



THE CONVERSION OF CELLULOSE TO GLUCOSE USING NOBLE METAL PLATINUM SUPPORTED ON ZEOLITE

(Penukaran Selulosa kepada Glukosa Menggunakan Mangkin Logam Adi Platinum Disokong pada Zeolit)

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Received: 15 September 2023; Accepted: 31 March 2024; Published: 29 June 2024

Abstract

Today, the chemical industry faces tremendous pressure to make breakthroughs in green materials, biofuels, and sustainable chemicals. In fact, the goal is to move industrial methods and technologies toward those that are cost-effective and environmentally friendly. Chemical transformation of renewable resources is essential to ensure the long-term sustainability of chemicals and to make sure that energy is sufficient for society in the future. The focus of this study was on the conversion of cellulose to glucose using platinum supported on zeolite as a catalyst. Wet impregnation method was employed to synthesize the catalyst, which was then characterized by thermogravimetric analysis (TGA), Fourier transform infrared (FTIR) spectroscopy, and Brunauer-Emmett-Teller (BET) analysis. According to the catalyst characterization performed using TGA, the nitrate precursors of the catalyst decomposed at temperatures between 200 and 280 °C. The finding was verified by FTIR, which showed a diminishing intensity of the peak around 1450–1300 cm⁻¹, indicating that the catalyst was free of precursors. Subsequently, the catalyst was calcined at 500 °C for 5 h. It is noteworthy that this catalyst has a large surface area of up to 648.13 m²/g. A catalytic reaction study on the conversion of cellulose to glucose was carried out to investigate several factors, including substrate loading and catalyst loading. It was discovered that 0.4 g of cellulose and 0.1 g of platinum supported on zeolite contributed to a high yield of glucose and conversion of cellulose (22.07% and 54.9%, respectively). The study shows that the chemical transformation of cellulose to glucose is possible with the supported noble metal catalyst.

Keywords: cellulose, glucose, heterogeneous catalyst, platinum supported on zeolite, supported noble metal catalyst

Abstrak

Industri kimia pada masa kini menghadapi tekanan yang sangat besar untuk melakukan penemuan dalam bahan hijau, bahan bakar-bio, dan bahan kimia yang mapan. Malah, matlamatnya adalah untuk menggerakkan kaedah dan teknologi perindustrian ke arah yang kos efektif dan mesra alam. Transformasi kimia bagi bahan boleh diperbaharui adalah penting untuk memastikan keberlanjutan jangka panjang bahan kimia dan tenaga yang mencukupi untuk masyarakat pada masa hadapan. Oleh itu, fokus kajian ini adalah kepada penukaran selulosa kepada glukosa menggunakan platinum yang disokong pada zeolit sebagai mangkin. Kaedah pengisitepuan basah digunakan untuk mensintesis mangkin, dan ia kemudiannya dicirikan oleh analisis termogravimetri

(TGA), spektroskopi inframerah transformasi Fourier (FTIR), dan analisis Brunauer-Emmett-Teller (BET). Menurut pencirian mangkin yang dilakukan menggunakan TGA, prekursor nitrat daripada mangkin boleh diuraikan pada suhu antara 200 dan 280 °C. Penemuan ini telah disahkan oleh FTIR yang menunjukkan keamatan puncak yang semakin berkurangan sekitar 1450–1300 cm⁻¹, menunjukkan mangkin adalah bebas daripada prekursor. Seterusnya, mangkin dikalsin pada 500 °C selama 5 jam. Selain itu, mangkin ini didapati mempunyai luas permukaan yang besar sehingga 648.13 m²/g. Kajian tindak balas mangkin mengenai penukaran selulosa kepada glukosa telah dijalankan untuk menyiasat beberapa faktor, termasuk pemuatan substrat dan pemuatan mangkin. 0.4 g selulosa dan 0.1 g platinum yang disokong pada zeolit didapati menyumbang kepada hasil glukosa dan penukaran selulosa yang tinggi (masing-masing 22.07% dan 54.9%). Kajian menunjukkan bahawa transformasi kimia selulosa kepada glukosa menunjukkan potensi dengan pemangkin logam adi yang disokong.

Kata kunci: selulosa, glukosa, mangkin heterogen, platinum disokong pada zeolit, mangkin logam adi sokongan

Introduction

The conversion of cellulose, the most abundant biomass on Earth, into valuable chemicals such as glucose, has gained significant attention in recent years due to its potential as a sustainable alternative to traditional fossil fuel-based processes [1]. The preference for utilizing biomass-derived resources is because these feedstocks do not compete with the food supply [2].

Glucose is utilized as a starting chemical in the chemical industry to produce various valuable compounds through biochemical and chemical processes. It serves as a renewable and abundant source for the synthesis of numerous products, including 5-hydroxymethylfurfural, levulinic acid, ethanol, organic acids (e.g., citric acid and gluconic acid), amino acids (e.g., lysine and glutamic acid), and polyols (e.g., sorbitol and xylitol) [3]. Glucose is also a key precursor in the synthesis of pharmaceuticals, polymers, and bio-based materials. Its versatility and availability make it an important starting material for diverse applications in the chemical industry, contributing to sustainable and environmentally friendly production processes.

Catalytic systems play a crucial role in facilitating this conversion, and researchers are continually exploring new catalysts to enhance the efficiency and selectivity of the reaction. One promising approach is the utilization of supported noble metal catalysts as heterogeneous catalysts, which have demonstrated remarkable performance in various biomass conversion reactions. Previously, homogeneous catalysts such as mineral acids, acidic ionic liquids, heteropolyacids, and Lewis acids (e.g., Al, Zr, and Cr salts) were utilized for cellulose degradation. Despite their high efficiency in

catalyzing cellulose conversion, homogeneous catalysts pose several challenges, including equipment corrosion, high separation costs, and environmental pollution. Although homogeneous catalysts are extensively utilized in industry, they have inherent drawbacks that add complexity to the process. These drawbacks include low stability, difficulties in isolating and separating the final product from the reaction mixture, and the need for recycling expensive noble metals or ligands [4].

Zeolites are crystalline aluminosilicates with well-defined nanoporous structures, which possess both Brønsted and Lewis acid sites, provide a high surface area and shape-selective properties, and offer excellent support for catalytic species [5]. Due to their adjustable acidity, exceptional thermal stability, and impressive shape-selective properties, zeolites have proven effective in dehydrating cellulose in water. The porous nature of zeolites also allows for easy access to reactants and products, facilitating efficient mass transfer. The incorporation of noble metals, such as platinum (Pt), palladium (Pd), and gold (Au) onto zeolite, on the other hand, results in unique catalytic properties, such as high activity, selectivity, and resistance to deactivation, making them ideal candidates for biomass conversion [6].

Several recent journal articles have highlighted the effectiveness of noble metals supported on zeolite catalysts for cellulose conversion. For example, Negoï et al. investigated the catalytic performance of noble metal-supported zeolites in the hydrolysis of cellulose to sorbitol [7]. The authors demonstrated that the catalyst exhibited remarkable activity and selectivity (85%) toward sorbitol production, with a yield of over 50%

under mild reaction conditions. In another study, Ya'Aini et al. employed zeolite as the catalyst for converting glucose to levulinic acid (LA) [8]. The researchers observed that the catalyst exhibited excellent stability and selectivity, achieving a high LA yield of 65% under optimized reaction conditions. The catalyst also demonstrated good recyclability, making it a promising candidate for industrial-scale applications.

These recent findings underscore the potential of noble metals supported on zeolite as heterogeneous catalysts for the conversion of cellulose to glucose. The combination of noble metals' unique catalytic properties with the advantageous features of zeolite support holds great promise for the development of efficient and sustainable processes for biomass utilization. Hence, in this study, we investigated the impact of catalyst and cellulose loading on the conversion of cellulose to glucose, utilizing Pt supported on zeolite. Further research and optimization of these catalyst systems are expected to pave the way for the commercial production of valuable chemicals from cellulose, contributing to the advancement of green chemistry and the transition toward a more sustainable future.

Materials and Methods

Materials

The catalyst was synthesized using platinum (IV) nitrate ($\text{Pt}(\text{NO}_3)_2$) with 15% w/w of Pt and zeolite Y purchased from Sigma-Aldrich. For the reaction work, microcrystalline cellulose (extra pure, 100%) from Acros Organics and glucose (>99.9%) were employed as the substrate and for standard reference calibration purposes, respectively. All the chemicals were of analytical grade and used without further purification unless otherwise stated. Distilled water was utilized for all the experiments.

Catalyst synthesis

The catalyst was synthesized through the wet impregnation method, which aimed to bind the active sites to the support material. $\text{Pt}(\text{NO}_3)_2$ and zeolite Y were employed for catalyst preparation. The objective was to achieve a Pt content of 4 wt.% on the zeolite support. Initially, the zeolite was weighed using an analytical balance and evenly distributed on a Petri dish.

Subsequently, 1.3g of $\text{Pt}(\text{NO}_3)_2$ was diluted with 2 mL of distilled water. The Pt solution was then dropped onto the zeolite sample in four separate batches using a syringe. During each application, the first 0.5 mL of the Pt solution was dropped in a circular motion, and the dispersion was carefully spread using a spatula. These steps were repeated until the entire Pt solution had been used. Following the impregnation process, the catalyst was dried in an oven at 120 °C overnight to eliminate any residual water content. Subsequently, the catalyst underwent calcination in a furnace for a duration of 5 h at a temperature of 500 °C. Finally, the catalyst was allowed to cool in a desiccator and then stored in a vial for future use.

Catalyst characterization

The catalyst underwent characterization through several analytical techniques, including thermogravimetric analysis (TGA), Fourier transform infrared (FTIR) spectroscopy, and Brunauer-Emmett-Teller (BET) analysis. A Mettler-Toledo STARe software system was used for TGA. Approximately 5–10 mg of the catalyst was weighed using an analytical balance and placed in an alumina crucible. The analysis was conducted over a temperature range from ambient (30 °C) to 600 °C, utilizing a heating rate of 15 °C/min and a continuous flow of high-purity air at 20 mL/min. The FTIR analysis was carried out using a Nicolet iS10 FTIR spectroscopy instrument, with a spectral range from 4000 cm^{-1} to 400 cm^{-1} . The spectral resolution was set at 8 cm^{-1} , and five scans were accumulated under an open-beam air background at room temperature. A Micromeritics 3Flex surface characterization analyzer was used to determine the surface area and pore volume of the catalyst. Prior to analysis, the catalyst was degassed for 12 h at 150 °C in nitrogen (N_2).

Reaction study

The pre-reduction of the catalyst was conducted in a 20 mL stainless steel batch reactor. Initially, 0.1g of catalyst and 10 mL of distilled water were loaded into the reactor. The pre-reduction process was carried out under conditions of 1 bar of hydrogen (H_2) at a temperature of 150 °C for a duration of 1 hr. Following the pre-reduction of the catalyst, 0.4g of cellulose was introduced into the reactor in preparation for the

subsequent reaction. The reactor was purged three times with N_2 to remove any residual air. Subsequently, the reactor was placed in an oil bath on a magnetic stirring hot plate. Once the desired temperature was reached, the stirrer and timer were set to initiate the catalytic reaction. The reactor was rapidly cooled by immersing it in a water bath to halt the reaction. The resulting product was collected and subjected to filtration. Liquid products were filtered using cotton wool, while solid products were filtered through filter paper. Following the filtration process, the liquid product was stored in a vial in preparation for high-performance liquid chromatography (HPLC) analysis. For the solid product, the filter paper was dried in an oven for 12 h at 80 °C, and the weights of the filter paper before and after drying were recorded to calculate cellulose conversion.

Product analysis

The liquid products obtained from the reaction were identified and quantified through HPLC with a

refractive index detector. For this analysis, a mobile phase of 0.005 M sulfuric acid with a flow rate of 0.5 mL/min was utilized. The column temperature was maintained at 40 °C throughout the analysis.

Results and Discussion

Catalyst characterization: TGA

Based on the TGA data presented in Figure 1, two distinct weight losses were observed in the temperature ranges of 50–100 °C (I) and 210–280 °C (II) for the catalyst prior to calcination. The initial weight loss can be attributed to the removal of adsorbed water [9, 10], while the subsequent weight loss is associated with the decomposition of nitrates and the elimination of organic compounds [11, 12]. Notably, beyond 300 °C, no additional weight loss was noted, indicating the absence of further decomposition. Consequently, a calcination temperature of 500 °C was selected to ensure complete decomposition of nitrate precursors.

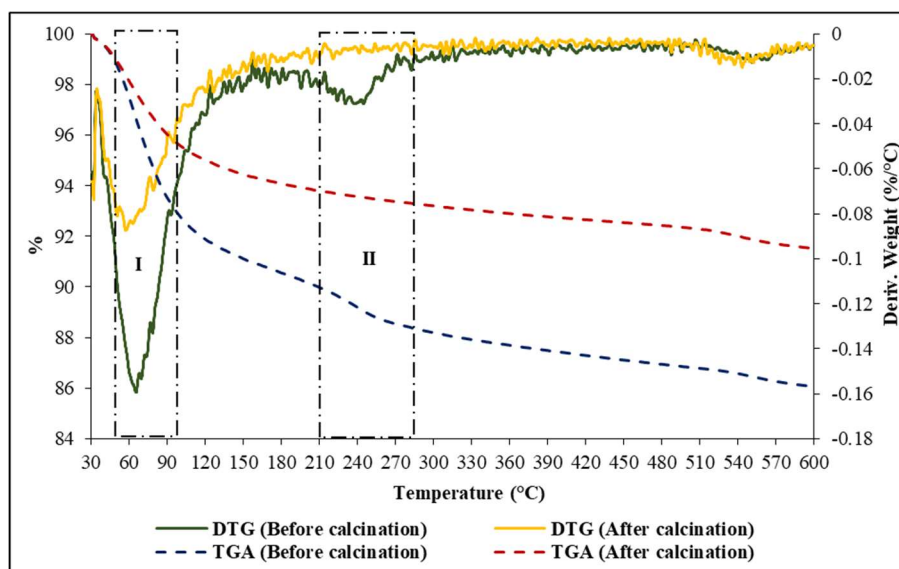


Figure 1. TGA and DTG of Pt/zeolite (before and after calcination)

Following the calcination process, the catalyst exhibited a single weight loss, which occurred within the temperature range of 50–100 °C, as indicated by the TGA pattern in Figure 1. Importantly, no weight loss was detected at temperatures exceeding 100 °C, signifying the complete decomposition of nitrates. Therefore, it is noteworthy that the optimal calcination

temperature for achieving a precursor-free catalyst is 500 °C [13]. This observation aligns with the FTIR analysis results, which reveal the absence of nitrate peaks in the catalyst after calcination.

Catalyst characterization: FTIR

According to the FTIR spectra presented in Figure 2, the

spectral region from 3600 to 3200 cm^{-1} (I) is associated with the stretching and bending modes related to water molecules and their hydrogen bonding [14,15]. The O–H stretch bands observed at 3600–3200 cm^{-1} in the catalyst before calcination appear broader compared to those in the catalyst after calcination, owing to the expansion and contraction absorption of hydrogen bonds. This finding is supported by Jamal et al. [16],

who also observed widened bands in their study. They examined various calcination temperatures and noted that lower temperatures resulted in broader bands at 3600–3200 cm^{-1} . This broadening effect is attributed to a decrease in the peak mode of O–H bond elongation and bending as the calcination temperature rises, which could reduce the absorption of water molecules on the catalyst surface.

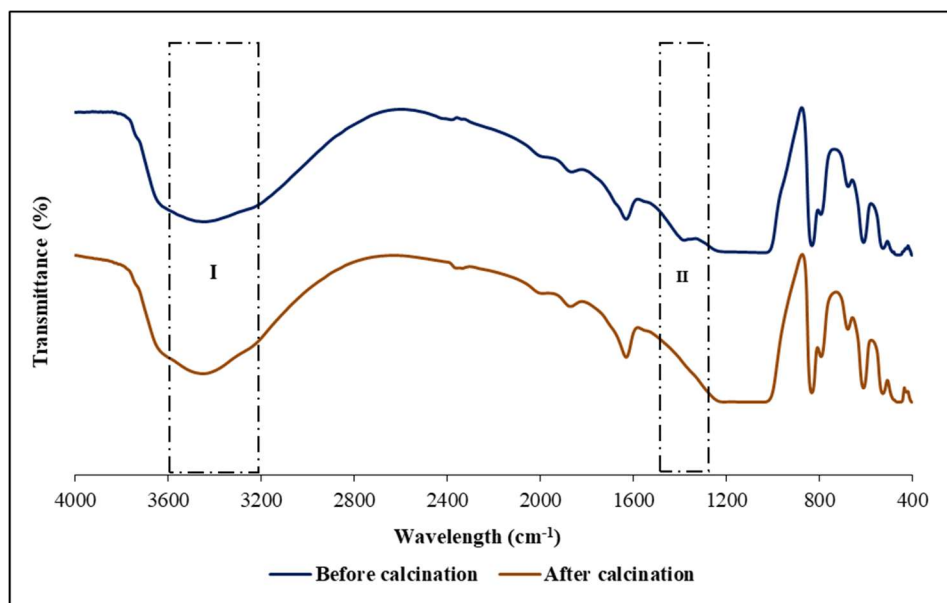


Figure 2. FTIR spectra of Pt/zeolite (before and after calcination)

In contrast, the spectral region between 1450 and 1300 cm^{-1} (II) signifies the presence of nitrate precursor ions (NO_3^-) within the catalyst prior to calcination [15,17]. Significantly, these NO_3^- bands were observed to disappear in the catalyst after undergoing calcination, providing clear evidence that nitrate decomposition was effectively accomplished at the calcination temperature of 500 °C. Consequently, it is important to highlight that the optimal temperature for eliminating the nitrate precursor is 500 °C.

Catalyst characterization: BET

Based on Table 1, the surface area and pore volume of the catalyst before calcination were notably high (665.40 m^2/g and 0.3575 cm^3/g , respectively). However,

these values experienced a slight decrease after catalyst calcination. Nevertheless, the reduction observed in both surface area and pore volume amounted to only 17.21 m^2/g and 0.0128 cm^3/g , respectively. According to the findings by Baharudin et al., this decrease in surface area can be attributed to particle aggregation during calcination [18]. It is worth noting that despite the reduction in surface area and pore volume due to calcination, this process has the potential to maintain the structural stability of the catalyst [19]. Furthermore, it is important to highlight that even after calcination, the catalyst still retains a substantial surface area and pore volume, measuring up to 648.13 m^2/g and 0.3447 cm^3/g , respectively, in comparison to previous research [13].

Table 1. BET surface area and pore volume of catalysts

Sample	Surface Area ^a (m ² /g)	Pore Volume ^b (cm ³ /g)
Pt/zeolite (Before calcination)	665.40	0.3575
Pd/zeolite (After calcination)	648.13	0.3447

^a: Surface area was obtained from the BET method, ^b: Pore volume was obtained from the BET method.

Effect of cellulose loading on the yield of glucose and conversion of cellulose

As illustrated in Figure 3, it becomes evident that as the cellulose loading increases from 0.2g to 0.3g, both the yield of glucose and the conversion of cellulose exhibit a consistent increase. However, when cellulose loading further increased from 0.4g to 0.5g, there is a noticeable decrease in glucose yield and cellulose conversion. This observed trend aligns with the outcomes of prior studies conducted by Joshi et al. and Zhang et al. [12,20]. Several factors may contribute to this condition,

including potential product feedback inhibition, a reduction in reactivity, or an insufficient dosage of catalyst for increased cellulose content. Moreover, higher cellulose loading might increase the likelihood of reactive compounds colliding and initiating cross-polymerization, leading to the formation of undesired products [21]. Consequently, the increase in cellulose loading ultimately results in the saturation of the catalyst's optimal surface area and active site availability.

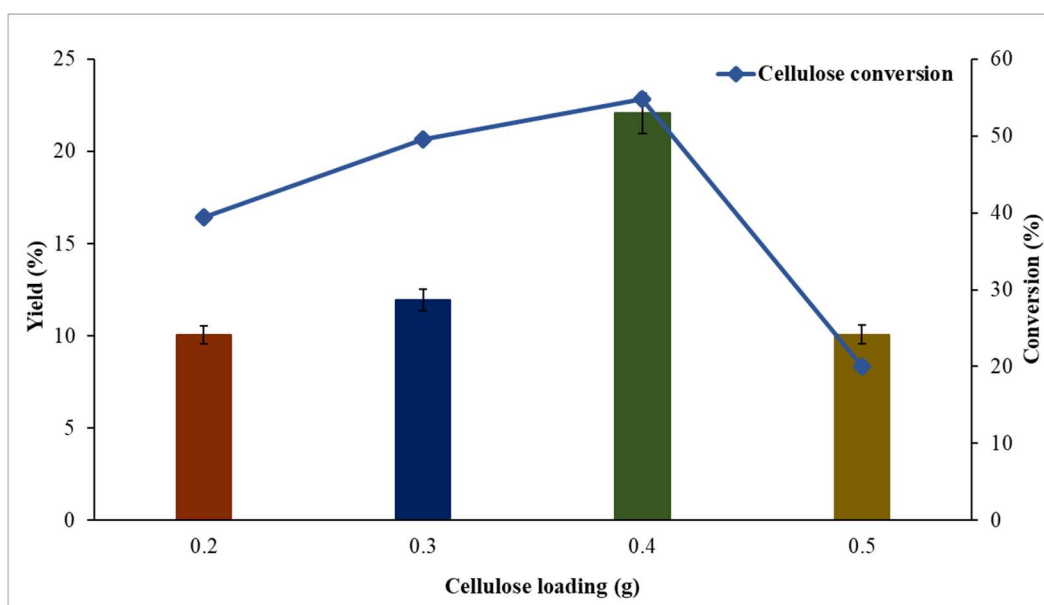


Figure 3. Effect of cellulose loading on the yield of glucose and cellulose conversion at 15 min

Note: a) Pre-reduction of catalyst conditions: 10 mL of distilled water, agitation speed of 1300 rpm, and 1 bar of H₂ for 1 h at 150 °C, b) Reaction conditions: 0.1 g of cellulose, reaction temperature of 210 °C, agitation speed of 1100 rpm, and reaction time of 15 min.

Effect of catalyst loading on the yield of glucose and conversion of cellulose

As shown in Figure 4, the highest cellulose conversion and glucose yield were 54.9% and 22.1%, respectively, when 0.1g of catalyst was employed in the reaction. It is

worth noting that as the catalyst loading increased from 0.04g to 0.1g, both glucose yield and cellulose conversion also exhibited an increasing trend. This condition can be attributed to the increasing availability of active sites on the catalyst, which allows more

cellulose to enter and break down into the desired product at an accelerated rate. The lowest glucose production was observed when using 0.04g of the catalyst due to the limited number of active sites available to efficiently convert cellulose into glucose. This observation aligns with findings from other studies, such as those conducted by Wang et al. [21] and Zhang et al. [20], which demonstrated that increasing catalyst loading favored higher product yields. However, Kamal et al. found an opposite trend, where an increase in catalyst loading led to a decrease in glucose yield and cellulose conversion [22]. This might be attributed to unwanted side reactions occurring at higher catalyst loadings. Additionally, high catalyst loading could increase the viscosity of the reaction mixture, resulting in a slower reaction [23]. This leads to reduced mixing efficiency and increased mass transfer resistance within

the mixture, ultimately limiting the availability of cellulose for the reaction. Consequently, it is important to refrain from adding additional catalyst once the required amount for the reaction has been reached.

The introduction of the catalyst accelerates the progress of the reaction. The use of the catalyst does not alter the equilibrium constant of the reaction; instead, it accelerates both the forward and reverse processes. This is achieved by providing an alternative pathway with a lower energy barrier, thus facilitating the production of the desired product [24]. As a point of reference, a control experiment without the use of a catalyst was conducted under the optimal conditions outlined in this paper. Without the catalyst, the glucose yield and cellulose conversion were at their lowest levels, which were 4.2% and 2.8%, respectively.

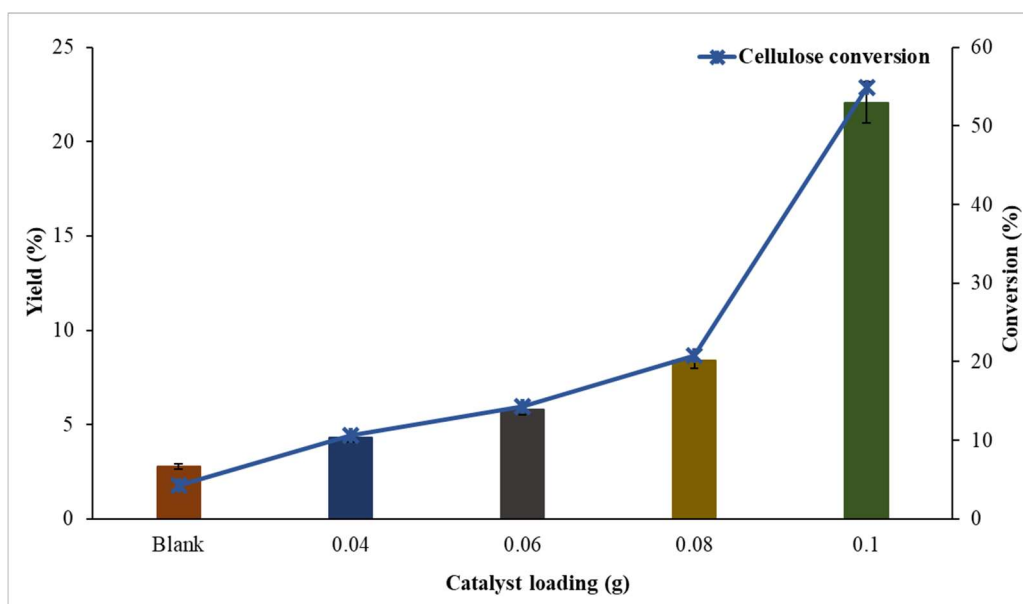


Figure 4. Effect of catalyst loading on the yield of glucose and cellulose conversion at 15 min

Note: a) Pre-reduction of catalyst conditions: 10 mL of distilled water, agitation speed of 1300 rpm, and 1 bar of H_2 for 1 h at 150 °C, b) Reaction conditions: 0.4 g of cellulose, reaction temperature of 210 °C, agitation speed of 1100 rpm, and reaction time of 15 min.

Conclusion

The catalyst synthesis involved $Pt(NO_3)_2$ and zeolite, aiming for a Pt content of 4 wt.% on the zeolite support prepared via the wet impregnation method. Catalyst characterization was performed using TGA, FTIR, and BET analysis. First, TGA was conducted from 30 °C to 600 °C, and the results revealed that the weight losses

are attributable to water and nitrate decomposition. Subsequently, the FTIR analysis detected nitrate bands prior to calcination, which disappeared after calcination, confirming successful nitrate decomposition. Despite minor decreases, the catalyst maintained a substantial surface area and pore volume of up to 648.13 m^2/g and 0.3447 cm^3/g , respectively, affirming its stable structure.

In addition, the influence of cellulose and catalyst loading on glucose yield and cellulose conversion was explored. The results revealed that the optimal conditions for maximizing glucose yield and cellulose conversion were achieved when employing 0.4g of cellulose and 0.1g of catalyst, resulting in yield and conversion of 22.1% and 54.9%, respectively. These findings underscore the important role of catalyst selection and loading in achieving efficient biomass conversion processes, which contribute to the advancement of sustainable and eco-friendly chemical production.

Acknowledgement

The authors would like to express their highest gratitude for the financial support from the Ministry of Higher Education (MOHE) (under the Fundamental Research Grant Scheme, FRGS/1/2018/TK02/UNIKL/02/3), Universiti Kuala Lumpur Malaysian Institute of Chemical and Bioengineering Technology (UniKL MICET), and Majlis Amanah Rakyat Malaysia (MARA).

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