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Research Article

Microwave-Assisted Catalytic Optimization of Pineapple Industrial Waste Conversion for Bioethanol Production

Livia Rhea Alvita[1*], Vida Elsyana[1], Rahmat Hidayat[1], Amelia Sri Rezki[2], Silvia Malum Padang[2], Rafli Rustiawan Jody[1]

[1]*Study Program of Applied Chemistry, Department of Agricultural Technology, Politeknik Negeri Lampung, Lampung 35141, Indonesia*
[2]*Study Program of Industrial Chemical Engineering Technology, Department of Engineering, Politeknik Negeri Lampung, Lampung 35141, Indonesia*

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ABSTRACT

Pineapple peel waste has high potential as a bioethanol feedstock due to its abundant lignocellulosic content, although its utilization remains limited to compost and animal feed. This study aims to optimize the catalytic conversion of pineapple peel waste into bioethanol using a microwave-assisted system with homogeneous (HCl) and combined acid–zeolite catalysts. The process involved physical pretreatment, delignification using 1% NaOH and 2% Al_2O_3 (135 W, 40 min), catalytic hydrolysis, four-day fermentation, and distillation. Characterization using FTIR and SEM–EDX revealed significant chemical and morphological changes. Lignin content decreased from 30.98% to 14.36%, while cellulose and hemicellulose increased to 32.82% and 40.67%, respectively. Hydrolysis with 0.5 N HCl and zeolite for 45 minutes produced the highest reducing sugar concentration of 18.36 g/L and total dissolved solids of 4.05 °Brix. Two-way ANOVA analysis confirmed that catalyst concentration, hydrolysis time, and their interaction had a significant effect on reducing sugar yield ($p<0.05$). Fermentation produced 7.9% ethanol content (GC–MS) and 6.51% yield. Overall, the integration of microwave-assisted hydrolysis and acid–zeolite catalysis effectively enhanced lignocellulosic conversion efficiency, demonstrating a promising approach for sustainable bioethanol production.

1. Introduction

The utilization of agro-industrial residues is a promising strategy to support sustainable resource management and the development of renewable energy. Among tropical biomass resources, pineapple industrial waste constitutes a lignocellulosic material rich in cellulose, hemicellulose, and lignin—components with significant biotechnological potential for bioethanol production. Converting this underutilized biomass into biofuel not only promotes energy diversification but also reduces organic waste and mitigates environmental pollution (Hoang *et al.* 2021).

Despite its potential, the efficiency of lignocellulosic biomass conversion remains limited due to the complex structural network between lignin and polysaccharides, which restricts enzymatic accessibility and hampers hydrolysis efficiency. As a result, bioethanol yield from pineapple waste remains lower than that from other biomass sources (Xiang *et al.* 2022). Therefore, advanced biotechnological approaches are required to enhance delignification and hydrolysis efficiency for the release of fermentable reducing sugars.

Catalytic systems—both homogeneous and heterogeneous—have been extensively explored to accelerate biomass depolymerization and hydrolysis. Homogeneous acid catalysts, such as hydrochloric acid (HCl) and sulfuric acid (H_2SO_4), have been reported to effectively cleave glycosidic linkages and increase sugar yield (Sivamani *et al.* 2019). In contrast, heterogeneous catalysts, including zeolites and sulfonated silica-based materials, exhibit high stability, easy recoverability, and enhanced performance under milder reaction

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*Corresponding Author
E-mail Address: liviarhea@polinela.ac.id

conditions (Lin *et al.* 2021; Belkadhi *et al.* 2022). These heterogeneous catalysts operate in a different phase from the reactants, enabling easier separation and reuse compared to homogeneous systems. Recent advances also highlight the synergistic potential of combining homogeneous and heterogeneous catalytic pathways in sequential systems, yielding superior conversion efficiency compared to single-catalyst processes (Chen *et al.* 2020).

In parallel, microwave-assisted technologies have emerged as powerful tools in bioconversion due to their ability to induce rapid, uniform, and selective heating. Microwave irradiation facilitates the disruption of lignocellulosic structures, enhances delignification, and improves enzymatic accessibility. Sharma *et al.* (2023) reported a glucose yield of 327.8 mg/g within 7 minutes, with up to 44% conversion efficiency, while Liu *et al.* (2024) demonstrated up to 80% energy savings compared to conventional heating. Furthermore, the synergistic combination of catalytic treatment and microwave irradiation has been shown to accelerate hydrolysis and fermentation, leading to higher bioethanol yields in shorter reaction times (Karungi *et al.* 2024; Laltha *et al.* 2021).

Overall, the integration of catalytic systems with microwave-assisted processes represents a transformative approach in the field of environmental biotechnology for the efficient and sustainable conversion of lignocellulosic biomass. Valorizing pineapple industrial waste as a renewable feedstock not only reinforces circular bioeconomy principles but also supports the development of eco-friendly bioethanol production technologies.

Therefore, this study aims to optimize the conversion of pineapple waste biomass into fermentable sugars through microwave-assisted catalytic hydrolysis, focusing on the effects of catalyst type (homogeneous and heterogeneous), catalyst concentration, and reaction duration on hydrolysis efficiency. The outcomes are expected to advance the understanding of microwave-assisted bioconversion mechanisms and promote sustainable bioenergy development from tropical agricultural residues.

2. Materials and Methods

2.1. Physical Pretreatment of Feedstock

Pineapple peel waste used in this study was collected from a pineapple processing industry located in Lampung Province, Indonesia. The raw material was first subjected to physical pretreatment by drying and grinding using a high-speed blender to obtain fine powder. The powder was sieved to a particle size of 80–100 mesh following the procedure reported by Agustian & Redjeki (2014), which enhances cellulose accessibility and conversion efficiency. The initial pineapple peel powder was analyzed for moisture and carbohydrate contents using the Luff–Schoorl method.

2.2. Chemical Pretreatment (Delignification)

The delignification process was carried out using 25 g of powdered pineapple peel. A mixed alkaline solution containing 1% NaOH and 2% Al_2O_3 (total volume 300 mL) was prepared. The biomass-to-solution ratio was maintained at 1:12 (w/v). The mixture was subjected to microwave irradiation at 135 W for 40 minutes to promote lignin removal (Naqqiya 2024) (Figure 1). After irradiation, the slurry was filtered and repeatedly washed with distilled water until the pH was neutral.

The delignified biomass was then analyzed for lignin, cellulose, hemicellulose, ash, °Brix, and reducing sugar contents, as well as for structural and functional changes using FTIR spectroscopy. Results were compared with untreated control samples (0% NaOH, 0% Al_2O_3, and 0 W microwave power).

2.3. Hydrolysis Process

Hydrolysis of the delignified pineapple peel (15 g each) was conducted using two catalytic systems:

- Homogeneous catalyst: Hydrochloric acid (HCl) with concentrations of 0.1 N, 0.3 N, and 0.5 N.
- Combined catalyst: A mixture of HCl (same concentrations) and activated synthetic zeolite (3 g).

Each biomass sample was mixed with 300 mL of catalytic solution, maintaining a solid-to-liquid ratio of 1:20 (w/v). The mixtures were irradiated in a microwave reactor at 135 W for 15, 30, and 45 minutes, respectively. After hydrolysis, the hydrolysates were cooled and analyzed for reducing sugar content using the 3,5-dinitrosalicylic acid (DNS) method. The hydrolysate with the highest sugar yield was selected for fermentation, and its pH was adjusted to 4–5 prior to inoculation.

2.4. Fermentation

Fermentation was performed using *Saccharomyces cerevisiae* as the fermenting microorganism. A 100 mL portion of the selected hydrolysate was supplemented with 8 g of yeast, 0.8 g urea, and a nutrient mix containing

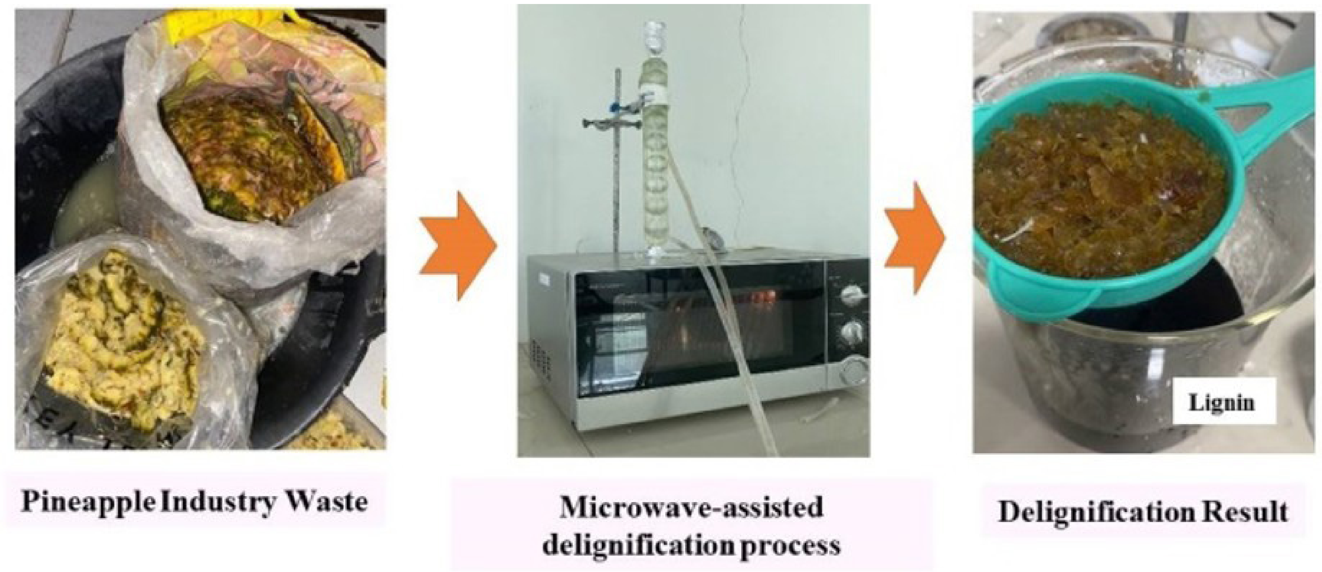


Figure 1. Delignification Process

(g/L): 0.02 $(NH_4)_2SO_4$, 0.01 K_2HPO_4, 0.01 KH_2PO_4, 0.02 $ZnSO_4$, 0.002 $MgSO_4$, and 0.02 yeast extract (Agustini & Febrian 2019). The mixture was incubated in tightly sealed Erlenmeyer flasks equipped with a gas-release tube to maintain anaerobic conditions. Fermentation was carried out for 96 hours (4 × 24 h) at ambient temperature.

2.5. Distillation

Purification of bioethanol was conducted by fractional distillation at 70–80°C for 2–3 hours until no further condensate was collected. The distillate was then analyzed to determine the ethanol content and purity.

2.6. Analytical Methods and Data Analysis

All samples, both before and after each processing step, were analyzed for their physicochemical properties. The following analyses were performed:

- pH: using a pH meter.
- Density: measured using a pycnometer.
- Moisture content: determined according to AOAC (1995).
- Carbohydrate content: measured by the Luff–Schoorl method.
- Reducing sugars: determined by the DNS method using glucose as a standard and measured at 540nm with a UV–Vis spectrophotometer.
- Soluble solids (°Brix): measured with a refractometer.
- Lignin, cellulose, hemicellulose, and ash contents: analyzed using the Chesson–Datta method.
- Functional group changes indicating lignin, cellulose, and hemicellulose degradation were examined by Fourier-transform infrared spectroscopy (FTIR).
- Morphological and elemental composition changes were observed using scanning electron microscopy coupled with energy-dispersive X-ray spectroscopy (SEM–EDX).
- Ethanol concentration and quality were analyzed using gas chromatography–mass spectrometry (GC–MS) using a TG5-MS capillary column with helium as the carrier gas (mobile phase), and verified by density measurements with a pycnometer. Ethanol yield (%) was calculated from the density–ethanol correlation table.

Data analysis was performed using Minitab software. A two-way analysis of variance (ANOVA) was applied to evaluate the effects of hydrolysis time and catalyst concentration on reducing sugar yield. The significance level was set at $p<0.05$. When significant effects were detected, Duncan's multiple-range test (DMRT) was used to identify the most influential treatment combinations.

3. Results

3.1. Characterization of Pineapple Peel Biomass

A comprehensive characterization of the raw material is essential for evaluating its suitability and conversion potential in bioethanol production. The pineapple peel waste utilized in this study exhibited physicochemical properties indicative of high lignocellulosic content, making it a promising feedstock for bioethanol synthesis. The preliminary proximate analysis revealed a moisture content of 9.36% and a total carbohydrate content of 38.23%, suggesting a relatively dry biomass with substantial fermentable material.

The detailed compositional analysis, performed using the Chesson–Data method, identified hemicellulose, cellulose, and lignin as the major structural components (Table 1). The proportions of these constituents were 20.17% hemicellulose, 18.06% cellulose, 30.98% lignin, and 4.55% ash.

The relatively high lignin content observed (30.98%) indicates a significant structural recalcitrance, which can impede the accessibility of hydrolytic agents to polysaccharides during conversion processes. Since cellulose and hemicellulose are the primary carbohydrate polymers that yield fermentable glucose upon hydrolysis, the delignification step is crucial for improving the efficiency of subsequent hydrolysis and fermentation.

3.2. Delignification Results

Microwave-assisted delignification at 135 W for 40 minutes, with the addition of 1% NaOH and 2% Al_2O_3, resulted in significant compositional changes in pineapple peel biomass. As presented in Figure 2, the lignin content decreased markedly from 30.98% to 14.36%, indicating substantial removal of the lignin fraction under the applied conditions.

Table 1. Chemical composition of pineapple peel biomass

Component	Content (%)
Hemicellulose	20.17
Cellulose	18.06
Lignin	30.98
Ash	4.55

Following delignification, the hemicellulose and cellulose contents increased from 20.17 to 40.67% and from 18.06 to 32.82%, respectively. This compositional shift reflects the relative enrichment of carbohydrate components as lignin was removed during treatment, suggesting effective structural modification of the lignocellulosic matrix.

Fourier-transform infrared (FTIR) spectroscopy analysis further confirmed the chemical alterations induced by the delignification process. The characteristic absorption peaks associated with lignin and carbohydrate functional groups are summarized in Table 2 and illustrated in Figure 3. A notable reduction in the absorption band corresponding to aromatic C=C stretching was observed, shifting from 1640.5 cm^{-1} to 1595.8 cm^{-1}, accompanied by a decrease in O–H stretching intensity (Figure 3). These spectral changes indicate the partial degradation of lignin and enhanced exposure of cellulose and hemicellulose structures.

3.3. Hydrolysis Performance and Soluble Sugar Yield

The hydrolysis of pineapple peel biomass produced soluble sugars, quantified in °Brix units. As shown in Figure 4, the highest °Brix value (4.05%) was obtained under the combined catalytic condition of 0.5 N HCl + zeolite at a reaction time of 45 minutes. This trend indicates a positive correlation between both catalyst concentration and hydrolysis duration with the total soluble sugar yield.

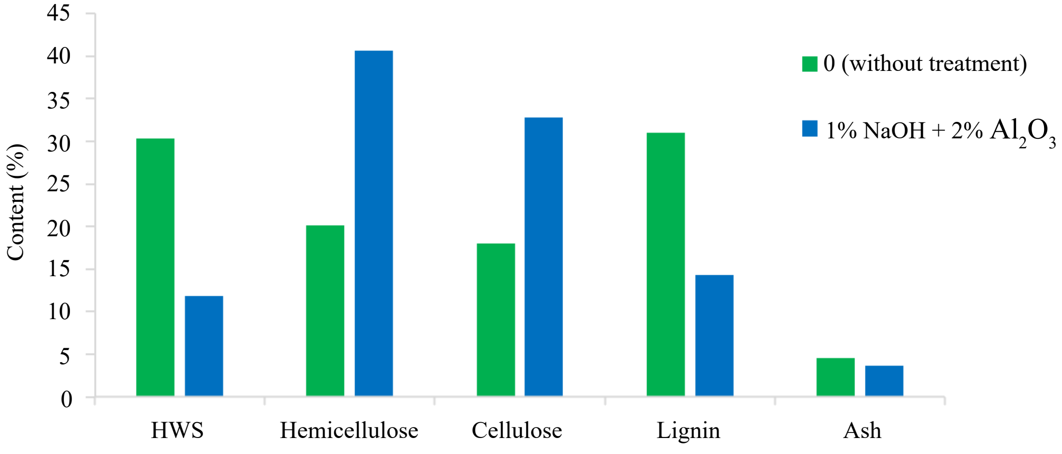


Figure 2. Effect of microwave-assisted delignification on lignin content of pineapple peel

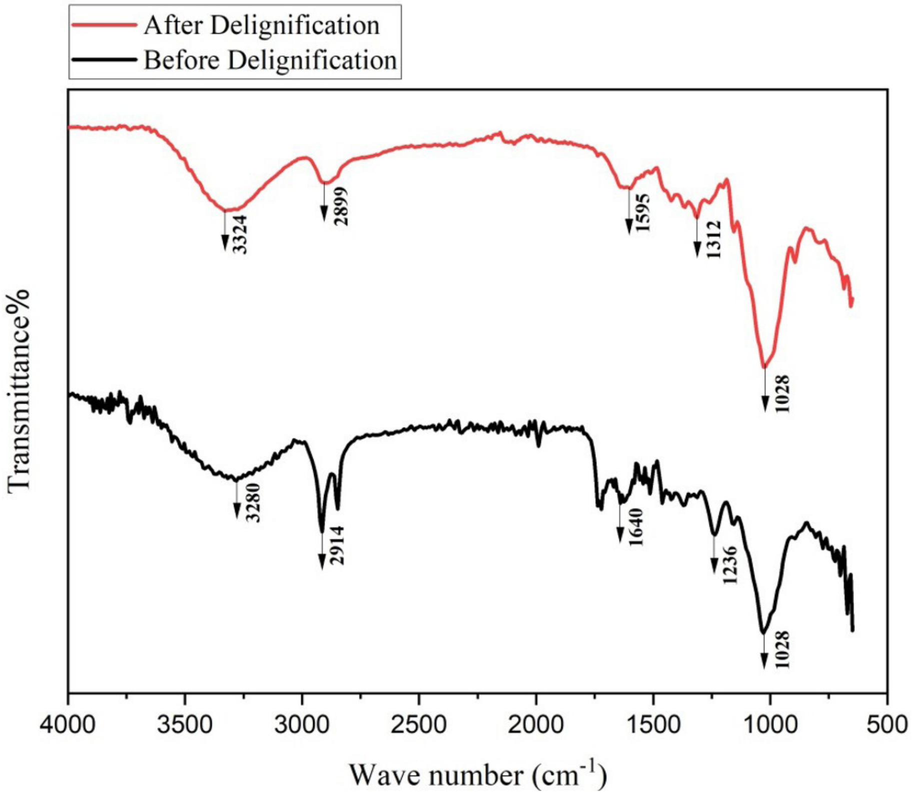


Figure 3. FTIR spectra of pineapple peel before and after microwave-assisted delignification

Table 2. FTIR absorption bands of pineapple peel before and after delignification

Functional group	Wavenumber (cm^{-1}) before	Functional group	Wavenumber (cm^{-1}) after
O–H	3280.1	O–H	3324.8
C–H	2914.8	C–H	2899.9
C=C (aromatic)	1640.5	C=C (aromatic)	1595.8
C–O (ester)	1236.9	C–O (ester)	1312.0
C–O–C	1028.7	C–O–C	1028.7

The analysis of reducing sugars (Figure 5) revealed a similar pattern. Increasing catalyst concentration and reaction time resulted in a substantial rise in reducing sugar yield. The optimum condition—0.5 N HCl combined with zeolite for 45 minutes—yielded 18.36 g/L of reducing sugars. The addition of zeolite notably enhanced the hydrolysis performance, as its high Si/Al ratio provides abundant acidic sites, facilitating polysaccharide cleavage and accelerating the depolymerization of cellulose into glucose monomers.

Two-way ANOVA confirmed that both catalyst concentration and reaction time, and their interaction, had a statistically significant influence on reducing sugar yield ($p<0.05$). The model demonstrated a strong fit, with a coefficient of determination (R^2) of 97.14%, indicating that these variables jointly explained most of the observed variation in sugar yield. Post-hoc Tukey's test further revealed that the treatment with 0.5 N HCl-zeolite produced significantly higher reducing sugar concentrations than other treatments, while the 45-minute hydrolysis time yielded results significantly superior to shorter durations (15 and 30 minutes).

To support the chemical analysis, morphological and elemental changes of the pineapple peel biomass before and after hydrolysis were examined using SEM–EDX (Figure 6). The SEM micrograph of untreated biomass (Figure 6A) exhibited a well-ordered fibrous structure with elongated fibrils and partially loosened surfaces following delignification, indicating enhanced accessibility for subsequent hydrolysis. In contrast, the hydrolyzed sample (Figure 6B) treated with the acid–zeolite catalytic system displayed a rougher, more fragmented surface, with the appearance of microcracks and newly formed pores, suggesting extensive disruption of the lignocellulosic matrix.

The EDX spectra corroborated the morphological observations. Prior to hydrolysis, the biomass surface was primarily composed of carbon (66.25 wt%) and oxygen (33.45 wt%), with a C/O atomic ratio of 1.98. After hydrolysis using acid and zeolite catalysts, the

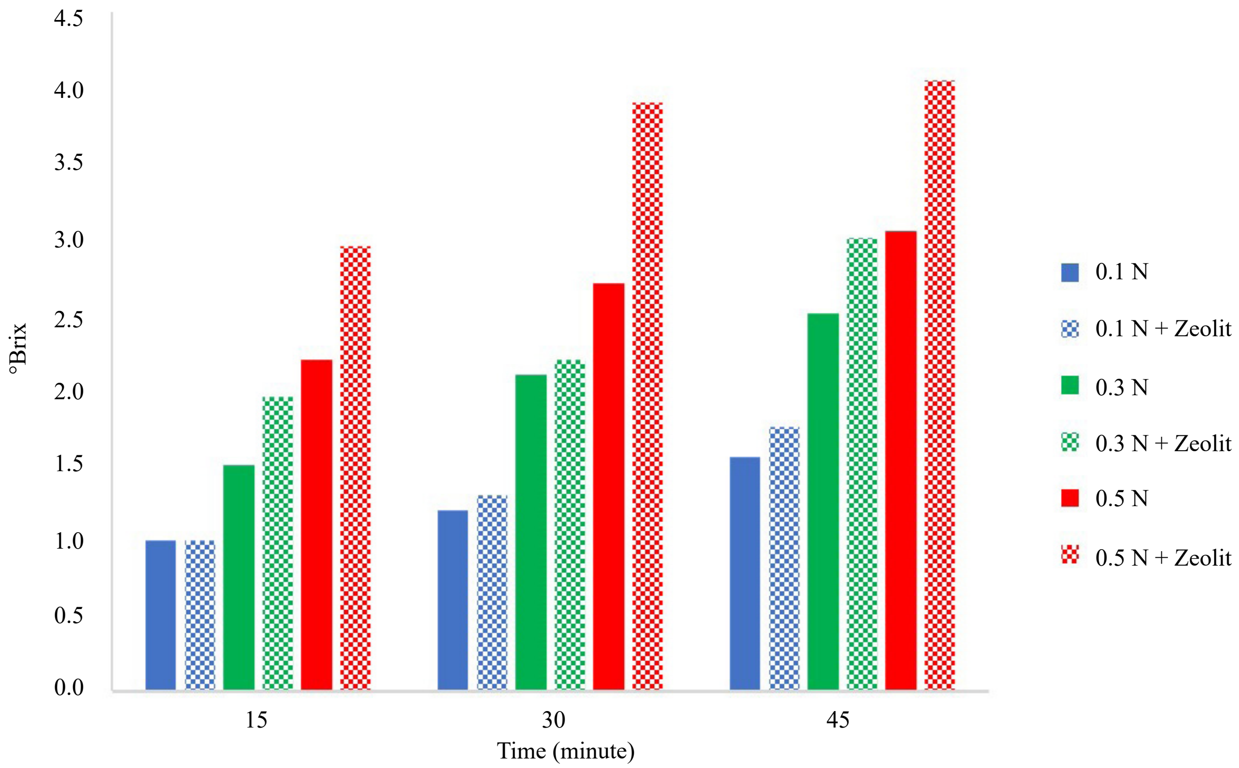


Figure 4. Soluble sugar content (°Brix) obtained from hydrolysis under different catalytic and time conditions

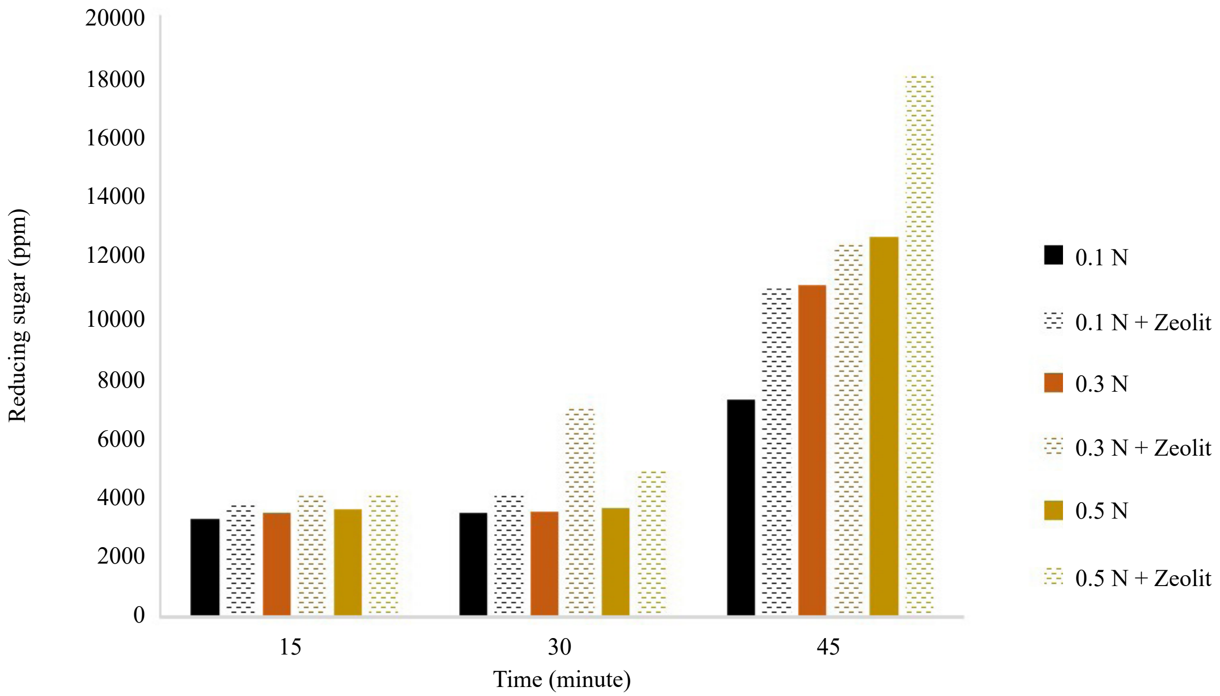
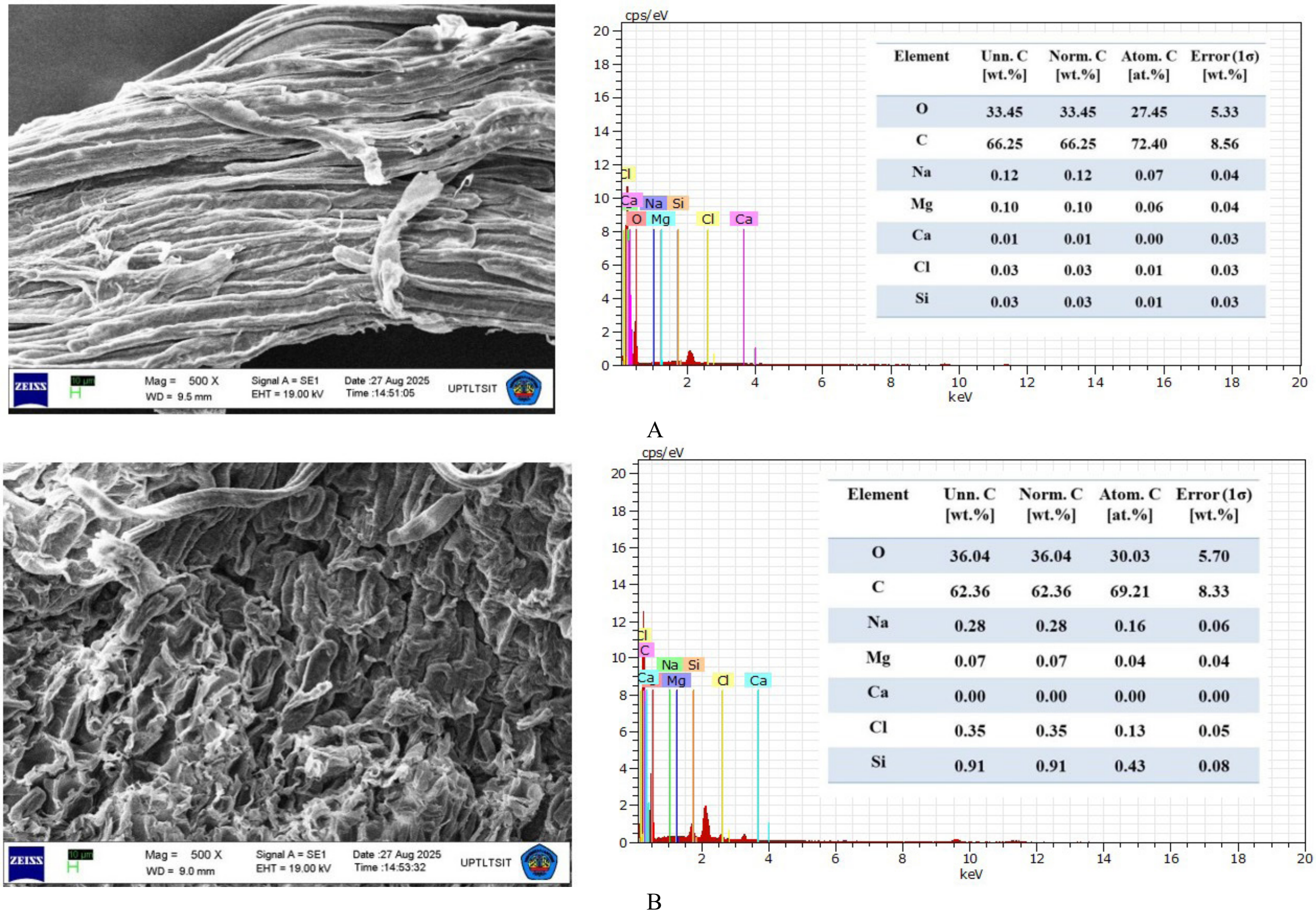


Figure 5. Reducing sugar concentration (g/L) as a function of catalyst concentration and hydrolysis time



Element	Unn. C [wt.%]	Norm. C [wt.%]	Atom. C [at.%]	Error (1σ) [wt.%]
O	33.45	33.45	27.45	5.33
C	66.25	66.25	72.40	8.56
Na	0.12	0.12	0.07	0.04
Mg	0.10	0.10	0.06	0.04
Ca	0.01	0.01	0.00	0.03
Cl	0.03	0.03	0.01	0.03
Si	0.03	0.03	0.01	0.03

Element	Unn. C [wt.%]	Norm. C [wt.%]	Atom. C [at.%]	Error (1σ) [wt.%]
O	36.04	36.04	30.03	5.70
C	62.36	62.36	69.21	8.33
Na	0.28	0.28	0.16	0.06
Mg	0.07	0.07	0.04	0.04
Ca	0.00	0.00	0.00	0.00
Cl	0.35	0.35	0.13	0.05
Si	0.91	0.91	0.43	0.08

Figure 6. SEM–EDX analysis of pineapple peel biomass (A) before and (B) after hydrolysis

carbon content decreased to 62.36 wt%, while oxygen increased to 36.04 wt%, lowering the C/O ratio to 1.73. This reduction reflects a higher oxidation level and the cleavage of glycosidic linkages within the cellulose backbone, consistent with polysaccharide depolymerization into monosaccharides. Additionally, the emergence of inorganic elements such as Na, Mg, Cl, and Si after hydrolysis suggests residual contributions from the acid catalyst (HCl) and from adsorbed zeolite particles during the reaction.

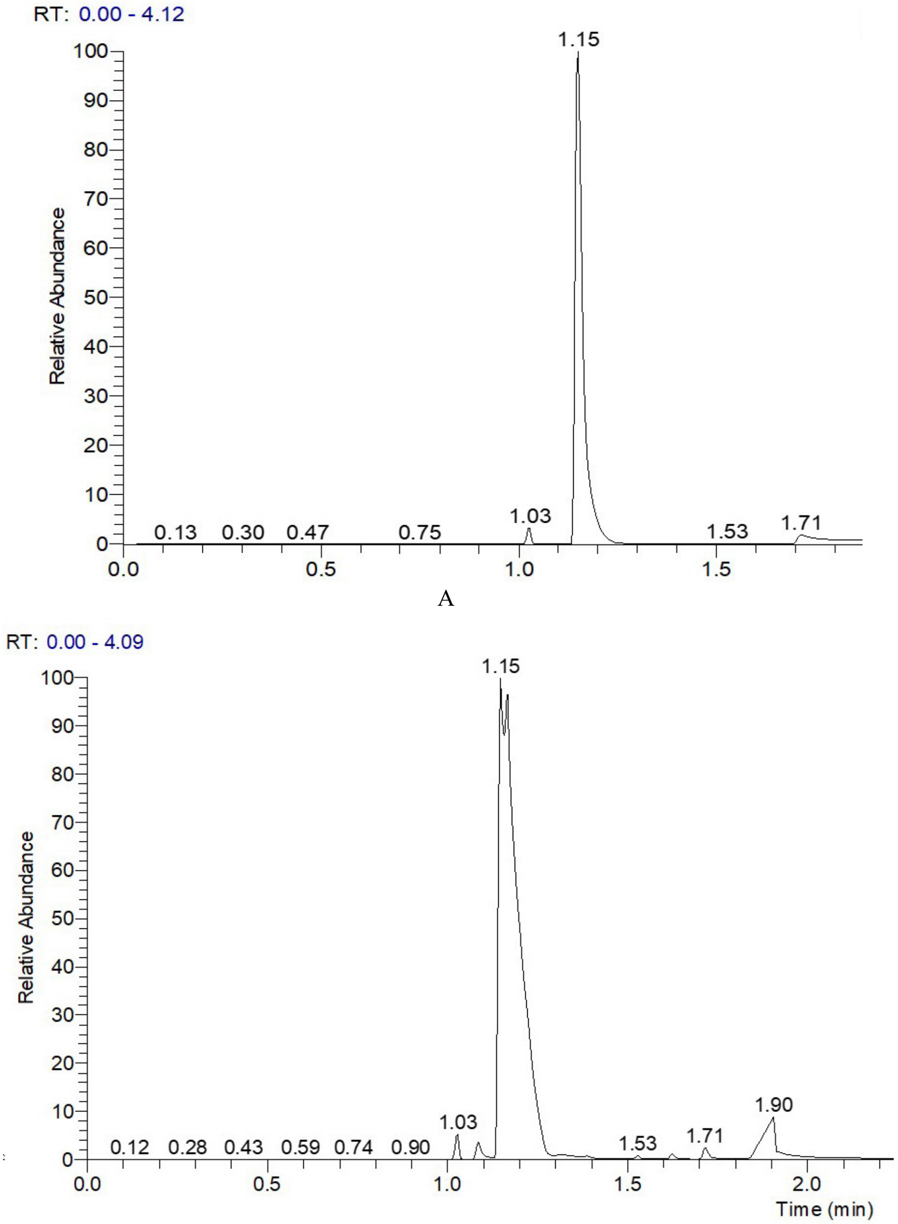
3.4. Fermentation Results and Bioethanol Characterization

The fermentation analysis was conducted to determine the ethanol concentration and to identify the chemical characteristics of the resulting compounds. Ethanol concentration was measured using two complementary methods: the pycnometer method and Gas Chromatography–Mass Spectrometry (GC–MS). The ethanol content determined by the pycnometer method is presented in Table 3.

Based on the measurement results, the density of the fermented sample was 0.97979 g/mL at 31.1°C. From this value, the ethanol concentration was calculated to be 9.067%, with an ethanol yield of 6.51%.

Analysis of volatile compounds in the fermentation product using GC–MS revealed a major peak at a retention time of 1.15–1.16 minutes, identified as ethanol. A 5% ethanol standard chromatogram was used as a reference to determine the retention time and peak area corresponding to ethanol. The chromatograms are shown in Figure 7, where Figure 7A represents the standard ethanol solution and Figure 7B shows the chromatogram of the fermentation sample.

In the chromatogram of the fermentation sample, the peak area (2,402,711,941.64) was noticeably higher than that of the standard (1,527,598,202.16), indicating a greater ethanol concentration in the sample. Based on the comparison of peak areas, the ethanol concentration in the sample was calculated to be 7.9%, as summarized in Table 4.



A

B

Figure 7. GC–MS chromatograms of (A) 5% ethanol standard and (B) fermentation sample

Table 3. Ethanol concentration analysis using the pycnometer method

Sample	Density (g/mL)	Temperature (°C)	Ethanol content (%)	Yield (%)
1	0.97979	31.1	9.067	6.51

The Gas Chromatography–Mass Spectrometry (GC–MS) analysis confirmed that ethanol was the major component of the fermentation product. The mass spectrum (Figure 8) exhibited a prominent molecular ion peak at m/z = 46, corresponding to the molecular weight of ethanol. The base peak observed at m/z = 31 represents the $(CH_2OH)^+$ fragment, which is characteristic of ethanol fragmentation. The Similarity Index (SI) of 845 and Reverse Similarity Index (RSI) of 851 showed a close match with the reference spectrum, confirming the compound identification as ethanol with a probability of 84.12% (Figure 8). The peak area percentage (91.65%) further indicated that ethanol was the dominant component in the fermentation product.

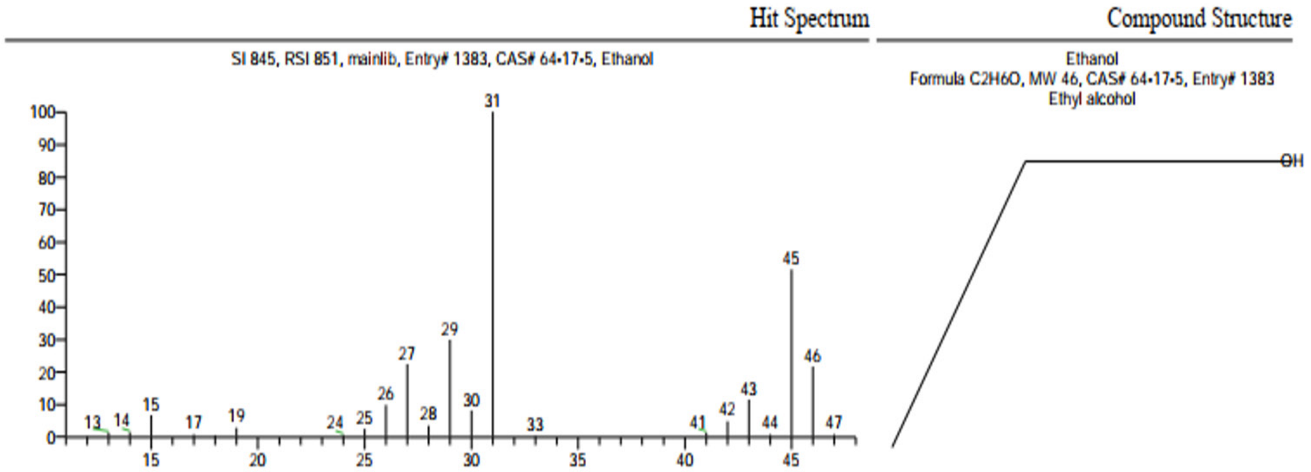


Figure 8. Mass spectrometry profile of the bioethanol sample

Table 4. GC–MS analysis results for ethanol determination

Sample code	Retention time (min)	Peak area	Standard concentration (%)	Ethanol Concentration in Sample (%) (b/a)×c
Ethanol Standard 5%	1.15	1,527,598,202.16 (a)	5.00 (c)	-
Fermentation Sample	1.16	2,402,711,941.64 (b)	-	7.9%

4. Discussion

The initial characterization analysis revealed that pineapple peel waste possesses high potential as a bioethanol feedstock due to its rich lignocellulosic and carbohydrate content. The low moisture content (9.36%) indicates the effectiveness of the drying process prior to chemical treatment, which contrasts sharply with the high moisture content of fresh pineapple peel (81.72–86.70%) as reported by Siskayanti *et al.* (2023). This drying step is crucial to prevent microbial decay and to enhance the efficiency of the subsequent pretreatment process (Aksari *et al.* 2024). The lignocellulosic composition—comprising 30.98% lignin, 18.06% cellulose, and 20.17% hemicellulose—classifies the biomass as a typical tropical lignocellulosic substrate with a complex structure and high recalcitrance to both chemical and enzymatic degradation. The relatively high lignin content acts as a structural binder, providing mechanical rigidity to the cell wall while simultaneously forming a barrier to the diffusion of enzymatic and hydrolytic reagents (Zoghlami & Paës 2019). Therefore, the delignification step is essential to improve carbohydrate accessibility prior to hydrolysis and fermentation.

Microwave-assisted delignification using a combination of a basic (NaOH) catalyst and a metal oxide (Al_2O_3) catalyst proved effective in reducing lignin content and enriching the carbohydrate fraction of pineapple peel biomass. This efficiency primarily results from the synergistic interaction between microwave energy and catalytic chemistry. Microwaves induce rapid, uniform heating of interacting with polar functional groups in biomass, promoting cleavage of aryl–ether and carbon–carbon linkages within the lignin network (Zhu *et al.* 2016; Qu *et al.* 2020). NaOH facilitates saponification of ester linkages between lignin and hemicellulose, while Al_2O_3 acts as a heterogeneous catalyst, enhancing heat transfer, accelerating aromatic decomposition, and destabilizing the lignin structure (Oregui Bengoechea *et al.* 2015; Huang *et al.* 2023). Under optimal conditions (1% NaOH, 2% Al_2O_3, 135 W, 40 min), lignin content decreased from 30.98 to 14.36%, indicating high efficiency in breaking lignin–carbohydrate bonds. These findings align with those of Rifqo *et al.* (2020), who achieved approximately 12% lignin using a similar chemical system without microwave assistance, demonstrating that microwave-induced electromagnetic and volumetric heating significantly accelerates reaction kinetics and mass transfer.

FTIR spectroscopy further supports the occurrence of chemical structure alterations following delignification. The shift of absorption bands from 1640.5 to 1595.8 cm^{-1} indicates degradation of aromatic C=C bonds characteristic of lignin, while changes in the 3280–3325 cm^{-1} region suggest the formation of new hydroxyl groups with increased polarity. These phenomena reflect the partial loss of phenylpropanoid structures and enhanced exposure of cellulose and hemicellulose functional groups at the biomass surface. This observation is consistent with the findings of Cao *et al.* (2020), who reported that microwave treatment increases cellulose fiber accessibility and enhances enzymatic hydrolysis efficiency up to 1.5-fold compared to conventional heating methods. Similarly, Nurfaizin & Hartati (2023) demonstrated that higher microwave power accelerates lignin degradation by increasing molecular collision energy and promoting partial oxidative reactions. Thus, microwave-assisted delignification with NaOH and Al_2O_3 yields a more open and reactive biomass structure, thereby improving subsequent hydrolysis performance with HCl and synthetic zeolite catalysts. This approach effectively enhances the conversion of lignocellulose into reducing sugars and ultimately bioethanol, offering a more efficient and industrially applicable pathway subject to further optimization of power, reaction time, and catalyst composition.

Hydrolysis of pineapple peel using a combination of HCl and zeolite under microwave heating demonstrated strong synergy between the homogeneous and heterogeneous catalysts, thereby enhancing overall lignocellulose conversion efficiency. Zeolite acts as a source of solid acid sites that amplify the protonation activity of HCl, thereby facilitating the cleavage of β-1,4-glycosidic bonds in cellulose and hemicellulose (Onda *et al.* 2008). Increasing catalyst concentration and reaction time corresponded to higher total soluble solids (°Brix) and reducing sugar concentrations, indicating a more efficient polysaccharide-to-monosaccharide conversion (Alvita *et al.* 2025). The optimum condition was achieved with 0.5 N HCl + zeolite for 45 minutes, producing 4.05% °Brix and 18.36 g/L reducing sugar. These results agree with Nulhakim *et al.* (2019), who reported that higher acid concentration enhances the depolymerization of cellulose and hemicellulose into glucose.

ANOVA analysis confirmed that both catalyst concentration and hydrolysis time significantly influenced hydrolysis performance ($p<0.05$), with a determination coefficient (R^2) of 97.14%, indicating a strong linear correlation between process variables and conversion yield. Tukey's post hoc test further revealed that the 0.5 N HCl + zeolite treatment for 45 minutes resulted in the highest reducing sugar yield, which was significantly higher than that of other treatments. The high efficiency is attributed to zeolite's strong acidity (high Si/Al ratio) and catalytic stability (Hernández-Giménez *et al.* 2021). The combination of homogeneous–heterogeneous catalysis under microwave heating thus effectively improves the hydrolysis efficiency of pineapple peel lignocellulose into fermentable sugars.

SEM–EDX analysis confirmed extensive physical and chemical alterations in the biomass after hydrolysis. The surface morphology became more porous and fragmented, indicating disruption of the lignocellulosic network and enhanced accessibility to catalysts and fermentative microorganisms. The decrease in the C/O ratio from 1.98 to 1.73 reflects increased oxidation, which biochemically correlates with the conversion of polysaccharides into simpler sugars. Additionally, the presence of Na, Cl, and Si suggests adsorption of catalyst residues on the biomass surface, which, in some cases, may provide additional active sites to accelerate the reaction (Hurst *et al.* 2021). The fermentation and distillation results demonstrated that pineapple peel waste has substantial potential as a lignocellulosic substrate for bioethanol production, particularly after optimized pretreatment and catalytic hydrolysis. The bioethanol concentration measured by density reflects the successful metabolic activity of microorganisms in converting reducing sugars into ethanol. A bioethanol concentration of approximately 9.07% indicates that the fermentation substrate had a favorable nutrient balance and operational conditions that supported efficient conversion, although the yield remained below the theoretical maximum. This limitation may be associated with the presence of inhibitory compounds generated during pretreatment, such as furfural and organic acids, which can suppress fermentative enzyme activity (Alvita *et al.* 2023).

GC–MS analysis confirmed ethanol as the dominant volatile compound, with a retention time of 1.16 min and molecular ion peak at m/z 46. The Similarity Index (SI = 845, RSI = 851) confirmed strong correspondence with ethanol spectra. The difference between GC–MS (7.9%) and pycnometer (9.07%) results indicates that GC–MS detects only volatile components, whereas the pycnometer method accounts for all dissolved substances affecting density. The distilled bioethanol yield of 6.51% demonstrates high efficiency and aligns with other studies using microwave pretreatment of lignocellulosic biomass. For instance, Conesa *et al.* (2016) reported that microwave pretreatment of pineapple industrial residues under optimized power and time conditions significantly improved fermentable sugar production and enhanced fermentation efficiency within the 7–10% range, although they did not explicitly report final ethanol concentrations. Furthermore, Mikulski & Kłosowski (2023) reported that microwave pretreatment combined with high pressure can increase ethanol yield by up to 30% across various lignocellulosic biomass types, thereby confirming the effectiveness of this method.

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