achieved at 90 minutes with a pressure of 2 bar, which successfully reduced silica content by 43.2%, with a permeate flux of 3.44 L/m².

Keywords: filter; membrane; rice husk; silica

Abstrak

Tujuan penelitian ini yaitu untuk mengetahui efektifitas penurunan silika pada air limbah PLTGU X dilihat dari nilai fluks permeat terhadap variabel waktu dan tekanan operasi dengan menggunakan membran keramik dari sekam padi. Metode penelitian yang digunakan yaitu dengan membuat membran keramik berbahan aditif sekam padi dengan diameter pori 365 nm dan luas permukaan 25 cm² dengan perbandingan komposisi tanah liat, sekam padi dan serbuk besi terdiri dari; 82.5%; 15%; 2.5%. Membran keramik dengan aditif sekam padi berhasil menurunkan kadar silika dari 1250 ppb menjadi 890 ppb pada tekanan 1,5 bar dengan waktu 90 menit dan dari 1250 ppb menjadi 710 ppb pada tekanan 2 bar dengan waktu 90 menit dan air limbah tersebut dapat diproses kembali di demin plant untuk menjadi air demineralisasi. Kinerja membran terbaik dalam proses filtrasi yaitu pada waktu 90 menit dengan tekanan 2 bar yang dapat menurunkan kadar silika 43,2 % dengan fluks permeat sebesar 3,44 L/m².

Kata Kunci: filter; membran; sekam padi; silika

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1. Introduction

The working principle of membrane is to act as a thin and highly selective barrier that separates two or more components of a fluid flow. In this context, the flow in membrane occurs due to the driving force, which can be the convection or diffusion of each molecule [1][2]. This has led to a significant increase in the use of porous ceramic membrane, suggesting potential opportunity for natural materials [3]. Ceramic membrane is usually made from clay, which has a high porosity level with a composition of hydrous aluminum silicate ($\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 \cdot 3\text{H}_2\text{O}$) and metal oxides such as Fe_2O_3 , MgO , and K_2O , as well as ions that can be exchanged with other ions [4]. In general, membrane technology is used to separate solutes from solvents, for example, separating silica content in water.

Water containing high levels of silica is considered unfit for use in the PLTGU (Gas and Steam Power Plant) process. This is due to the production of silica crust that causes blockages in the steam pipe to the turbine. Therefore, silica content in boiler water should be controlled by using filter membrane.

Generally, synthetic membrane is used in industries such as polyamide, polysulfone, and polycarbonate [5][6]. However, there is natural membrane found in living cells, such as cellulose and its derivatives, including cellulose acetate. Previous research has proven that cellulose acetate membrane from nutmeg seed shells using the phase inversion method produces characteristic tests that can be applied further [7]. This derivative is obtained from Nata de Coco with the addition of polyethylene glycol additives, which meet the requirements as ultrafiltration membrane. [8] There is also a derivative

from banana stems and water hyacinth that can be used in the purification of drilled well water [9].

As an effective method, PLTGU X currently uses silica filter membrane made from empty oil palm bunch waste. According to Bramanta, oil palm fiber and shell ash have many minerals, one of which is silica at 27.5% and 36.1% [10]. The research by Laelasari stated that rice husk ash had silica content of approximately 16.98% [11]. Silica ash can be produced from the controlled burning of rice husk at high temperatures above 650°C . The weight of rice husk is approximately 20% of the weight of the grain, with 15% as ash. With a high silica content, it shows that the performance of membrane made from rice husk additives is quite good [12].

The water filtration process usually uses zeolite and activated carbon compounds. Silica (SiO_4) and Alumina (AlO_4) are the main minerals in the zeolite compounds that have cavities. After modifying by adding clay, white Portland cement, and PVA, membrane has advantages in the filtration process because of high selectivity to Mn ions [13]. Additionally, natural zeolite is also easy to obtain with selective filtration capabilities and molecular-sized pores [14]. In this context, rice husk is very likely to be used as an alternative material for processing waste from various industries because of silica content [15].

Based on the background, this research aimed to determine the effectiveness of silica reduction in PLTGU X wastewater from the permeate flux value against time and operating pressure variables, using ceramic membrane from rice husk. To achieve the objective, zeolite was combined with rice husk containing

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silica to improve the performance of ceramic membrane in removing silica levels in PLTGU X wastewater. The effectiveness of this method was determined by knowing the reduction in silica levels using a filter membrane from rice husk in PLTGU X wastewater to be reprocessed into deionized water at the PLTGU X. The results were expected to maximize the use of abundant rice husk waste, specifically in agricultural areas such as South Sumatra.

2. Research Methods

2.1. Tools and Materials

The materials used in this research consisted of rice husk, clay, zeolite, iron powder, ammonium molybdate solution, oxalic acid solution, hydrochloric acid solution, ascorbic acid solution, and distilled water. Meanwhile, the instrumentation tools included Scanning Electron Microscopy (SEM), FTIR Spectrometer, and UV-Vis Spectrophotometry. The sample used was PLTGU X wastewater.

2.2. Data Collection Methods

The data obtained were sourced from literature related to the research conducted, both from the company and outside, such as books, journals, and websites. Besides the literature review, the data listed were also obtained directly from the PLTGU X Laboratory, discussion, and Q&A activities, whose sources were obtained from PLTGU X employees.

2.3. Making Ceramic Membrane

In this research, ceramic membrane made was cylindrical, including the printing and sintering processes. Membrane was made with two variations of the mixture composition where the first

was from clay, rice husk additives, zeolite, and iron powder with a composition ratio of 82.5%, 7.5%, 7.5%, and 2.5%, respectively. The second was made from clay, zeolite, and iron powder with a composition ratio of 82.5%, 15%, and 2.5%, respectively, printed using a cylindrical mold made of stainless steel and compacted for 15 minutes. This treatment was necessary to ensure the pressure given could be evenly distributed on membrane and sintered at a temperature of 500°C.

Ceramic membrane was used for wastewater treatment by varying the feed flow rate at a pressure difference (ΔP) of 1.5 and 2 bar. Observations were made at different operating times ranging from 15, 30, 45, 60, 75, and 90 minutes.

2.4. Silica Analysis

Silica analysis was carried out using UV-Vis Spectrophotometry. A total of 2 mL of 7.5% (w/v) ammonium molybdate solution and 1 mL of 1:1 HCl were added to 2 mL of sample and 50 mL of distilled water. Furthermore, 15 ppm of orthophosphate was added to the boiler feed. Ammonium molybdate in acidic conditions was reacted with silica and orthophosphate to produce heteropoly acid. The resulting molybdosilicic acid was then reduced with 1 mL of 10% (w/v) ascorbic acid to make a blue complex compound whose absorbance was measured at a wavelength of 660 nm. A 2 mL of 10% (w/v) oxalic acid solution was added to reduce molybdophosphoric acid without removing molybdosilicic acid. Moreover, it should be observed that reactive silica removed all silicic acid polymers ($\text{SiO}_2 \cdot \text{H}_2\text{O}$).

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2.5. Permeate Flux Analysis

Permeate flux usually showed whether membrane was performing optimally. Additionally, membrane performance was determined based on the amount of permeability, selectivity to certain chemical compounds, and the rejection percentage of unwanted compounds in the feed. The permeate flux value was obtained by Equation 1.

$$J_v = \frac{V}{A \times t} \dots\dots\dots(1)$$

J_v represented the permeate flux in $L/m^2 \cdot \text{hour}$; V portrayed the permeate volume in Liters; A showed membrane surface area in m^2 , and t indicated the time in hours.

2.6. Ceramic Membrane Filtration Test

Wastewater was fed into a 500 L storage tank. This test used a 100 L sample of PLTGU X blowdown wastewater. With the help of a pressure pump, wastewater from the storage tank was flowed into housing-1 containing a sponge filter with a pore diameter of $0.5 \mu m$. The pump pressure was set at 1.5 and 2 bar by regulating the feed flow rate using a feed flowmeter. Subsequently, water filtered with a $0.5 \mu m$ pore diameter sponge filter was flowed into housing-2 and 3, each

containing a sponge filter with a pore diameter of $0.1 \mu m$ and activated carbon.

As shown in Figure 1, wastewater passed through the sponge filter, and activated carbon was returned to housing 4, which contains rice husk ceramic membrane. Subsequently, wastewater that had passed through membrane process was collected in a container as permeate. Sampling was carried out every 15, 30, 45, 60, 75, and 90 minutes. Each sample was calculated for the volume of permeate produced and analyzed for silica content in the permeate. The testing process is shown in Figure 1.

3. Results and Discussion

3.1. Membrane Performance Analysis

3.1.1. Permeate Flux Analysis

Membrane performance is better when the permeability value and selectivity level of membrane are greater. Sylvani et al. stated that the number of membrane pores affected the permeate flux value and water permeability [16]. When water flows easily through membrane, the permeability value is higher. Meanwhile, thicker membrane makes it difficult for water to pass through, reducing the permeate flux value.

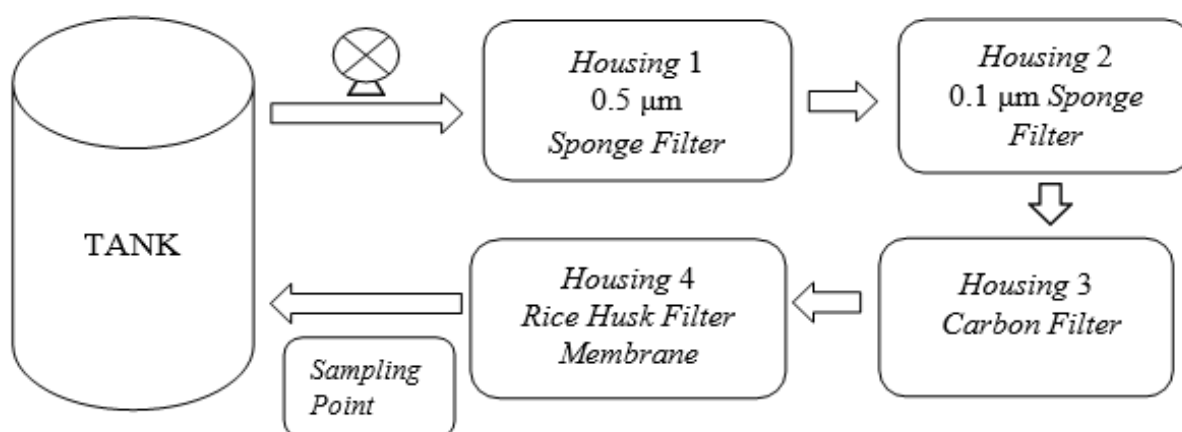


Figure 1. Membrane Filtration Testing Flow Chart

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In this research, the best membrane permeate flux value for PLTGU X wastewater was obtained at a pressure of 2 bar with rice husk additives at the 15th minute of 18.08 L/m². Meanwhile, membrane without additives was obtained at the 15th minute with a pressure of 1.5 bar, which was only 15.68 L/m². This is due to the influence of pressure and time passed by membrane, which has not reached the saturation point. According to Diana et al., the lower permeate flux results are due to differences in the compounds in ceramic membrane, affecting the pores on membrane surface [17].

From Tables 2 and 3, longer time makes more impurities settle on membrane surface, which causes the permeate flux value to decrease. Meanwhile, the effect of pressure shows that the permeate flux increases with higher operating pressure. This occurs because of the influence of the main driving force of membrane operation. When pressure is applied, particles with a size smaller than the pores will easily pass through membrane pores. Meanwhile, larger particles, such as contaminants, will remain in it. As a result of the applied driving force, the pores can also enlarge, which causes membrane pores to become larger. Therefore, the solution feed rate is faster and passes through membrane.

3.1.2. Analysis of Silica in Liquid Waste of PLTGU X

Silica can cause fouling in boiler pipe parts when carried by water or hot steam at high temperature and pressure. When this happens, the boiler's heat transfer capacity will decrease, causing uneven heat transfer. Silica dissolved in hot steam will settle on the turbine fins at low temperatures. As shown in Table 1, it

can be observed that the use of ceramic membrane made from rice husk additives is capable of reducing silica values. Based on the results of laboratory tests, the best reduction in silica levels was obtained in silica filter membrane made from rice husk additives, which were able to reduce by 43.2% from 1250 ppb to 710 ppb within 90 minutes at a pressure of 2 bar. Meanwhile, for membrane without rice husk additives, it was only able to reduce silica levels by 23.68% or 296 ppb.

Table 1. Silica Level Reduction Data

Time (Min)	1.5 Bar		2 Bar	
	With Rice Husk (ppb)	Without Rice Husk (ppb)	With Rice Husk (ppb)	Without Rice Husk (ppb)
0	1250	1250	1250	1250
15	1120	1241	1100	1220
30	1050	1233	950	1210
45	980	1190	895	1130
60	920	1150	800	1000
75	900	1170	760	993
90	890	1123	710	954

Table 2. Ceramic Membrane Permeate Flux Value at 1.5 Bar Pressure

Time (Min)	Permeate Volume		Permeate Flux	
	With Rice Husk (L)	Without Rice Husk (L)	With Rice Husk (L/m ²)	Without Rice Husk (L/m ²)
15	0.0105	0.0098	16,80	15,68
30	0.0109	0.0095	8,72	7,60
45	0.0109	0.0098	5,81	5,23
60	0.0105	0.0095	4,20	3,80
75	0.0109	0.0098	3,49	3,14
90	0.0105	0.0098	2,80	2,61

Table 3. Ceramic Membrane Permeate Flux Value at 2 Bar Pressure

Time (Min)	Permeate Volume		Permeate Flux	
	With Rice Husk (L)	Without Rice Husk (L)	With Rice Husk (L/m ²)	Without Rice Husk (L/m ²)
15	0.0113	0.0080	18,08	12,80
30	0.0105	0.0081	8,40	6,48
45	0.0125	0.0083	6,67	4,43
60	0.0136	0.0085	5,44	3,40
75	0.0129	0.0087	4,13	2,78
90	0.0129	0.0085	3,44	2,27

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Based on the results, membrane without rice husk additives were observed to be less effective in reducing silica levels. From Table 1, there are non-linear results at minute 75 at conditions 1.5. This is because there is seepage in the column, which causes PLTGU X wastewater not to pass through membrane despite the effectiveness. The operating pressure conditions also affect silica levels, as 2 bar has greater effect than 1.5 bar. This is possibly due to the influence of pressure, which enlarges membrane pores, causing a high potential for impurity particles to escape from membrane surface.

The adsorption process using rice husk membrane containing silica content greatly affects the reduction of silica. During this process, the phenomenon of concentration polarization occurs due to blockage of membrane pores by silica. This causes silica concentration on membrane surface to be higher than concentration passing through membrane. The polarization occurs as a result of the diffusion of substances moving towards the surface or through membrane, which causes the accumulation of substances on the surface. In areas farther from membrane surface, the concentration of substances remains lower. Additionally, a dialysis process occurs, namely the transfer of silica molecules from the solution due to diffusion through membrane.

3.1.3. SEM Analysis on membrane Surface

Photomicrographs of ceramic membrane with a composition of clay, zeolite, and iron powder are shown in Figure 2. Meanwhile, ceramic membrane with a composition of clay, rice husk additives, zeolite, and iron powder is shown in Figure 3.

The morphology between ceramic membrane with and without rice husk additive shows significant difference. The use of rice husk additives produces membrane surface pores that are dense and evenly distributed. Although ceramic membrane with rice husk has a random and non-uniform pore structure and different grain boundary sizes and ratios, it is better than sample without additives. The more pores formed from the addition of rice husks, the more pollutant material or particles are expected to be blocked on the membrane surface.

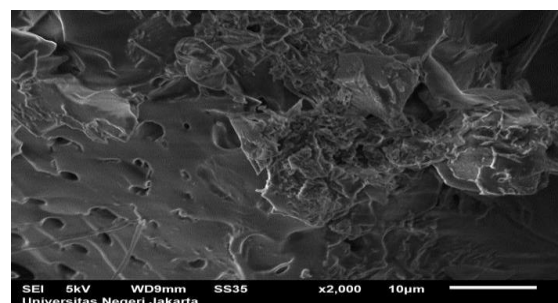


Figure 2. SEM Image of Ceramic Membrane without Rice Husk Additive at 2000x Magnification

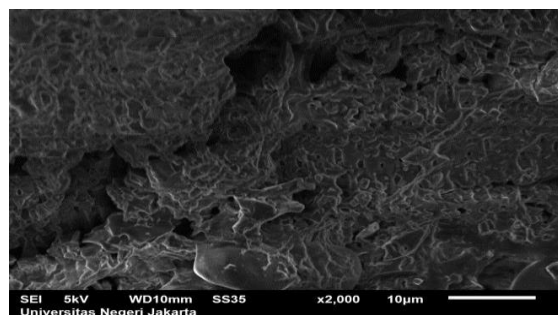


Figure 3. SEM Image of Ceramic Membrane with Rice Husk Additive at 2000x Magnification

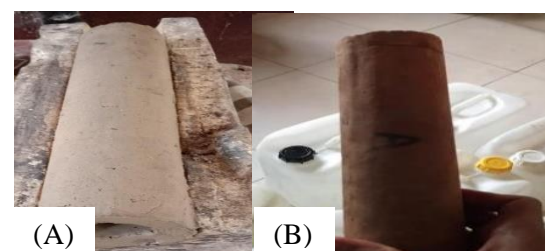


Figure 4. Ceramic Membrane (A) with Rice Husk Additives, (B) without Rice Husk Additive

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Based on the ceramic membrane images in Figures 4, the addition of rice husk additives makes membrane color more inclined to that of rice husk. Meanwhile, membrane without rice husk tends to resemble clay. On both membrane, the distribution of iron powder can also be observed with visual black spots on membrane surface.

The FTIR results (Figure 5) of rice husk show that the ceramic membrane with rice husk additives contains H stretching vibrations in the form of -OH and NH in the wave range of 3417.67 with an intensity of 92.02%. This wide peak shows the presence of OH groups originating from carbohydrate compounds that are

components of rice husk. The fingerprint area shows inorganic compounds -C-NO₂- in the wave range of 1352 with an intensity of 87.59% [18]. These organic compounds may be formed from membrane combustion process at high temperatures.

4. Conclusion

In conclusion, the effectiveness of reducing silica content in PLTGU X wastewater with silica filter membrane made from rice husk additives showed an optimal result at 90 minutes with a pressure of 2 bar. Silica content was reduced by 43.2% with a permeate flux of 3,44 L/m².

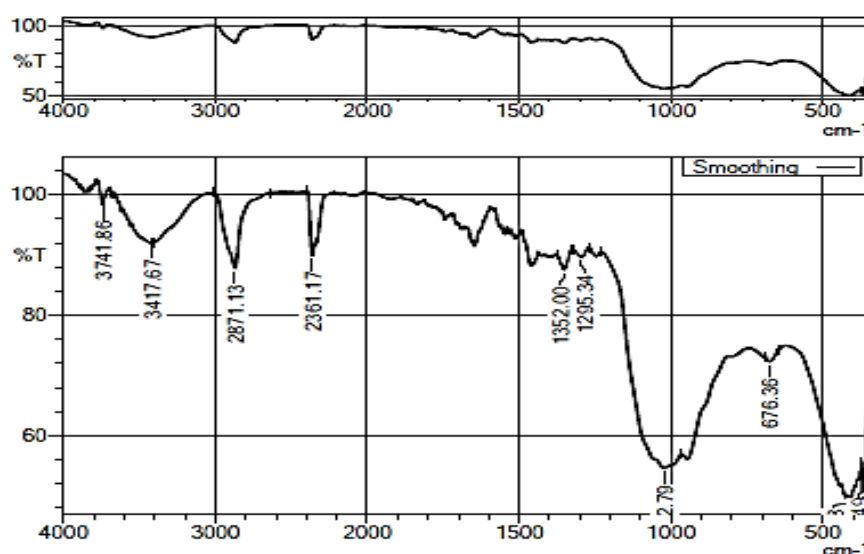


Figure 5. FTIR Analysis of Rice Husk

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