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Microwave Assisted Alkali Pretreatment of Elephant Grass using Sodium Hydroxide and Potassium Hydroxide

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Abstract

Microwave-assisted alkali pretreatment of Pennisetum purpureum, elephant grass (EG) prior to the hydrolysis process was conducted using sodium hydroxide (NaOH) and potassium hydroxide (KOH) to investigate the effect on lignin content and crystallinity index of cellulose. Microwave-assisted alkali pretreatment of EG was performed using NaOH and KOH at the same concentration of 2.5 M at various residence times of 10, 20, 30, 40 and 60 minutes with a microwave power of 180 W. Lignin content was significantly reduced, measuring 46.08% and 48.9% as the pretreatment time increased at 60 minutes for NaOH and KOH solution, respectively. The optimal condition for the pretreatment was found to be 20 minutes of irradiation, as it resulted in the highest crystallinity index of cellulose at 64.64% and 63.43% for NaOH and KOH pretreatment, respectively.

1. Introduction

In recent times, there has been a growing interest in nanocrystal cellulose (NCC) due to its unique properties, characterised by a nanoscale structure that can be isolated from various sources such as plants, animals, or bacteria [1]. NCC has been widely used in nanocomposite industry as reinforcing nanofillers in composite matrices because of its excellent strength of mechanical properties [2]-[6]. There are many researchers in biomedical industry that used NCC because of its biocompatibity. According to Li et al. [4], NCC is polymer matrices that have strong biocompatibity with metallic particles such as silver. The isolation process of NCC typically involves three steps: (i) pretreatment, (ii) bleaching and (iii) acid hydrolysis. The initial step aims to degrade lignin, while the subsequent stages focus on further lignin and hemicellulose removal, followed by cellulose disruption to enhance crystallinity index.

While numerous studies have investigated biomas pretreatment for cellulose to bioethanol conversion, considering factors like crystallinity index, crystal size, lignin and hemicellulose removal [7]-[12]. There is a noticeable gap in knowledge concerning an efficient pretreatment process for NCC isolation. Much of the existing literature predominantly analyses the final product, neglecting a comprehensive examination of the process itself [13]-[15].

Pretreatment is conducted physically or chemically and involves adjustment of biomass in a short duration of hydrolysis of cellulose and hemicellulose and has a good yield. Compared to other pretreatments, sodium hydroxide (NaOH), potassium hydroxide (KOH) and calcium hydroxide used as alkaline solutions are mild and more environmentally friendly because of the low concentration used for removing lignin content of biomass. In some applications such as medical and composite, it is important to produce NCC that are have high crystallinity index because of its high thermal stability and great rigidity [16]. Therefore, the crystallinity index is significant to be studied since the first part of the NCC isolation process which is the pretreatment process.

Previously, most researchers used NaOH solution in alkaline pretreatment by using conventional heating method where heat was transferred from external source to surface of material using conduction, convection or radiation and into the interior material by thermal conduction [17] with high temperature range of 50-180 °C. Collazo-Bigliardi et al. [18] have pretreated coffee husk by NaOH solution at reflux temperature (105-110 °C) for 3 hours and acacia bark has been pretreated by Taflic et al. [19] using NaOH solution at 121 °C for 30 min. NaOH catalytic organosolv pretreatment of sugarcane bagasse has removed up to 75.5% lignin with optimum condition at 180 °C for 45 minutes [20]. As mention by Wu et al. [21], sodium ion from NaOH contributed to water pollution. KOH pretreatment is recommended because it acts as a plant nutrient and more environmental friendly than NaOH. Capsosiphon Fulvescens has been pretreated by KOH solution at 80 °C for 2 hours in preparation of polymorph I and II NCC isolation [22].

Compared to conventional heating pretreatment, microwave-assisted pretreatment has heating medium, which is faster, uniform and more direct. In contrast with conventional hot plates, the microwave heat sources heated more polar parts throughout the sample and generate 'hot spot' within heterogeneous samples. Thus, it is predicted that an 'explosion' effect occurs within the particles which enhance interruption of recalcitrant structures of lignocellulose. Ishfaq Bhat et al. [23] reported that microwave assisted sodium chloride of rice straw have effectively removed lignin up to 93.51%. Zhou et al. [24] proved that microwave assisted lignin isolation produced more yield compared to the conventional heating method at same condition of 190 °C.

Despite the extensive research in the field, a comparative study between two different alkaline pretreatments, NaOH and KOH, for NCC isolation from elephant grass (EG) using microwave assisted technique is lacking. EG, with its rapid growth, has emerged as a promising source of lignocellulosic biomass in Malaysia since the 1920s [25]. This study aims to study microwave-assisted pretreatment EG using NaOH and KOH, measuring dielectric properties and temperature, and assessing lignin degradation and crystallinity index using TAPPI method and XRD respectively. The expectation is a reduction in lignin content and an increase in crystallinity index following the pretreatment process.

2. Methodology

2.1 Preparation of Raw Material

EG was cut roughly into 2.5 cm length and dried in an oven at 110 °C until moisture content is less than 10%. The dried sample was grounded and sieved to obtain 150 - 355 µm. The samples were sealed in plastic bags and placed in a desiccator to maintain its moisture content.

2.2 Pretreatment of Elephant Grass (EG)

Prior to microwave pretreatment, sample to solvent ratio of 1:10 with 5 g of EG to 50 ml of 2.5 M NaOH was mixed. Microwave pretreatment was conducted for 20 minutes at 180 W. The temperature of the sample in microwave was measured using Pro'sKit Infrared Thermometer MT-4612 for every 10 minutes. The mixture was then filtered and washed with distilled water several times until filtrate reached a neutral pH before it was oven-dried at 60 °C for 6 hours. The same procedure was conducted using 2.5 M KOH

2.3 Measurement of Dielectric Properties

Five g of EG was mixed with 50 ml of 2.5 M NaOH solution. Dielectric properties were measured using Vector Network Analyzer at frequencies ranging from 1.45 to 3.45 GHz at 25±1 °C. A probe sensor was immersed into the sample to measure its dielectric properties after calibrated using distilled water. The measurement was repeated thrice for each sample. The same procedure was repeated using 2.5 M KOH solution. Penetration depth was calculated using Eq. (1) [26]:

$$D_p = \frac{\lambda_0}{2\pi\left(2\varepsilon'\right)^{0.5}}\left[\left[\left\{1+\left(\frac{\varepsilon''}{\varepsilon'}\right)^2\right\}^{0.5}\right]-1\right]^{-0.5} \tag{1}$$

where D_p is the penetration depth, λ_0 is wavelength of microwave frequency, which is 0.1224 m, ε' is dielectric constant and ε'' is dielectric loss of material.

The conversion of electromagnetic waves into heat can be related by tangent loss ($\tan\delta$) as presented in Eq. (2) [26]:

$$\tan\delta = \frac{\varepsilon''}{\varepsilon'} \tag{2}$$

2.4 Lignin degradation using TAPPI method

The Kappa number (K) method calibrated with Klason lignin method was used to determine lignin degradation according to TAPPI T 222 om-88 [27].

2.5 Crystallinity Index (CrI)

X-ray diffraction (XRD) patterns were obtained using an X-ray diffractometer (PANalytical XpertPro) at room temperature. Samples were scanned with a step of 0.04° and scanning time of 5.0 min with 2θ angle ranging from 5° to 90°. Crystallinity index (*CrI*) was calculated from heights of 200 peak (I_{200}, 2θ = 22°) and intensity minimum between the 200 and 110 peaks (I_{am}, 2θ = 18°) using Segal method [28] as in Eq. (3). I_{200} represents crystalline regions, while I_{am} represents amorphous regions.

$$CrI(\%) = \frac{I_{200} - I_{am}}{I_{200}} \times 100 \tag{3}$$

3. Results and Discussion

3.1 Dielectric Properties

The dielectric properties of a material refer to its ability to absorb and generate heat through interaction with microwave electromagnetic wave. The dielectric constant signifies a material's capacity to be polarized by an external electric field (microwave wave) and store energy, while the dielectric loss factor indicates the material's ability to dissipate absorbed energy in a form of heat. These properties can be related as loss tangent or dissipation factor (tan d), describing the material's capability to absorb and convert electromagnetic wave energy into heat [29]. Table 1 illustrates the notable differences in dielectric properties between raw elephant grass and its mixure in an alkaline solution. The low values of dielectric properties of raw EG align with those reported in literature for dry grassy biomass [26]. Interestingly, besides its primary role in pretreatment, the addition of NaOH and KOH solutions become polarized by microwave electromagnetic wave, converting into heat. Comparing NaOH and KOH, the dielectric constant and dielectric loss for KOH were higher, while the loss tangent was lower than NaOH. This suggests that NaOH is more effective in converting microwave electromagnetic wave into heat, given its higher loss tangent value (the ratio of dielectric loss to dielectric constant) than KOH [30].

Table 1 *Dielectric properties of the sample*

Dielectric Property	Raw EG	NaOH 2.5 M solution	KOH 2.5 M solution
Dielectric constant, ε'	1.6904 ± 0.0730	16.4601 ± 3.2610	38.2200 ± 3.3208
Dielectric loss, ε''	0.2597 ± 0.0113	132.8094 ± 44.0209	186.8641 ± 16.0334
Loss tangent, tanδ	0.1536 ± 0.0046	8.5848 ± 0.0002	4.9924 ± 0.1741
Penetration Depth, Dp (cm	0.0979 ± 4.55 E-03	0.0012 ± 1.52 E-04	0.0012 ± 5.78 E-05

Studying the dissemination of the microwave energy into the material is crucial for understanding the penetration depth, a parameter essential for designing and scaling up microwave heating system [31]. The penetration depth, denoted as 0.0012 m for both KOH and NaOH, indicates the depth at which microwave energy is absorbed into the mixture. Materials with thickness exceeding the penetration depth are heated through conduction. Therefore, determining the penetration depth is vital for ensuring efficient microwave heating. Interestingly, despite the higher loss tangent and lower dielectric constant of the NaOH solution compared to, both alkalis exhibit the same penetration depth at room temperature. This finding underscores the importance of considering penetration depth as a key factor in achiving effective microwave heating.

3.2 Temperature Profile of Microwave assisted Alkali Pretreatment

Fig. 1 shows temperature profile of microwave assisted alkali pretreatment for NaOH and KOH. NaOH has more fluctuated temperature profile than KOH and showed that the NaOH has higher loss tangent which indicate more conversion of microwave energy into heat than KOH which is in agreement with calculated loss tangent shown in Table 1. The highest temperature for NaOH and KOH were 88.57±0.5488 °C and 76.90±1.3267 °C respectively at 30 minutes irradiation. Although KOH has high capability to polarized and dissipated the electromagnetic wave into heat (high dielectric constant), its loss tangent was lower than NaOH solution [32].

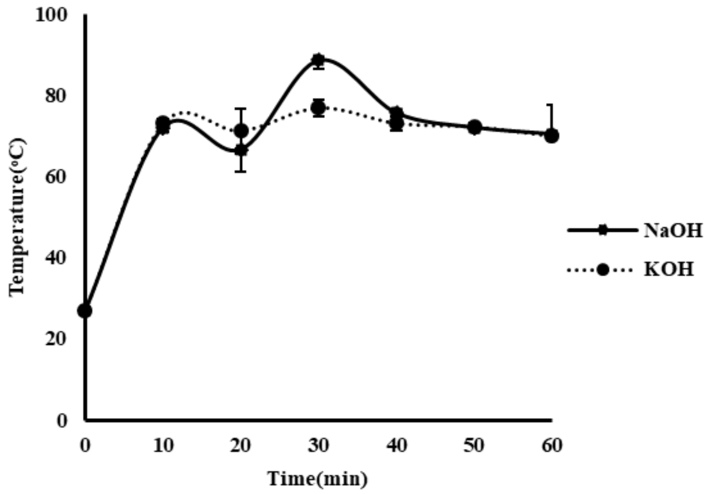

Fig. 1 *Temperature profile of microwave assisted alkali pretreatment at 180 W*

3.3 Effects of Microwave Irradiation Time on Lignin Degradation

Fig. 2 illustrates the percentage of lignin degradation in elephant grass (EG) following NaOH and KOH pre-treatments. Lignin degradation increased with pretreatment time for both alkaline processes, with the degradation slightly higher for KOH compared to NaOH [33]. After 10 minutes of KOH and NaOH pretreatments, 43.26% and 36.87% of lignin were respectively removed from the EG. This removal percentage steadily increased to 46.08% for NaOH pretreatment at 60 minutes. In contrast, for KOH pretreatment, the removal percentage slightly decreased reaching 48.90% at 60 minutes. Notably, after 10 minutes, lignin degradation for both KOH and NaOH pretreatments gradually increased and almost become constant at 60 minutes. This stabilization was attributed to the high lignin content, which degraded in the solution, and the degradation process did not continue once an equilibrium was reached.

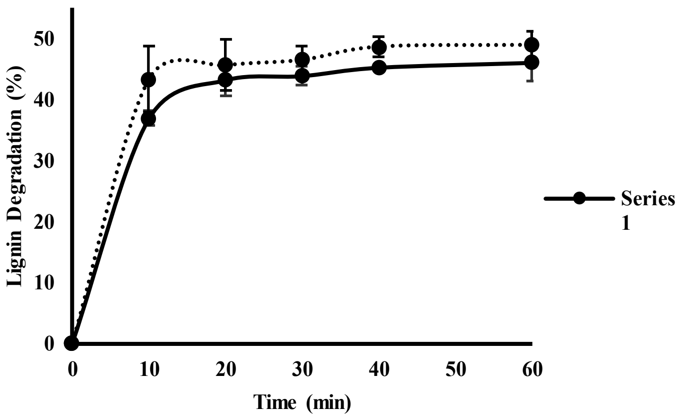

Fig. 2 *Percentage of lignin degradation of treated elephant grass versus pretreatment time*

3.4 Crystallinity Index (CrI)

In Fig. 3, the highest peak observed was in the crystalline region at 2θ=22º, I_{200} , while the second highest peak was in the amorphous region at 2θ=18º, I_{am}. Table 2 presents the calculated crystallinity index (Crl) using Eq. (2). For untreated elephant grass (EG), the CrI is 46.13%. With KOH pretreatment, the CrI increased to 58.13% and 63.43% at 10 and 20 minutes, respectively due to the high temperature during heating and the significanct removal of lignin. However, at 30, 40 and 60 minutes, the CrI decreased, reaching to 60.52% attributed to the highest heating temperature altering the morphology of the EG fibers [34]. The diffusion of the base into the cellulose expanded its chain and rearranging the amorphous region and resulting in an increased CrI [35]. The highest temperature detected at 30 minutes (Fig. 1) damaged the crystalline structure of cellulose and increasing the amorphous region and leading to a decrease in CrI [36]. A similar trend was observed in CrI for NaOH pretreatment, which increased from 58.96% to 64.64% at 10 to 20 minutes but decreased to 61.92% at 30 minutes and reached 54.98% at 60 minutes of irradiation, for the same reasons as observed in KOH pretreatment. These results suggest that the optimum irradiation time for alkaline pretreatment at 180 W was 20 minutes as it produced the highest crystalline cellulose.

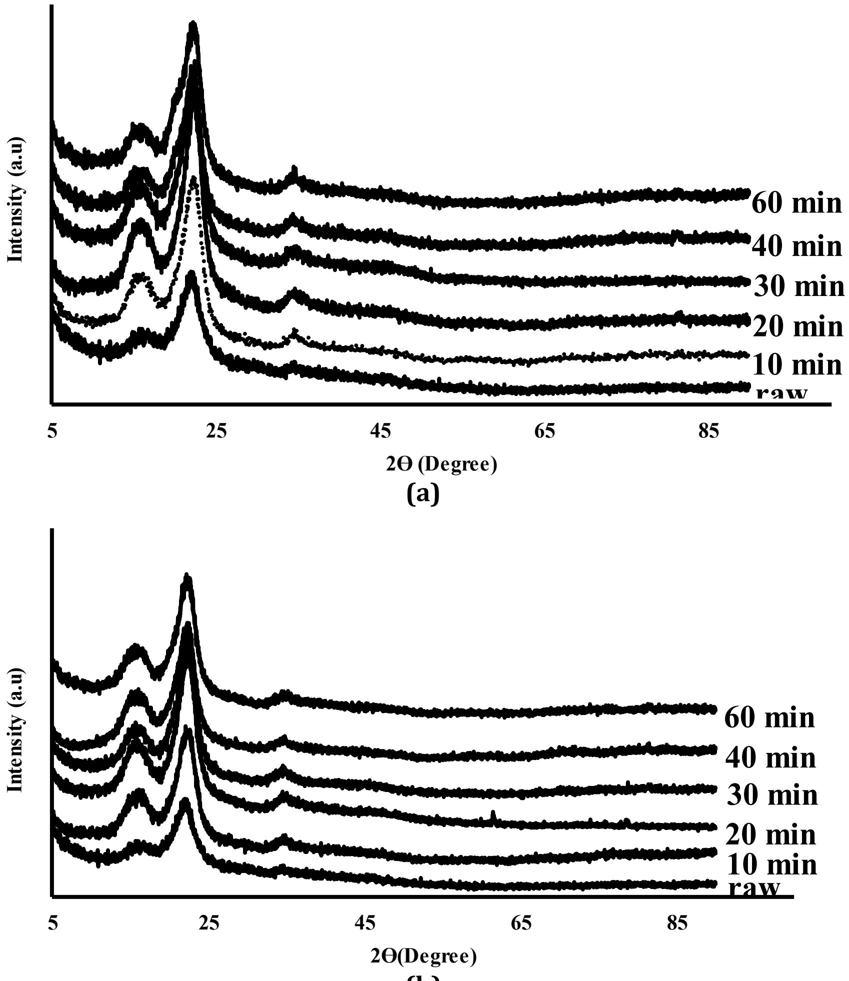

Fig. 3 *XRD patterns of pretreated elephant grass using (a) NaOH solution, and (b) KOH solution*

Table 2 *Crystallinity index of untreated and pretreated elephant grass*

Crystallinity Index (%)		
Untreated	46.13	
Pretreated	NaOH 2.5 M	KOH 2.5 M
10 min	58.96	58.13
20 min	64.64	63.43
30 min	61.92	60.68
40 min	55.13	60.08
60 min	54.98	60.52

4. Conclusion

Microwave heating at 180 W for 20 minutes is recommended because, under these conditions, a substantial amount of lignin was removed, leading to a high crystallinity index of elephant grass (EG) cellulose. As time increased, the lignin content in the EG degraded, and crystallinity index for both treatments decreased at 30 minutes due to overheated conditions, raising the heating temperature to 88.57±0.5488 and 76.90±1.3267 °C for NaOH and KOH, respectively. The highest crystallinity of EG, measuring 64.64% and 63.43 % along with a significant reduction in lignin content to 43.21% and 45.55 %, was achieved with 20 minutes of NaOH and KOH pretreatments, respectively. This study emphasizes the significance of power and time during pretreatment with alkaline solutions as crucial factors for lignin removal and increased crystallinity index in EG, essential steps before bleaching and acid hydrolysis for producing nanocystalline cellulose (NCC).

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Conflict of Interest

Authors declare that there is no conflict of interest regarding the publication of the paper.

Author Contribution

The authors confirm contribution to the paper as follows: ***study conception and design:*** *Syaidatul Akma Mohd Zuki, Norazah Abd Rahman, Noor Fitrah Abu Bakar;* ***data collection:*** *Nur Aliah Abd Latiff;* ***analysis and interpretation of results:*** *Syaidatul Akma Mohd Zuki, Nur Aliah Abd Latiff;* ***draft manuscript preparation:*** *Syaidatul Akma Mohd Zuki. All authors reviewed the results and approved the final version of the manuscript.*

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