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Production of Hydrogen and Methane from Pyrolysis of Corn Cob Biomass Using Sodium Bentonite Catalyst

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Abstract

This study investigates the production of methane (CH₄) and hydrogen (H₂) from the pyrolysis of corn cob biomass using an ex situ method, comparing the performance with and without a sodium bentonite catalyst. The raw material, corn cob waste, is ground, dried, and sieved using a 40-mesh screen. Pyrolysis is conducted at temperatures up to 550°C, with cleaned and dried sodium bentonite serving as the catalyst. The characterization of the catalyst includes swelling tests, BET, SEM, and DTA/TGA analyses to determine its physical and chemical properties. The results indicate that the use of sodium bentonite catalyst increases H₂ production, reaching a peak of 842 ppm at 489 seconds and 442°C, compared to 656 ppm at 378 seconds and 458°C without the catalyst. Sodium bentonite accelerates the reaction rate, reduces activation energy, and minimizes tar formation, thereby improving the efficiency and quality of the resulting syngas. The study demonstrates that sodium bentonite is effective in enhancing hydrogen production from biomass pyrolysis, offering an environmentally friendly and economical solution for industrial applications.

Keywords: corncob biomass, pyrolysis, sodium bentonite, methane, hydrogen.

1. INTRODUCTION

The development of biomass-based renewable energy continues to advance in order to meet the increasing energy demands. Biomass is a collective term for gases derived from living organisms, primarily composed of cellulose, hemicellulose, and lignin. Biomass sources include plants, forest vegetation, photosynthetic algae microbes, and other types (mainly referring to biomass waste, including agricultural waste, forestry waste, and urban waste) [1]. Corn cobs are a highly abundant agricultural waste, particularly in countries like Indonesia, which is the highest corn producer among ASEAN countries, but they have not been widely utilized as a value-added [2], [3]. Anukam et al. [4] found that the percentage weight of properties of corn cobs such as moisture, volatile matter, and fixed carbon content, as well as its three main elemental components (C, H, and O), indicate that corn cob biomass can be used as a raw material for renewable energy. The conversion of biomass into energy typically employs four main methods: co-combustion, biochemical fermentation, pyrolysis, and gasification [5], [6].

Pyrolysis is the thermochemical conversion of organic materials into syngas, which is rich in methane and hydrogen, in the absence of oxygen. The pyrolysis process has parameters such as final temperature, pressure, heating rate, and residence time, which significantly affect the quality of the product [7]. Biomass pyrolysis requires significantly high temperatures to optimally decompose the biomass. The pyrolysis process generates syngas, which contains a high concentration of methane (CH₄) and hydrogen (H₂), as well as tar and other complex compounds that impair the quality of syngas with low hydrogen content [8]. Therefore, the addition of catalysts is necessary to enhance the reaction rate, lower the operating temperature, minimize tar formation, and improve the efficiency and quality of the produced syngas [9].

The type of catalyst significantly influences the syngas production from biomass pyrolysis. Mineral-based catalysts are a particular choice in biomass pyrolysis due to their various benefits, especially in terms of efficiency and environmental friendliness, such as dolomite, zeolite, and bentonite. Bentonite can reduce tar formation and complex by-products, lower temperatures, accelerate reactions, increase product selectivity, and has thermal stability [10]. The acidic properties of bentonite help break down complex organic molecules into simple gases, increasing biomass conversion efficiency. As an abundant and non-toxic natural material, bentonite offers an environmentally friendly and effective catalytic solution, making it a superior choice compared to some metal catalysts [11]. Sodium bentonite, in particular, has a higher expansion capacity and better ion exchange ability, making it highly effective in enhancing reactivity and final product quality in the pyrolysis process [12]. Its highly

absorptive properties help reduce tar formation and complex by-products, producing cleaner and higher quality syngas. Sodium bentonite is also thermally stable, resistant to high temperatures, easily available, and economical, making it an efficient and environmentally friendly choice in improving the efficiency and yield of biomass pyrolysis [13].

The use of catalysts in the pyrolysis process can be conducted using two methods: in situ, where the catalyst is in direct contact with the biomass, and ex situ, where the catalyst is placed separately from the biomass. The ex situ method involves transferring the pyrolysis vapor to a secondary reactor for catalytic upgrading, which can enhance tar reduction efficiency and product quality [14]. The properties of ex situ char, such as cooling rate, can be adjusted to optimize performance in processes like oxy-steam combustion [15]. The aim of this study is to determine the characteristics of sodium bentonite and to assess its impact on the biomass pyrolysis process of corn cobs using the ex situ method on the production of CH₄ and H₂.

2. RESEARCH METHODS

2.1. Materials

The corn cobs used in this study were sourced from corn farmers in Malang Regency, East Java. They were ground, sieved using a 40-mesh screen, and then dried in an oven at 150°C for 2 hours to reduce the moisture content to 9%. The particle properties of the corn cobs are shown in Table 1. The catalyst used in this study, sodium bentonite, was obtained from a chemical store in Malang. Sodium bentonite powder was cleaned with distilled water and stirred with a magnetic stirrer for 1 hour at 300 rpm. After cleaning, the bentonite was dried in an oven at 120°C for 2 hours, then ground and sieved to pass through a 325-mesh screen. The chemical composition of sodium bentonite is presented in Table 2.

Table 1. Chemical properties of Corn Cob Particles [16].

Chemical Properties	
Ash content	1,33%
Lignin	35,2%
Cellulose	41,5%
Hemicellulose	13,0%
Others	8,97%

Table 2. Chemical Composition of Sodium Bentonite [17].

Component	(%)
Silicon dioxide (SiO ₂)	58,54
Aluminum oxide (Al ₂ O ₃)	14,73
Calcium carbonate (CaCO ₃)	9,95
Iron (III) oxide (Fe ₂ O ₃)	5,09
Magnesium oxide (MgO)	3,18
Sodium oxide (Na ₂ O)	1,76
Potassium oxide (K ₂ O)	1,25
Sulphur trioxide (SO ₃)	0,92
Titanium dioxide (TiO ₂)	0,72
Other minerals	3,86

2.2. Catalyst Characterization

The catalyst characterization involved testing to determine the physical, chemical, and morphological properties of sodium bentonite. The tests conducted included swelling index, Brunauer-Emmett-Teller (BET), Scanning Electron Microscope (SEM), and differential thermal analysis (DTA) / thermogravimetric analysis (TGA). The swelling index test, conducted according to ASTM D5890 standards, assessed the expansion capability of the bentonite when in contact with water, serving as an indicator of its quality, with higher values indicating better quality [18]. In this test, 2 grams of bentonite were slowly introduced into a graduated cylinder containing 100 mL of distilled water until settled at the bottom. The sample was left for 24 hours, and the expansion of sodium bentonite was recorded.

SEM, an electron microscope used to investigate the surface of solid objects, has a magnification range of 10 to 300,000x, a depth of field from 4 to 0.4 mm, and a resolution from 1 to 10 nm. SEM testing was conducted using

an Inspect S50 FEI to analyze the size, shape, and phase distribution, as well as the chemical composition of the sodium bentonite catalyst.

Differential Thermal Analysis (DTA) and Thermogravimetric Analysis (TGA) were performed using a DTG-60/Shimadzu. DTA measures the temperature difference between a sample and a reference material as a function of time or temperature, while TGA measures weight variation as a function of heating temperature. These characterizations determine the weight loss (vapor emission) or weight gain (gas fixation) of the material sample. These techniques are commonly used to assess sample purity, decomposition behavior, thermal degradation, and chemical reactions involving weight changes due to adsorption, desorption, and chemical kinetics.

2.3. Pyrolysis Testing

Pyrolysis testing was conducted using the experimental setup shown in Fig. 2. The pyrolysis reactor (1) is made of stainless steel with a diameter of 8 inches and a height of 5 inches. Before the pyrolysis process, nitrogen was flowed at 1000 mL/min for 10 minutes to create an inert atmosphere (2). Inside the reactor, 100 grams of corn cobs were placed (3). Heating was performed using an LPG (Liquefied Petroleum Gas) burner at a constant rate until reaching 550°C (4). The sodium bentonite catalysts were positioned above the reactor to ensure that the volatile gases from the pyrolysis reaction passed through the catalyst (5). The gas then flowed through a stainless steel pipe with a 10 mm diameter and a length of 60 cm (6), passing through a spiral pipe to lower the gas temperature (7), and into a collection tube placed in an ice bath to cool and condense the water vapor (8). A type-K thermocouple (9) was installed inside the reactor to monitor the temperature distribution during pyrolysis. The pyrolysis gas passed through pre-calibrated methane (MQ-4) and hydrogen (MQ-8) gas sensors (10), which were placed at the outlet of the third tube to measure the concentrations of methane and hydrogen gases produced during the pyrolysis process. An Arduino Uno (11) was used as an interface to send temperature, methane, and hydrogen concentration data to a computer (12). After the testing, the reactor was cooled to room temperature and cleaned of pyrolysis residue. The tests were conducted with variations including no catalyst and sodium bentonite catalyst.

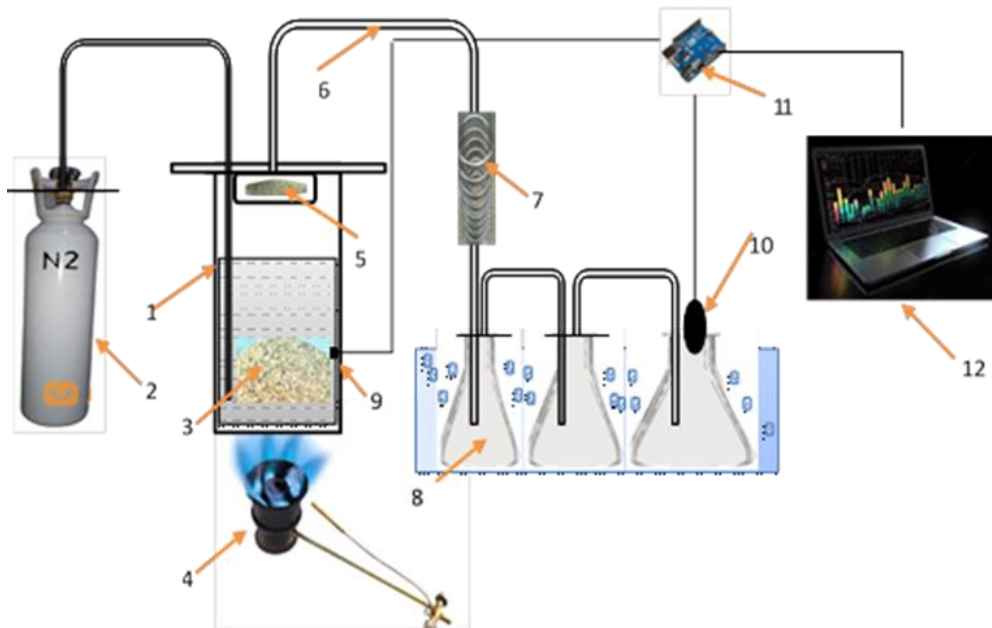


Fig. 1. Experimental setup for the pyrolysis of corncob biomass.

3. RESULTS AND DISCUSSION

3.1. Characteristics

a. Swelling Index

The calculation results show that the swelling index is 40 ml/g, indicating that sodium bentonite has excellent water absorption capacity and significant swelling ability. Sodium bentonite contains sodium ions located between the layers of montmorillonite, which can exchange places with other ions in the solution through a process known as cation exchange. When sodium bentonite comes into contact with water, the sodium ions exchange with

hydrogen ions (H⁺) or other ions in the water, causing water to enter the layer structure. Sodium bentonite can expand significantly due to its layered crystal structure, cation exchange capacity, hydrophilic properties, and the osmotic pressure generated by water absorption.

The swelling index test results indicate that sodium bentonite can increase the active surface area available for catalytic reactions, allowing more biomass molecules to interact with active sites on the catalyst. This can also assist in the formation of micro and mesopores, enhancing catalyst activity at lower temperatures and thereby reducing energy requirements during the pyrolysis process [19]. The swelling index test procedure is illustrated in Fig. 2.

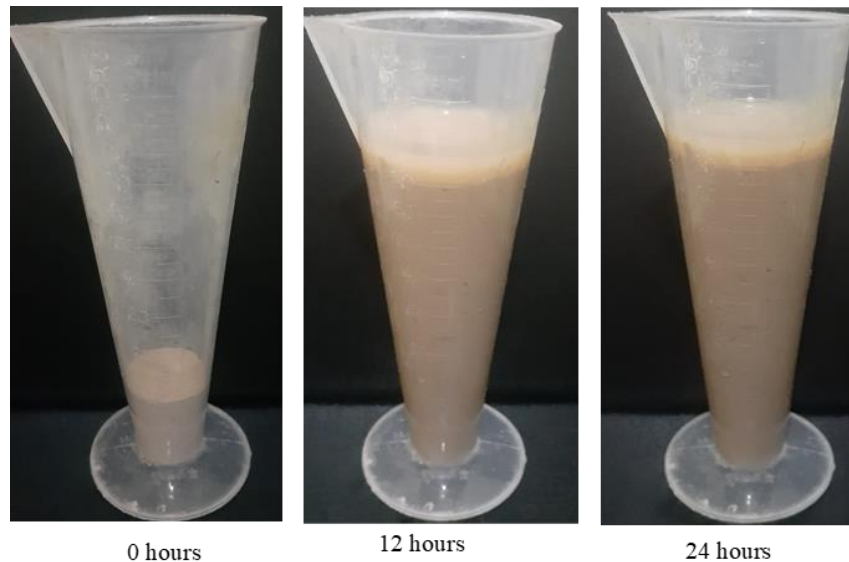


Fig. 2. Swelling Index Testing Process

b. BET Testing

The Brunauer-Emmett-Teller (BET) plot in Fig. 3. Illustrates the linear relationship between the relative pressure (P/P_0) and the amount of adsorbed sodium bentonite. The data points align along the appropriate line, indicating that the BET model is suitable for analyzing this adsorption isotherm. This plot spans relative pressures from 0.05 to 0.25, suggesting that the monolayer adsorption capacity is assessed within this range. The linear trend confirms the validity of the BET equation in describing the surface area and porosity characteristics of the material [20]. Compared to similar studies, the observed linearity in the BET plot of sodium bentonite aligns with previous findings where the BET model was successfully applied to various porous materials. In contrast, deviations from linearity often indicate the presence of micropores or non-uniform surface energy. The lack of significant deviations in the current plot suggests a homogeneous surface with uniform pore sizes for the sodium bentonite sample, unlike findings on heterogeneous materials where BET plots exhibit nonlinear characteristics.

The BET results indicate that the material has a high surface area (54.1263 m²/g), which is ideal for catalytic reactivity. The high C value suggests strong gas adsorption on the catalyst surface, indicating a high affinity for nitrogen gas. A high BET constant indicates strong interactions between the material surface and gas molecules [21]. The BET testing results for sodium bentonite demonstrate physicochemical properties well-suited for use as a catalyst in biomass pyrolysis processes for syngas production. The large surface area, high monolayer volume, and strong affinity for adsorbent gases all contribute to increased efficiency in converting biomass to cleaner, higher-quality syngas. Additionally, the thermal stability and good pore structure ensure that the catalyst can be effectively used under harsh pyrolysis conditions, significantly enhancing syngas output [12].

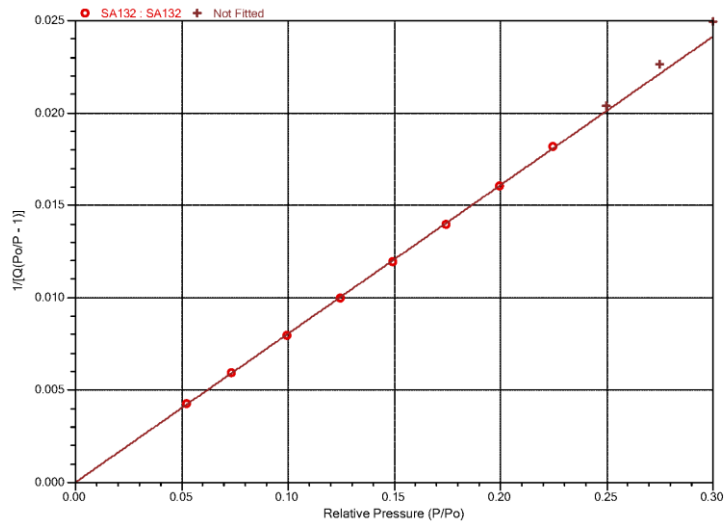
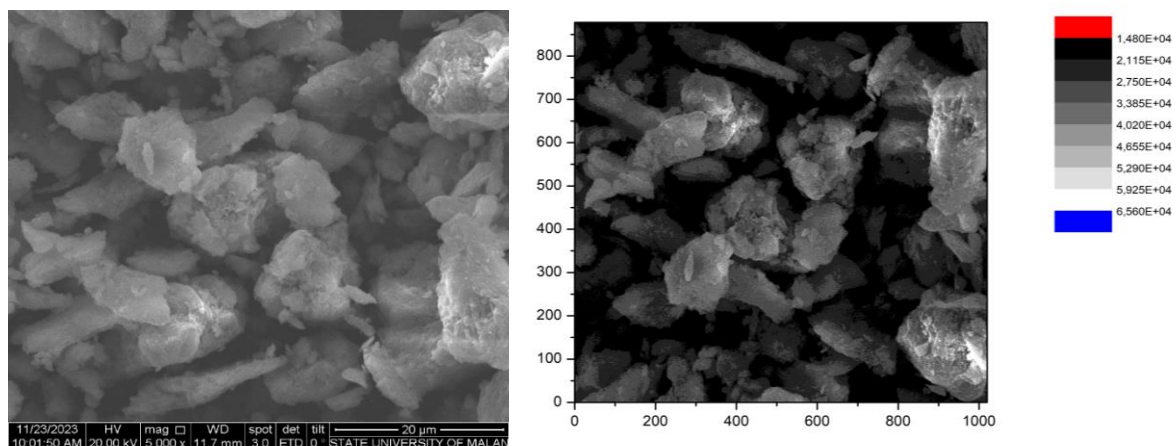


Fig. 3. Sodium Bentonite BET results

c. Morphological Analysis from SEM Testing

The SEM test results of sodium bentonite are presented in Fig. 4. Fig. 4(a). Reveals an irregular and layered structure, typical of sodium bentonite. The particles appear plate-like, with sizes around 20 micrometers. The rough and cracked surfaces of these particles indicate a high adsorption capacity and potential for swelling when hydrated. This layered structure provides a large surface area, supporting both adsorption capacity and chemical reactivity. These characteristics make sodium bentonite highly useful in industrial applications such as drilling mud, foundry sand, and as a binder in various processes. Its adsorption properties also make it effective in waste treatment and environmental remediation. Overall, the morphology and texture of sodium bentonite, as revealed through SEM images, underscore its potential in various industrial and environmental applications [22], [23].

Fig. 4(b) shows the porosity of sodium bentonite and the pore distribution within the material, indicated by varying color intensities on the adjacent color scale. This scale ranges from intensity values of 1.480E+04 (red) to 6.560E+04 (blue), depicting variations in material density across different areas. Lighter areas (e.g., gray to white) indicate lower porosity or denser material, while darker areas indicate higher porosity or more hollow material. This distribution aligns with the layered structure observed in the SEM image, showing numerous gaps and spaces between layers, reflecting high porosity. This high porosity is crucial in applications such as adsorption and sealing, as it allows the material to effectively absorb and retain fluids or gases. Additionally, it supports the bentonite's ability to swell when hydrated, enhancing its effectiveness in waste treatment and environmental remediation. In conclusion, the SEM images and porosity collectively illustrate the functional potential of bentonite in various industrial and environmental applications [24].



(a)

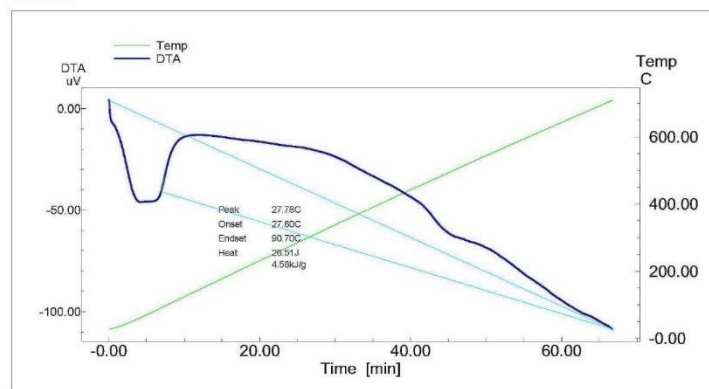
(b)

Fig. 4. Test Results: (a) SEM of Sodium Bentonite, (b) Porosity

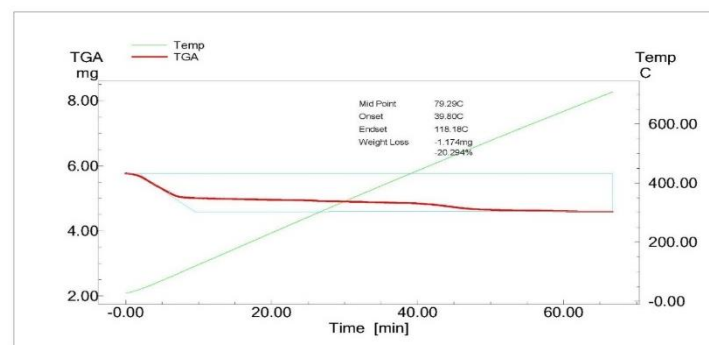
d. DTA/TGA Test Results

The DTA and TGA analysis results of sodium bentonite weighing 5.785 mg are presented in Fig. 5. In Fig. 5(a), the DTA results show a heat change beginning around 27.60°C, indicating the onset of an endothermic or exothermic process. A peak in heat change occurs at 27.78°C, signifying a major reaction or physical change in the sample, with the main heat change process concluding by 90.70°C. The enthalpy change of 26.51 Joules or 4.58 kJ/g reflects the energy involved in this process. These results indicate that sodium bentonite undergoes significant heat changes between 27.60°C and 90.70°C, with a peak at 27.78°C. These changes may be related to dehydration or chemical reactions that affect the structure and catalytic activity. For biomass pyrolysis applications, thermal stability up to these temperatures and the associated heat changes are crucial to ensure the catalyst remains active and effective in optimizing pyrolysis yields. The enthalpy analysis provides insights into the energy interactions between the catalyst and biomass during the pyrolysis process [25].

Fig. 5(b), the TGA results show a weight loss beginning around 39.80°C, likely due to the evaporation of water or other volatile substances adsorbed on the material's surface. By 79.29°C, most of these volatiles have evaporated, resulting in a more stable weight. The main evaporation or decomposition process concludes by 118.18°C, indicating that most volatiles have been released from the sample. The weight loss of 1.174 mg, or 20.294% of the initial sample weight, indicates a significant volatile content in the material [25]. This data is crucial for determining the thermal stability of the material. The temperature range of weight loss indicates critical points where the material undergoes significant changes. The significant weight loss shows the sample contains a considerable amount of volatiles or adsorbed water. The TGA results indicate that sodium bentonite exhibits significant volatility up to 118.18°C, which is important for its application as a catalyst in ex-situ biomass pyrolysis. At this temperature, the release of volatiles allows sodium bentonite to remain stable and active. The weight loss of 20.294% demonstrates the material's ability to adsorb volatile substances from biomass. Thermal stability up to this temperature ensures that sodium bentonite can effectively facilitate the decomposition of biomass into desired products, such as biochar, bio-oil, and pyrolysis gas, enhancing the efficiency and yield of the pyrolysis process [26].



(a)



(b)

Fig. 5. Sodium Bentonite Test Results: (a) DTA, (b) TGA

3.2. Production of CH₄ and H₂ from Corn Cob Pyrolysis

a. Production of CH₄

The results of CH₄ production from the pyrolysis of corn cob biomass are presented in Fig. 6. This figure compares the performance of the ex-situ pyrolysis process with and without the sodium bentonite catalyst, examining methane (CH₄) production as a function of time and temperature. The graph depicting CH₄ production over time shows that CH₄ production increases more rapidly and reaches higher levels when using the sodium bentonite catalyst, peaking at 569 ppm at 448 seconds. In contrast, without the catalyst, CH₄ production peaks at approximately 442 ppm at 472 seconds. The graph illustrating CH₄ production versus temperature reveals a similar trend, with the catalyst enabling faster and higher CH₄ production, reaching a peak of around 569 ppm at approximately 421°C. Without the catalyst, CH₄ production peaks at around 442 ppm at a temperature of 423°C.

The increased CH₄ production with the sodium bentonite catalyst aligns with literature findings demonstrating the effectiveness of clay minerals in catalytic processes. The structure of bentonite, characterized by its high surface area and active sites, facilitates the decomposition of hydrocarbons at lower temperatures, leading to increased methane production [25]. Additionally, the stability of the bentonite catalyst at high temperatures supports sustained and efficient reactions, as evidenced by the stable CH₄ production over time. Previous studies have also highlighted bentonite's superior catalytic properties in hydrocarbon pyrolysis reactions.

The results of this study underscore the significant potential of sodium bentonite as an effective and economical catalyst for enhancing methane production from corn cob biomass pyrolysis. The notable increase in CH₄ production, both in terms of time and temperature, suggests that sodium bentonite can improve the efficiency of energy production processes, which is crucial for industrial applications [27]. The catalyst's efficiency at lower temperatures also indicates potential energy savings and reduced operational costs, making this method more economical. These findings open opportunities for further research to optimize the catalytic properties of bentonite and other clay minerals, potentially leading to more sustainable and efficient industrial processes.

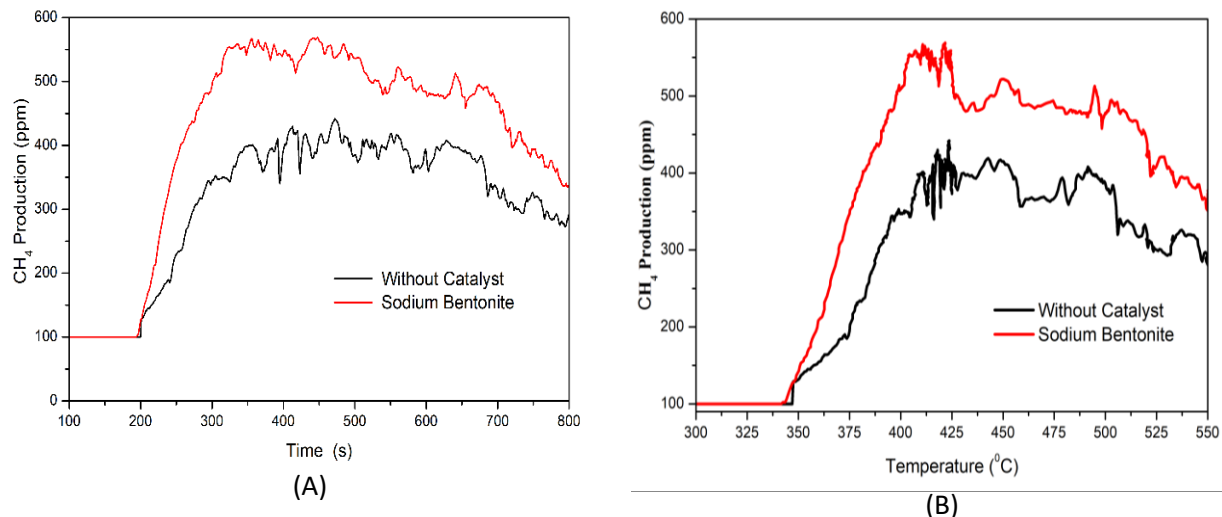


Fig. 6. CH₄ Production: (A) Effect of Time, (B) Effect of Temperature

b. Production of H₂

The hydrogen (H₂) production results from the ex situ pyrolysis of corncob biomass are presented in Fig. 7. This figure compares the performance of the pyrolysis process with and without the presence of a sodium bentonite catalyst. The graph showing the relationship between H₂ production and time reveals that H₂ production increases more rapidly and reaches a higher level when using the sodium bentonite catalyst, peaking at 842 ppm at 489 seconds. Conversely, without the catalyst, H₂ production peaks at 656 ppm at 378 seconds. The graph illustrating the relationship between H₂ production and temperature shows a similar trend, with higher and faster H₂

production peaking at 842 ppm at 442°C with the sodium bentonite catalyst. Without the catalyst, H₂ production peaks at around 656 ppm at 458°C.

The sodium bentonite catalyst accelerates the pyrolysis reaction rate, lowers the activation energy, and enables hydrogen production in a shorter time and at a lower temperature. The catalyst enhances the effectiveness of mass and energy transfer in the pyrolysis process, allowing more biomass molecules to decompose into hydrogen gas. Sodium bentonite acts as a promoter in the thermal decomposition of biomass, speeding up the formation of free radicals that play a role in breaking chemical bonds within organic molecules [28]. The catalyst also provides active sites that support more efficient reforming and cracking reactions, resulting in greater hydrogen production. Additionally, using a sodium bentonite catalyst helps minimize tar compound formation and increases selectivity toward hydrogen production, making it an effective choice for enhancing the efficiency of biomass pyrolysis processes. The significant increase in H₂ production, both in terms of time and temperature, indicates that sodium bentonite can enhance the efficiency of energy production processes, which is crucial for industrial applications.

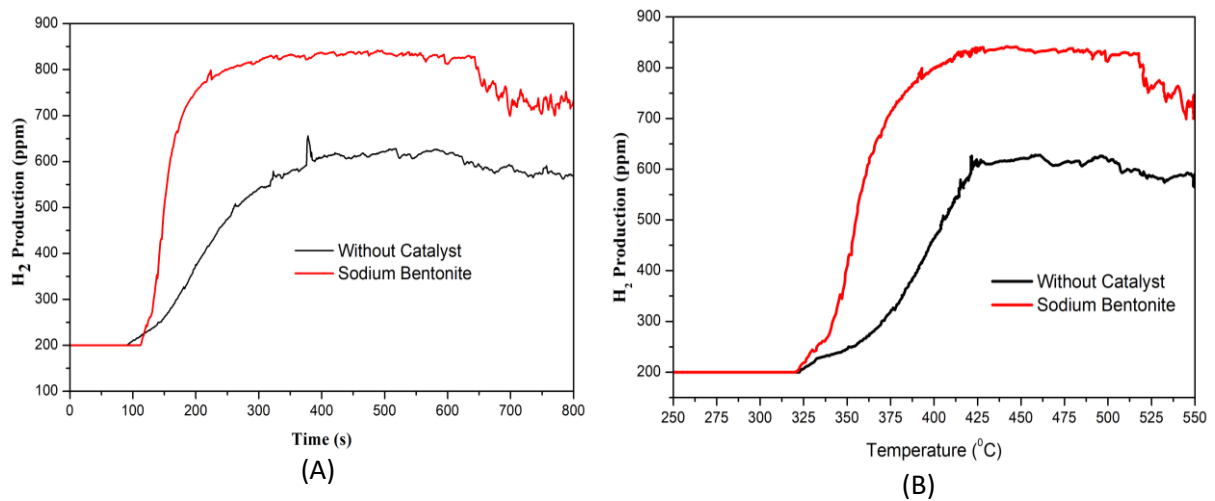


Fig. 7. H₂ Production (A) Effect of Time (B) Effect of Temperature

4. CONCLUSION

The addition of a sodium bentonite catalyst in the pyrolysis process has been proven to enhance the production of hydrogen (H₂) and methane (CH₄) gases. With this catalyst, H₂ production peaked at 842 ppm at 489 seconds and 442°C, whereas without the catalyst, the peak was only 656 ppm at 378 seconds and 458°C. Similarly, CH₄ production increased more rapidly and reached higher levels with the catalyst, peaking at 569 ppm at 448 seconds and 421°C, compared to 442 ppm at 472 seconds and 423°C without the catalyst.

The sodium bentonite catalyst accelerates the pyrolysis reaction rate, lowers the operating temperature, and minimizes tar formation, thereby improving the efficiency and quality of the resulting syngas. This catalyst also enhances the effectiveness of mass and energy transfer and provides active sites that support more efficient reforming and cracking reactions. With its environmentally friendly, thermally stable, and economical properties, sodium bentonite is an effective choice for improving the efficiency of biomass pyrolysis processes, which is crucial for industrial applications.

ACKNOWLEDGEMENT

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