ISSN 2355-8776

Effect of Temperature and Time on Alkaline Pretreatment and Alkaline Microwave-Assisted Pretreatment on Banana Stem Composition

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ARTICLE INFO

Article history
Received October 04, 2023
Revised November 04, 2023
Accepted November 05, 2023

Keywords
Alkaline
Banana Stem
Cellulose
Lignin
Microwave-Assisted

ABSTRACT

Banana stem waste is a source of lignocellulose biomass with a high cellulose content and abundant availability in Indonesia. In this work, we investigated the effect of time and temperature on the decrease in the rate of lignin in lignocellulose. Banana stem waste was pretreated with alkaline and alkaline microwave-assisted so that the percentage of lignin contained in lignocellulose biomass was reduced and the percentage of cellulose was increased. In alkaline pretreatment, 6% KOH is added to lignocellulose and heated to a hotplate during contact time variation (10, 20, 30, 40, and 50 min) with temperature variations (55, 65, 75, 85, and 95°C). In pretreatment, an alkaline microwave–assisted 6% KOH solution was added to lignocellulose and heated for 20 min with temperature variations (55, 65, 75, 85, and 95°C), then was put into a microwave that has a wave power of 360 Hz with variations in contact time (55, 65, 75, 85 and 95 min). After pretreatment, the sample was analyzed using the Chesson method to determine the percentage of cellulose, hemicellulose, and lignin. Analysis showed that alkaline and microwave-assisted alkaline pretreatments effectively reduce the lignin percentage and increase the cellulose percentage in lignocellulose. The most remarkable performance in both pretreatments was obtained when working at 95°C with a contact time of 50 min. Based on process optimization, it was concluded that microwave-assisted alkaline pretreatment reduced the percentage of lignin more and increased the percentage of cellulose compared to alkaline KOH pretreatment, which decreased the rate of lignin by 43.26% and increased the rate of cellulose by 60.68%. For further research, it can be continued to the next process, namely hydrolysis to produce glucose as a raw material for the bioethanol production.

1. Introduction

South Sumatra is one of the largest banana-producing provinces in Indonesia. Banana stems have cellulose content of 50-60%, hemicellulose of 20% and lignin of 5%, so that banana stems can be used as a source of lignocellulose biomass [1]. According to the Central Bureau of Statistics and the Directorate General of Horticulture, in 2015-2019, the harvest area of banana plants in Indonesia reached 105,801 ha [2]. In addition, based on data from the Central Statistics Agency in 2021, banana plant production in Indonesia came to 8,741,147 tons, while South Sumatra Province itself got 354,143 tons, which is the 6th province that produces the most banana plants in Indonesia [3]. The large area of banana crop harvest and production causes an abundance of banana stem waste, so it has excellent potential to be used as raw material for various processes, one of which is the delignification process [4]. Delignification is decreasing/removing lignin levels because it can block cellulose degradation carried out by microbes or chemicals [5].

The pretreatment method (delignification) can be done both conventionally and unconventionally. One of the unconventional pretreatments is using a microwave. Pretreatment with microwave and alkaline combination using various raw materials can result in higher glucose content and more significant lignin removal from alkaline solutions [6]. Compared to conventional procedures, chemical reactions carried out in microwaves are usually faster, more economical, and more environmentally friendly [7].

Several previous studies reported on pretreatment methods (delignification) to lower lignin levels and raise cellulose levels. Previous research examined microwave-alkali pretreatment of Tetraselmis suecica biomass (microalgae). Their study found that the highest sugar concentration was 9.81 mg/mL with the microwave-assisted alkaline (MAK) method and 9.98 mg/mL with the AK (alkaline) method, which was processed for 72 hours. Although the sugar concentrations obtained from MAK and AK were comparable, it was found that AK pretreatment took significantly longer (±120 minutes) to get conversion results similar to MAK pretreatment [8]. On the other hand, previous studies also examined the effect of temperature and alkaline pretreatment time on cellulose isolation of banana stem waste with temperature variations of 60, 70, and 80°C and variations in cooking time of 60, 90, and 120 min. The highest cellulose content was 51.64% at the highest pretreatment temperature of 80°C and the shortest cooking time of 60 min. The result suggested that the length of pretreatment time can cause an increase in cellulose levels as more hemicellulose and lignin chains are broken. Still, if the pretreatment time is too long, it can cause the severed monomers to react with existing polymers to produce new lignin chains [9], [10]. Furthermore, previous research also investigated the delignification of corn weevils with the alkaline microwave method. According to their study, cellulose levels were reduced in corn weevils in treatment with variations in NaOH 2N concentrations and variations in the length of microwave exposure for 40 minutes, which was 69.937% [6].

Based on the explanation above, the pretreatment process (delignification) dramatically affects the amount of lignin level reduction and the increase in cellulose levels produced. Very few studies have used combined pretreatment between KOH and microwave to reduce lignin content in banana stems. Therefore, the research contribution is on the delignification of banana stems using the microwave-assisted alkaline pretreatment method. Banana stem waste will be more optimally utilized to reduce environmental pollution.

2. Research Methodology

2.1. Materials

Banana stems are obtained from banana plantations in Lahat Regency, South Sumatra Province, Indonesia. Potassium Hydroxide (KOH) (>85%) Pro Analysis is purchased from the brands E. Merck, Germany and Sulphuric Acid (H_2SO_4) (98%) Pro Analysis purchased search brand SmartLab. All chemicals are used without further processing.

2.2. Procedures

1) Preparation of Raw Materials

Banana stems are cut with a knife to a small size of about 5 cm and dried using an oven at 80°C for 2-3 days [11]. The dried banana stems are then mashed using a grinder until close to smooth (size: 30 mesh). The Chesson method tested Fine banana stems to determine the composition of lignin, hemicellulose and cellulose before the pretreatment process [12].

2) Pretreatment

The alkaline pretreatment and the alkaline microwave-assisted pretreatment were carried out. The alkaline pretreatment uses KOH with a concentration of 6% at temperature variations (55, 65, 75, 85 and 95°C) and reaction contact time variations (10, 20, 30, 40, and 50 min) with a solid to liquid ratio of 1:10. The pretreatment process was carried out by adding 75 g of samples with 750 mL of KOH solution (6% w/v). In alkaline pretreatment, the biomass and KOH solution were mixed in a 1 L beaker and stirred at (55-95°C) for (10-50 min). Then, the sample was neutralized by aquadest, filtered and dried in the oven at 100-110°C temperature. The sample is stored and then analyzed by the Chesson method.

In the microwave-assisted alkaline pretreatment process, the process and KOH concentration variation are the same as the first method, with a reaction time of 20 minutes. The sample reacted with the KOH solution and was then put into a microwave with 360 Hz and time variations (10-50 min). Then, the slurry was neutralized by aquadest, filtered and dried in the oven at a temperature of 100-110°C. The sample was stored and then analyzed by the Chesson method. The pretreatment flow diagram in this study data can be seen in Fig. 1.

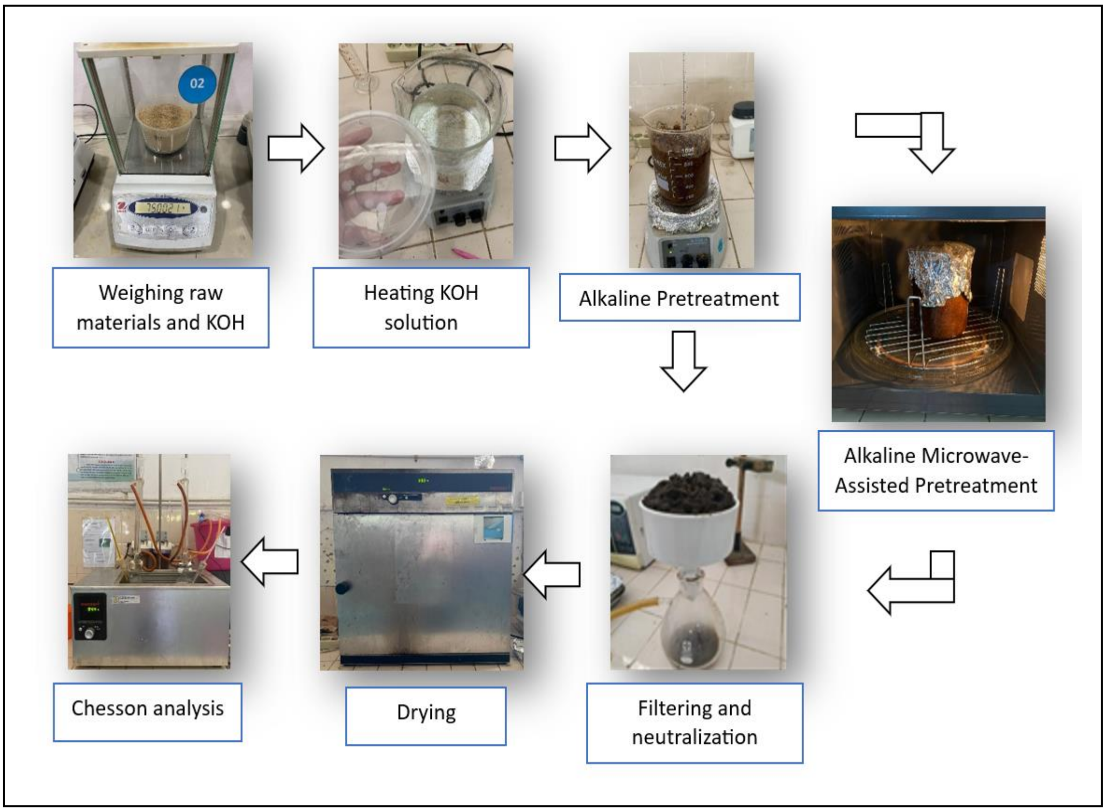

Fig. 1. Research Procedure Flow Diagram

3) Analysis of Banana Stem Composition with the Chesson Method (Datta, 1981)

1 g (a) of dry sample was refluxed for 1 h using 150 mL H_2O at 100°C, then dried using an oven and weighed mass (b). The sample was followed by a second reflux for 1 h using 150 mL of H_2SO_4 1 N at 100°C, and then the sample was filtered and neutralized with aquadest and dried and weighed by mass (c). Next, the sample was soaked with 10 mL of H_2SO_4 72% for 4 h at room temperature, and then 150 ml H_2SO_4 1 N and refluxed at 100°C for 1 h. Then, the sample was filtered, neutralized with aqua dest, dried, and weighed by mass (d). Finally, the sample was opened in a furnace with a temperature of 550°C for 1 h and then weighed the mass (e). To calculate the levels of lignin, cellulose, and hemicellulose (%) in the sample as follows:

$$\text{Hemicellulose} = (\text{b-c})/\text{a} \times 100\% \tag{1}$$

$$\text{Cellulose} = (\text{c-d})/\text{a} \times 100\% \tag{2}$$

$$\text{Lignin} = (\text{d-e})/\text{a} \times 100\% \tag{3}$$

where
a : ODW (Oven Dry Weight) initial sample of cellulose biomass.
b : ODW residue in reflux with hot water.
c : ODW sample residue after reflux with 1 N H_2SO_4
d : ODW sample residue after being treated with 72% H_2SO_4 and refluxed with 1 NH_2SO_4.
e : Ash from sample residue.

3. Results and Discussion

3.1. Effect of Time on KOH Alkaline Pretreatment on Banana Stem Composition

In alkaline pretreatment, KOH compounds were used because they are relatively affordable, use mild reaction conditions, alkaline chemicals can be recovered, have high selectivity for lignin separation and less react in degrading cellulose and hemicellulose [13], [14]. The percentage of lignin, cellulose and hemicellulose in banana stems before and after alkaline KOH pretreatment that has been analyzed using the Chesson method can be seen in Fig. 2.

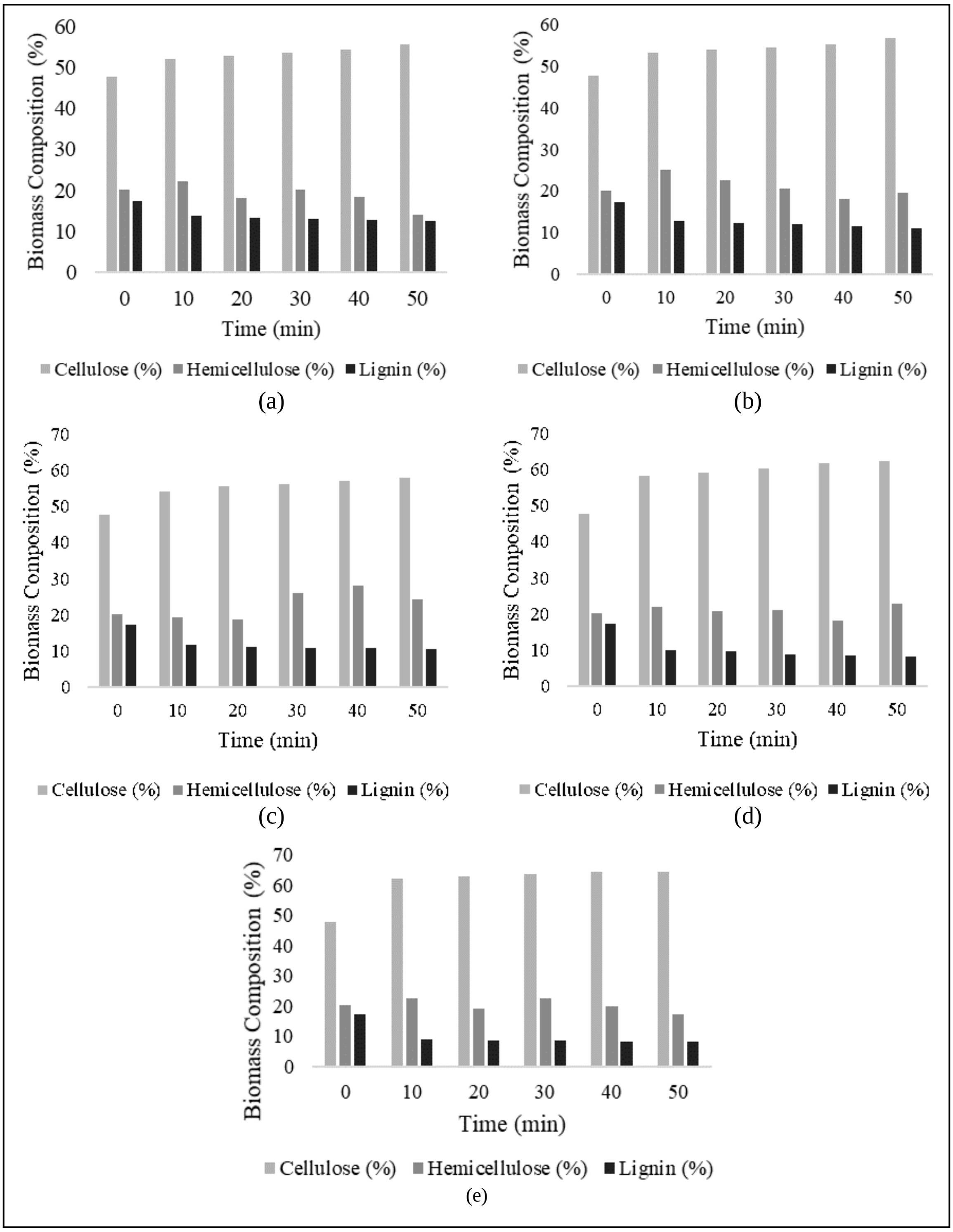

Fig. 2. Percentage of Biomass using 6% KOH Pretreatment (a) at 55°C, (b) at 65°C, (c) at 75°C, (d) at 85°C, (f) at 95°C

The percentage of banana stem composition before pretreatment is lignin by 17.40%, cellulose by 47.66%, and hemicellulose by 20.17%. The lowest lignin percentage and the highest cellulose percentage at (10, 20, 30, 40, and 50) minutes can be seen in Fig. 2., respectively (8.95; 8.62; 8.42; 8.26; and 8.13)% for the lowest lignin percentage and (62.01; 62.71; 63.73; 64.18; and 64.38)% for the highest percentage of cellulose obtained at 95°C. The longer contact time in pretreatment leads to a decrease in the percentage of lignin and a significant increase in the percentage of cellulose.

The composition of banana stems obtained from each increase in contact time during pretreatment shows a downward trend in lignin percentage and an upward trend in cellulose percentage in each temperature variation. The longer the delignification contact time used, the lower the percentage of lignin obtained, this is due to the amount of lignin that is degraded or lignin is lost with the length of reaction contact time [15]. This is of course also influenced by the concentration of KOH used, where this alkaline solution acts as a chemical bond-breaking agent and damages the lignin structure in the amorphous crystal and separates some hemicellulose so that it can change its structure and properties to be more soluble in water [16]. As the percentage of lignin decreases, the percentage of cellulose will actually increase. This is due to the breaking of cellulose and lignin bonds and the release of lignin into the water phase during pretreatment using KOH [11], [17]. Based on the results of the analysis, the most optimal KOH alkaline pretreatment operation conditions in reducing the percentage of lignin and increasing the percentage of cellulose are heating at a contact time of 50 minutes.

3.2. Effect of Temperature on Alkaline Pretreatment on Banana Stem Composition

In addition to time, alkaline pretreatment is also influenced by temperature. The overall percentage of the composition of banana stems after pretreatment can be seen in Fig. 2. Furthermore, the effect of temperature on lignin and cellulose levels at the optimum contact time of 50 minutes can be seen in Fig. 3.

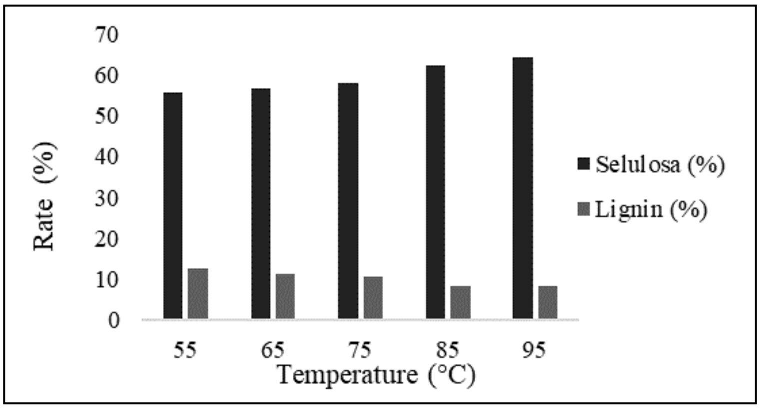

Fig. 3. Effect of Temperature on The Rate of Lignin and Cellulose during 50 Minutes of Alkaline Pretreatment

The percentage of banana stem composition before pretreatment is lignin by 17.40%, cellulose by 47.66%, and hemicellulose by 20.17%. The lowest lignin percentage and highest cellulose percentage at Temperature (55, 65, 75, 85, and 95)°C can be seen in Fig 7, i.e. (12.48; 11.04; 10.54; 8.15; 8.13)% for the lowest lignin percentage and (55.65; 56.59; 57.91; 62.37; 64.38)% for the highest cellulose percentage obtained at 50-min contact time. The increase in temperature in pretreatment causes a decrease in the percentage of lignin and a significant increase in the percentage of cellulose.

The increase in temperature during pretreatment shows a downward trend in lignin percentage and an upward trend in cellulose percentage in each variation in contact time. The higher the operating temperature used, the percentage of lignin contained in the sample will decrease, this is due to the amount of lignin that is degraded or lignin is lost as the temperature increases in addition to the breaking of cellulose and lignin bonds and the release of lignin into the water phase so that cellulose levels will increase [15]. The KOH solution used acts as a destroyer of the lignin structure in the amorphous part of the crystal and separates some of the hemicellulose so that it can change the structure and properties to become more soluble in air [16]. This can be shown by lignin decreases of (28.26; 36.54; 39.42; 53.15 and 53.26)% at each temperature variation with a contact time of 50 minutes. As the percentage of lignin decreases, the percentage of cellulose will actually increase. This is due to the breaking of cellulose and lignin bonds and the release of lignin into the water phase during pretreatment using KOH [11], [17].

Based on the results of the analysis, the most optimal KOH alkaline pretreatment operation conditions in reducing the percentage of lignin and increasing the percentage of cellulose are heating at a temperature of 95ºC with a contact time of 50 minutes.

3.3. Effect of Time on Alkaline Microwave-Assisted Pretreatment on Banana Stem Composition

In this study, the alkaline microwave–assisted pretreatment method is a combination of pretreatment methods that use KOH compounds and microwave tools. Pretreatment using a microwave is done because it produces irradiation, which helps reduce the process time and activation energy of pretreatment to accelerate the destruction of lignin structures [18]. The levels of lignin, cellulose and hemicellulose in banana stems before and after alkaline microwave-assisted pretreatment that have been analyzed using the Chesson method can be seen in Fig. 4.

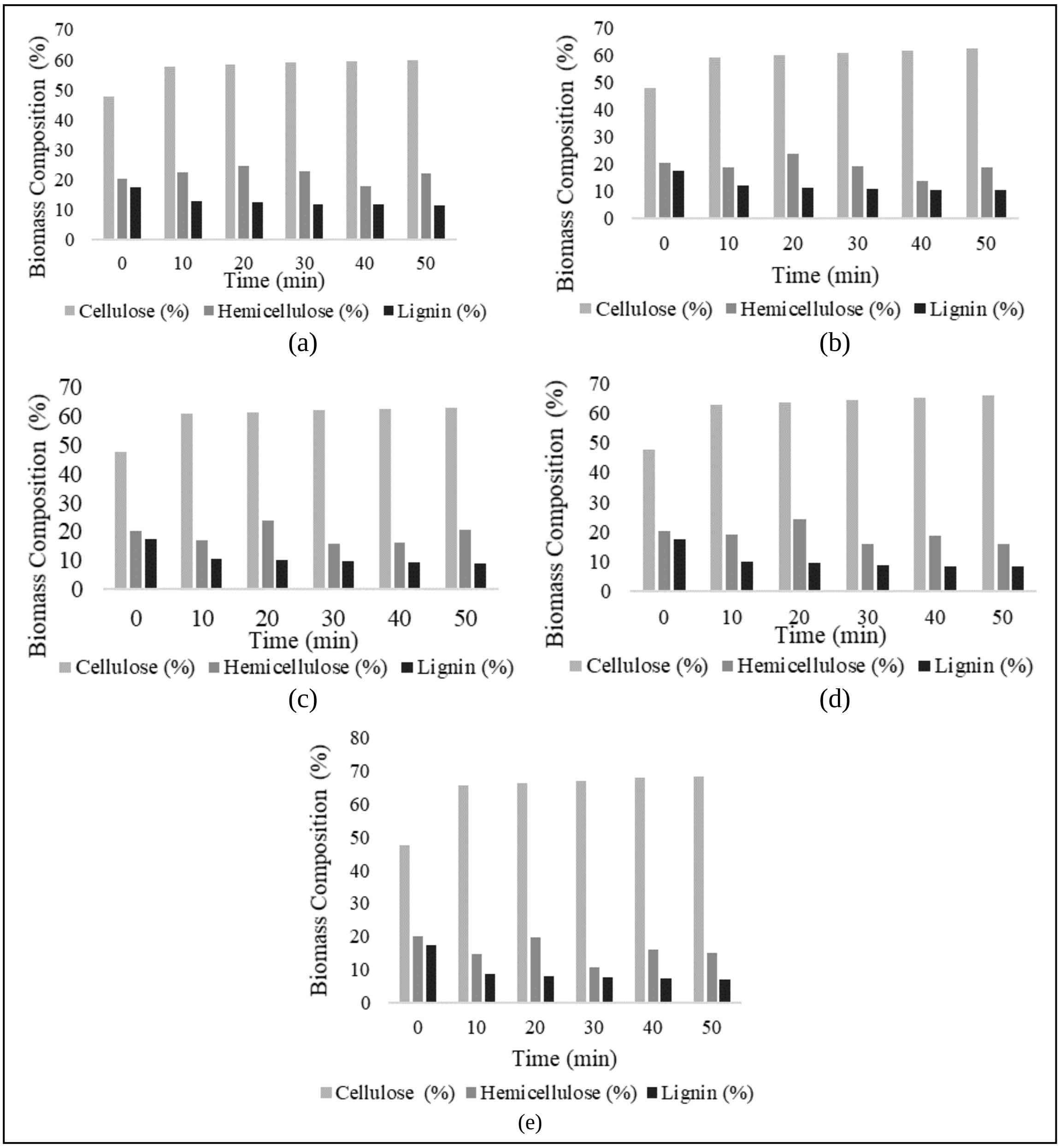

Fig. 4. Percentage of Biomass using Microwave – Assisted Alkaline Pretreatment (a) at 55°C, (f) at 95°C, (b) using Pretreatment Alkaline Microwave – Assist at 65°C, using Alkaline Microwave – Assisted Pretreatment (c) at 75°C, (d) at 85°C

The percentage of banana stem composition before pretreatment is lignin by 17.40%, cellulose by 47.66%, and hemicellulose by 20.17%. In this alkaline microwave-assisted pretreatment the lowest lignin percentages and the highest cellulose percentages at (10, 20, 30, 40, and 50) minutes can be seen in Fig. 4. i.e. (8.71; 8.02; 7.74; 7.18; and 6.84)% for the lowest lignin percentage and (65.57; 66.31; 67.05; 67.82; and 68.27)% for the highest percentage of cellulose obtained at 95ºC. The longer contact time in pretreatment leads to a decrease in the percentage of lignin and a significant increase in the percentage of cellulose.

The composition of banana stems obtained from each increase in contact time in the pretreatment process showed a downward trend in lignin percentage and an upward trend in cellulose percentage at each variation in contact time in the microwave. The longer the contact time of delignification in the microwave used, the percentage of lignin contained in the sample will decrease while the percentage of cellulose will increase [15]. This is due to the longer the contact time in the microwave, will cause structural damage to the cell wall and accelerate chemical reactions so that lignin and hemicellulose decomposition occur [19]. A decrease in the percentage of lignin will certainly affect the increase in the percentage of cellulose. Based on the results of the analysis, the optimum contact time condition in alkaline microwave-assisted pretreatment is 50 minutes.

3.4. Effect of Temperature on Alkaline Microwave-Assisted Pretreatment on Banana Stem Composition

In addition to time, alkaline microwave-assisted pretreatment is also affected by temperature. The overall percentage of the composition of banana stems after pretreatment can be seen in Fig. 4. Furthermore, the effect of temperature on lignin and cellulose levels at the optimum contact time of 50 minutes can be seen in Fig. 5.

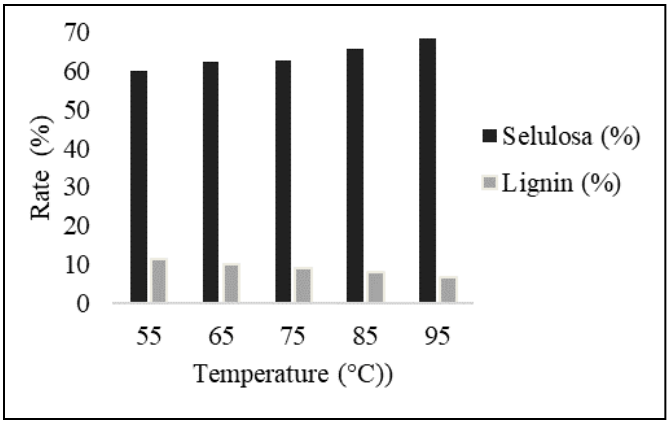

Fig. 5. Effect of Temperature on The Rate of Lignin and Cellulose during 50 Minutes of Alkaline Microwave-Assisted Pretreatment

The lowest percentage of lignin and the highest percentage of cellulose at temperature (55, 65, 75, 85, and 95) ºC can be seen in Fig. 5., respectively (11.27; 10.13; 9.01; 8.17; and 6.84))% for the lowest percentage of lignin and (59.91; 62.20; 62.74; 65.76; and 68.27)% for the highest percentage of cellulose obtained at an optimum contact time of 50 minutes in the microwave. The increase in temperature in pretreatment causes a decrease in the percentage of lignin and a significant increase in the percentage of cellulose.

The composition of banana stems obtained from each increase in temperature during pretreatment showed a downward trend in lignin percentage and an upward trend in cellulose percentage at each variation in microwave contact time. The higher the operating temperature and the longer the contact time in the microwave used, the percentage of lignin contained in the sample will decrease while the percentage of cellulose will increase [15]. This is due to the longer the contact time in the microwave, will cause structural damage to the cell wall and accelerate chemical reactions so that lignin and hemicellulose decomposition occur [19]. Pretreatment using microwave assistance is more effective than using alkali, this can be seen from the decrease in lignin by (35.21; 41.76; 48.20; 53.03; and 60.68)% at each temperature variation with a contact time in the microwave for 50 minutes. A decrease in the percentage of lignin will undoubtedly affect the increase in cellulose. It is caused by microwave-generated microwaves penetrating the lignocellulose wall and directly vibrating molecules, causing an increase in temperature and destroying the lignocellulose structure. These resulting vibrations break inter- and intra-molecular hydrogen bonds, which refers to an increase in the percentage of cellulose [8], [14]. Based on the results of the analysis, the optimum condition in alkaline microwave assisted pretreatment is heating at 95ºC with a contact time in the microwave for 50 minutes.

Comparing the two pretreatment methods from the observed data, it can be said that alkaline microwave-assisted pretreatment is more effective than pretreatment using alkaline KOH alone. Microwave-assisted alkaline pretreatment resulted in a decrease in lignin percentage and a higher increase in cellulose percentage compared to KOH alkaline pretreatment. Under optimum conditions, alkaline pretreatment produces the highest cellulose content of 64.38% and the smallest lingnin content of 8.13%, while alkaline microwave-assisted pretreatment produces the highest cellulose content of 68.27% and the smallest lingnin content of 6.84%. In addition, according to Kassim et al [8], the delignification process using alkaline pretreatment requires a longer time than alkaline microwave pretreatment – assisted in producing the same cellulose concentration at a fixed alkaline concentration [8], [20].

4. Conclusion

Based on the results of research and alteration, it can be concluded that both pretreatment methods are effective in reducing the percentage of lignin and increasing the rate of cellulose in banana stem biomass. The temperature and contact time during pretreatment significantly affect the banana stem composition. The higher the temperature and the longer the contact time, the better the composition of the banana stem biomass produced. In both pretreatment methods, namely alkaline KOH pretreatment and alkaline microwave-assisted pretreatment, the most optimal results were obtained at 95°C with a contact time of 50 min. However, alkaline microwave-assisted pretreatment is more effective than alkaline pretreatment because the effects of alkaline microwave-assisted pretreatment analysis obtained an optimal lignin percentage of 6.84% and cellulose of 68.27%. In comparison, alkaline pretreatment received an optimal lignin percentage of 8.13% and a cellulose percentage of 64.38%. Further research, it can be continued to the next process, namely hydrolysis to produce glucose as raw material for the bioethanol production. In addition, alkaline pretreatment or alkaline microwave-assisted pretreatment can also be done using other type of biomass.

Acknowledgment

The research/publication of this article was funded by DIPA of the Public Service Agency of Universitas Sriwijaya 2023. Number SP DIPA-023.17.2.677515/2023, On November 30, 2022. Under the Rector’s Decree Number: 0188/UN9.3.1/SK/2023, On April l8, 2023.

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