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Application of Palladium Mesoporous Carbon Composite Obtained from a Sustainable Source for Catalyzing Hydrogen Generation Reaction

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Abstract: Alternative fuel sources are necessary in today's economic and environmental climate. Hydrogen fuel arises as an environmentally friendly and energy dense option; however, the volatility of hydrogen gas makes it dangerous to store and utilize. The evolution of hydrogen from hydrogen feedstock materials may prove to overcome this safety barrier, but a catalyst for this reaction is necessary to optimize production. In this work, a composite catalyst comprised of palladium nanoparticles embedded on mesoporous carbon materials (Pd-MCM) was synthesized and characterized by Transmission Electron Microscope (TEM), Powder X-Ray diffraction (P-XRD), Scanning Electron Microscope (SEM) and Energy Dispersive Spectroscope (EDS). Various reaction conditions such as concentration of reactant, temperature, and pH were applied in measuring the catalytic activity of Pd-MCM. Results show the catalytic activity of the Pd-MCM composite catalysts increased with increasing concentrations of sodium borohydride, increasing temperature, and lower pH. The reaction involving the Pd-MCM composite had an activation energy of 27.9 kJ mol⁻¹. Reusability trials showed the Pd-MCM composite remained stable for up to five consecutive trials.

Keywords: palladium nanoparticles; mesoporous carbon; sustainable source; hydrogen production



Citation: Biehler, E.; Quach, Q.; Abdel-Fattah, T.M. Application of Palladium Mesoporous Carbon Composite Obtained from a Sustainable Source for Catalyzing Hydrogen Generation Reaction. J. Compos. Sci. 2024, 8, 270. https:// doi.org/10.3390/jcs8070270

Academic Editors: Yizhu Lei and Francesco Tornabene

Received: 30 May 2024 Revised: 1 July 2024 Accepted: 8 July 2024 Published: 12 July 2024



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1. Introduction

The inevitable depletion of non-renewable energy sources, and the environmental consequences of their continued use, warrants research into cleaner alternative energy sources. One viable solution is the use of hydrogen as a fuel. Alongside being environmentally friendly, hydrogen gas releases three times the amount of energy per kilogram in fuel cells when compared to gasoline [1]. Hydrogen gas, when utilized as a fuel, emits no harmful pollutants or greenhouse gases during combustion, making it a clean and sustainable energy option. Unlike fossil fuels, which release carbon dioxide and other pollutants into the atmosphere, hydrogen combustion produces only water vapor as a byproduct. This characteristic significantly reduces the environmental impact of energy production and helps mitigate climate change. Furthermore, hydrogen boasts an impressive energy density, offering approximately three times the energy content per kilogram compared to gasoline. This high energy density makes hydrogen an efficient and potent fuel source, particularly in applications such as fuel cells. In fuel cell systems, hydrogen undergoes electrochemical reactions to generate electricity, with water as the only byproduct. This process not only produces clean electricity but also offers high efficiency and minimal environmental impact. By harnessing hydrogen as a fuel, we can transition towards a more sustainable and renewable energy future. Its clean-burning properties, coupled with its high energy density and versatility, make hydrogen a key player in addressing the challenges of energy security, environmental sustainability, and climate change mitigation. Continued research

and investment in hydrogen technologies are essential to unlocking its full potential as a cornerstone of the clean energy landscape.

The use of hydrogen gas, however, is limited by the ability to store it safely. This issue can be mitigated via the application of a number of different methods to produce hydrogen on demand rather than store it in large quantities. Perhaps the most common method of producing hydrogen gas is through steam methane reforming. This method involves the reaction of hydrocarbons and water to form a mixture of hydrogen and carbon monoxide or carbon dioxide [2]. A major downside of this method is it requires an energy input of heat, and it releases greenhouse gases in the process. Another method of hydrogen production is through electrolysis, where an electric current is passed through an electrolyte. Like steam methane reforming, this process is energy intensive; however, hydrogen gas is considered to be green hydrogen with no harmful byproducts [3,4]. One way scientists have attempted to reduce the cost of electrolysis is to use energy from the sun to produce hydrogen via a photovoltaic reaction [5]. Similar to regular solar cells, this method suffers from the intermittency of solar energy. Some studies have attempted to combine wind energy with photovoltaic cells to provide energy for hydrolysis when solar is not available; however, this method also suffers from the environmental concerns associated with wind energy [6]. A different way to achieve the hydrogen evolution reaction (HER) without electrolysis is with a hydrogen feedstock material. Generally speaking, a feedstock material is one that contains a high percentage of hydrogen such as ammonia borane (H₃NBH₃) [7]. While this material contains a staggering 19.5% hydrogen by weight, it is not commonly used and expensive to produce. Metal hydrides like Mg(BH₄)₂ and LiBH₄ also contain large percentages of hydrogen by weight and are capable of producing hydrogen; however, they do not meet the DOE's requirements for energy production [8–10]. One promising metal hydride that contains 10.8% hydrogen by weight is known as sodium borohydride(NaBH₄) and produces hydrogen gas steadily, as seen in Equation (1) [11].

$$NaBH_4 (s) + 2H_2O (l) \rightarrow NaBO_2 + 4H_2 (\Delta H < 0)$$
 (1)

Without a catalyst, the HER proceeds slowly in aqueous solution. Recent advances in the field of nanotechnology have significantly enhanced the development of effective catalysts. These breakthroughs have enabled the design and synthesis of catalysts with improved performance, precision, and versatility. By manipulating materials at the nanoscale, researchers have been able to create catalysts with tailored properties, such as increased surface area, higher reactivity, and greater selectivity. These innovations not only enhance the efficiency of catalytic processes but also open up new possibilities for applications in various industries, including energy, environmental remediation, and chemical manufacturing. One notable advancement is the use of mesoporous carbon materials (MCM) [12–14]. These materials are particularly appealing because they are derived from sustainable sources such as corn starch, which makes them an environmentally friendly option. Utilizing renewable resources like corn starch reduces the reliance on non-renewable, fossil fuelbased materials, thereby lowering the carbon footprint associated with their production. Additionally, the use of biodegradable and abundant agricultural byproducts in the synthesis of mesoporous carbon materials supports sustainable agricultural practices and promotes a circular economy. This sustainable approach not only aligns with global efforts to mitigate environmental impact but also enhances the economic viability of producing high-performance catalysts for a variety of industrial applications. The unique structure of mesoporous carbon, characterized by pore sizes ranging from 2 to 50 nanometers, provides a high surface area, which is crucial for facilitating chemical reactions. This high surface area allows for more active sites where reactions can occur, thereby improving the efficiency and effectiveness of the catalytic processes [15,16].

While mesoporous carbon materials (MCM) can function effectively as independent catalysts, their catalytic activity can be significantly optimized by the incorporation of nanomaterials. Nanomaterials, defined as materials with at least one dimension between 1 nanometer and 100 nanometers, can enhance the properties of MCM by increasing the

number of active sites and improving the overall reaction kinetics. The integration of nanomaterials into MCM can lead to a synergistic effect, where the unique properties of both materials combine to create a more efficient and effective catalyst. This can result in higher catalytic activity, greater selectivity for desired reactions, and improved stability under reaction conditions. The use of nanomaterials also allows for fine-tuning of the catalyst's properties, enabling precise control over the reaction environment and further optimizing the catalytic performance for specific applications. Nanoparticles, and two-dimensional materials like nanofilms have a high surface area to volume ratio which increases their catalytic capability [17,18]. Despite this high surface area, nanoparticles have a tendency to agglomerate in solution, thus reducing their catalytic activity [19,20]. The incorporation of nanoparticles into mesoporous carbon materials (MCM) serves to both increase catalytic activity and reduce agglomeration through the formation of a stable nanocomposite. This process enhances the overall performance of the catalyst in several ways. Firstly, the addition of nanoparticles increases the number of active sites available for chemical reactions, thereby boosting the catalytic activity. The nanoparticles, due to their high surface area-to-volume ratio, provide additional reactive surfaces that facilitate more efficient interactions with reactant molecules. Secondly, incorporating nanoparticles helps to mitigate the issue of agglomeration, which is a common challenge in catalyst design. Agglomeration can lead to a decrease in active surface area and negatively impact the efficiency of the catalyst. By forming a stable nanocomposite, the nanoparticles are more evenly dispersed within the mesoporous carbon matrix. This uniform distribution prevents the nanoparticles from clumping together, maintaining the high surface area and ensuring consistent catalytic performance. Furthermore, the stable nanocomposite structure enhances the durability and longevity of the catalyst. The strong interactions between the nanoparticles and the MCM support help to stabilize the nanoparticles, making them less prone to detachment or deactivation during catalytic processes. This stability is crucial for maintaining the catalyst's effectiveness over extended periods and under varying reaction conditions. Overall, the strategic incorporation of nanoparticles into MCM not only amplifies catalytic activity but also enhances the structural integrity and operational stability of the catalyst, making it a highly efficient and reliable choice for various industrial applications.

In this work, palladium nanoparticles are utilized as the nanoparticle portion of the catalyst, while mesoporous carbon materials (MCM) serve as the support structure. Palladium (Pd) is chosen due to its exceptional catalytic properties [21–26], particularly in facilitating hydrogenation reactions. Hydrogenation, a process where hydrogen molecules are added to other compounds, is a fundamental reaction in both organic and industrial chemistry. Palladium is highly effective in this role because of its ability to adsorb hydrogen molecules on its surface, dissociate them into individual hydrogen atoms, and subsequently facilitate their addition to unsaturated bonds in various substrates. The integration of palladium nanoparticles into MCM provides a dual advantage. The mesoporous structure of MCM, characterized by its high surface area and uniform pore size, offers an ideal support matrix that ensures the even dispersion of palladium nanoparticles. This uniform dispersion is crucial for maximizing the exposure of active palladium sites to the reactants, thereby enhancing the overall catalytic efficiency. Additionally, the stability and robustness of the MCM protect the palladium nanoparticles from agglomeration and deactivation, ensuring consistent catalytic performance over time. This synergistic combination of palladium's high catalytic activity and MCM's supportive structure results in a highly effective and durable catalyst system. The catalytic activity of palladium is significantly enhanced when it is utilized in the nanoparticle range, leading to an increased rate of sodium borohydride hydrolysis. This enhancement is primarily due to the nanoparticles' high surface areato-volume ratio, which provides a greater number of active sites for the catalytic process. As a result, reactions occur more efficiently and at a faster rate. After synthesizing the palladium nanoparticle-mesoporous carbon material (Pd-MCM) composite, a series of characterizations were performed to evaluate its structure and properties. These characteri-

zations included techniques such as transmission electron microscopy (TEM) to visualize the dispersion of palladium nanoparticles within the mesoporous carbon matrix, X-ray diffraction (XRD) to confirm the crystalline structure of the nanoparticles, and surface area analysis using BET (Brunauer-Emmett-Teller) method to measure the high surface area provided by the mesoporous carbon. The catalytic properties of the Pd-MCM composite were then rigorously tested under various conditions. Experiments were conducted at different temperatures to determine the optimal thermal conditions for catalytic activity. The pH levels were varied to assess the catalyst's performance across a range of acidic and basic environments, which is critical for understanding its versatility in different chemical processes. Additionally, the concentration of the reactant sodium borohydride (NaBH₄) was adjusted to observe the catalyst's behavior and efficiency at different reactant levels. Finally, the structural stability and reusability of the Pd-MCM composite were evaluated. The catalyst was subjected to multiple cycles of use, specifically being reused five times in a row, to monitor any changes in its performance. This involved measuring the volume of hydrogen produced during each cycle to detect any potential decrease in catalytic efficiency or structural integrity. Consistent hydrogen production across multiple uses would indicate that the Pd-MCM composite maintains its catalytic activity and structural stability, making it a durable and reliable catalyst for practical applications.

2. Experimental

2.1. Synthesis of Materials:

In total, 100 mL of a 1 mM solution of palladium (II) chloride (Sigma Aldrich, St. Louis, MI, USA) was boiled. After that, 5 mL of 1% w/w aqueous sodium citrate (Sigma Aldrich) was slowly added to the solution and stirred until the color change was observed. The obtained palladium nanoparticle solution was centrifuged at 5000 rpm for 10 min to removed unreacted reactants.

The mesoporous carbon was synthesized from environmentally friendly corn starch [12–14,27]. The nanocomposite material was created by combining 40 mL of nanoparticle solution with 1 g of mesoporous carbon. Incipient wetness impregnation was used to allow the catalyst to attach to the mesoporous carbon structure. After filtration of the product, the precipitate was dried for 24 h at $100\,^{\circ}\text{C}$.

2.2. Characterization

Transmission electron microscope (TEM, JEM-2100F, JEOL, Akishima, Tokyo, Japan) was used to show the interaction between nanoparticles and mesoporous carbon. Samples were made by placing 1 μ L of the nanocomposite solution on a TEM sample grid and the grid was allowed to dry overnight in an oven.

Powdered X-ray diffraction (P-XRD, Rigaku Miniflex II, Cu K α X-ray, nickel filters, Rigaku, Tokyo, Japan) was used to determine the crystal structure of Pd-MCM and MCM. The powdered samples were placed on the sample holder and scanned from 10° to 70° .

To determine the elemental composition of the nanocomposite material, Scanning Electron Microscopy (SEM, JEOL JSM-6060LV, JEOL, Akishima, Tokyo, Japan)/Energy Dispersive Spectroscopy (EDS, Thermo Scientific UltraDry, Thermo Fischer Scientific, Waltham, MA, USA)) was used. A powdered nanomaterial was placed on a sample holder and analyzed.

2.3. Catalytic Tests

The catalyst was tested for its catalytic activity by using gravimetric water displacement measurement system [28]. This was accomplished through the use of the catalyst in a hydrogen evolution reaction. In the reaction, 0.01 g of the nanocomposite catalyst and 100 mL of DI water as solvent were used in all trials. Sodium borohydride (NaBH₄) was added and functioned as the source of hydrogen and, upon hydrolysis in the solution, hydrogen gas was generated. Further, magnetic stirring was implemented to maintain an even suspension of the heterogenous catalyst. Using a gravimetric water displacement

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system, the amount of hydrogen gas generated was measured and recorded using Ohaus Pioneer Balance (Pa124) with proprietary mass logging software. Reaction conditions such as NaBH $_4$ concentration (793 µmol, 952 µmol, 1057 µmol), pH (6,7,8), and temperature (273 K, 288 K 295 K, 303 K) were varied to determine optimal conditions. Each specific condition was measured three times and the averages were calculated.

2.4. Catalytic Reusability

The reusability of the catalysts was determined via five catalytic reactions. The NaBH $_4$ reactant was replenished after each subsequent trial. For each trial, 952 μ mol solution of NaBH $_4$ was added to 100 mL of deionized water at pH 7 and 295 K containing 0.01 g of catalyst.

3. Results and Discussion

3.1. Catalyst Characterization

The TEM micrographs in Figure 1 show the mesoporous carbon embedded with palladium nanoparticles (Pd-MCM). No individual palladium nanoparticles could be observed in Figure 1a or Figure 1b; however, in Figure 1c incredibly small nanoparticles can be seen and several examples are circled. The transmission electron microscopy (TEM) images depicted in Figure 1 offer a detailed depiction of the structure of the mesoporous carbon matrix integrated with palladium nanoparticles, denoted as Pd-MCM. Upon examination of Figure 1a,b, individual palladium nanoparticles are not readily discernible. However, in Figure 1c, the magnified view reveals the presence of nanoparticles, with several instances highlighted by encirclement. These nanoparticles hold significant importance within the composite material. Their incorporation into the mesoporous carbon matrix imparts catalytic functionality to the Pd-MCM composite. These nanoparticles serve as active sites for catalytic reactions, facilitating the transformation of reactant molecules into desired products. The circled examples in Figure 1c underscore the presence and distribution of these crucial nanoparticles within the composite structure. Their observation corroborates the successful synthesis of the Pd-MCM composite and validates its potential for catalytic applications. The cumulative effect of these nanoparticles within the composite matrix is instrumental in driving catalytic activity, exemplifying the intricate interplay between structure and functionality in nanomaterial-based catalysts. The method used to synthesize these PdNPs has been previously used by this team and it was found that the nanoparticles generally range in size from 2 to 4 nm in diameter, while Figure 1d indicates the nanoparticles were smaller in this study with an average particle size of 1.5 nm in diameter [28]. Figure 2a shows the SEM micrographs of the Pd-MCM at a scale range of 0.5 micron. Two incredibly large spots can be seen having diameters around 2 microns and much smaller spots can be seen scattered throughout the image. It is likely that these two large spots are agglomerations of nanoparticles. Figure 2b depicts elemental analysis of the SEM micrograph, focusing on the large spots, confirming their identity to be palladium, which supports the agglomeration theory.

Figure 3 shows the P-XRD spectra of the Pd-MCM catalysts. The spectra for the mesoporous carbon without the addition of nanoparticles show that the catalyst possesses a highly crystalline carbon structure due to the sharp peak within the 20° to 30° range. The P-XRD spectra also exhibits a significantly smaller peak within the 40° to 45° range, characteristic of carbon, which is reported in other studies on mesoporous carbon and is indicative of graphitic characteristics [29]. In the composite material, all these peaks were observed to have been shifted up. This indicates that the presence of nanoparticles has changed the lattice structure of the mesoporous backbone and has been observed in similar work [30]. The peaks that are normally associated with PdNPs were not observed; however, this is likely due to the small size of the nanoparticles and limitations of the instrument used.

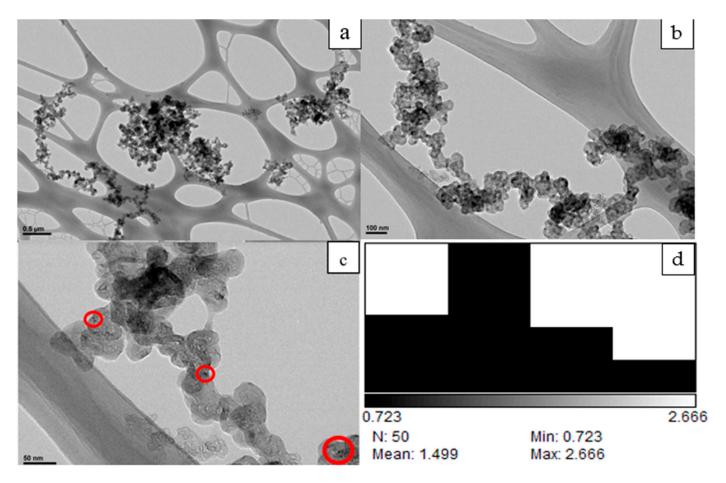


Figure 1. TEM images of the PdNP-MCM catalyst at magnifications of (a) $0.5 \mu m$, (b) 100 nm and (c) 50 nm. A histogram (d) depicts the particle size distribution.

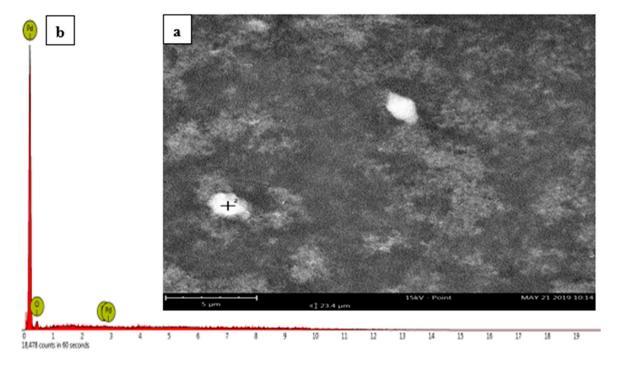


Figure 2. SEM/EDX analysis with **(a)** the SEM micrograph of the Pd-MCM catalyst and **(b)** the EDS spectrum correlating to the indicated palladium nanoparticle.

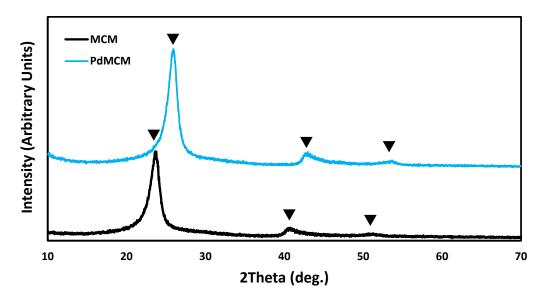


Figure 3. P-XRD Spectra for the Pd-MCM catalyst. Triangles indicated the characteristic peaks of the MCM backbone.

3.2. Catalytic Trials

Before different trials were tested, a set of trials was run at 295 K, pH 7, and using 952 μmol of NaBH4. These trials then became the standard for comparison for all the other reaction conditions. At the standard conditions, after two hours, this reaction produced on average 15.8 mL of hydrogen. The first conditions tested were varying concentrations of NaBH4 as seen in Figure 4. When tested at 793 μmol , deemed low concentration, it was observed that there was less total volume of hydrogen produced after two hours of 12.3 mL. High concentration (1057 μmol) had the opposite effect, with an increase in hydrogen produced up to 51.6 mL after two hours. These results show a positive linear relationship between the concentration of NaBH4 used and the volume of hydrogen produced. This result was expected as based on Equation (1), the equilibrium law states that the increase in NaBH4 would shift the reaction to the right, increasing the amount of hydrogen product.

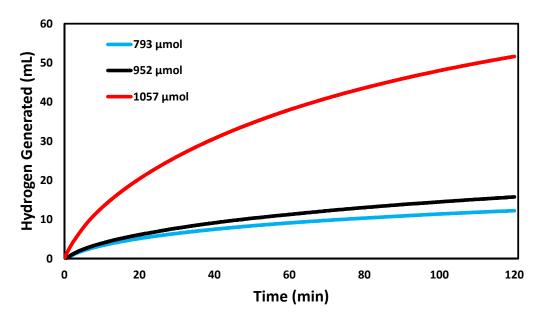


Figure 4. Volume of hydrogen generated versus time in minutes with the Pd-MCM catalyst when varying NaBH₄ concentrations.

Standard conditions were tested against adjusting the pH of the reaction (Figure 5). When the pH was raised from 7 to 8, there was a decline in the amount of hydrogen produced to 12.0 mL. A lowered pH from 7 to 6 resulted in a marked increase in hydrogen generated, to 51.6 mL. The work of Schlesinger et al. in 1953 found that lowering the pH of this reaction results in an increase in the number of hydroxide ions in the reaction which results in more hydrogen released [31].

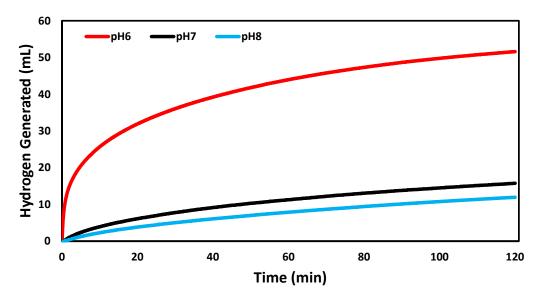


Figure 5. Volume of hydrogen generated versus time in minutes with the Pd-MCM catalyst when varying pH's.

Figure 6 shows the results of the catalytic ability of Pd-MCM when tested at different temperatures. A clear direct relationship between temperature and hydrogen produced was observed with 273 K producing the least amount of hydrogen at only 6.8 mL. At 288 K the volume of hydrogen produced was more than 273 K but still less than 295 K with 10.3 mL. Raising the reaction temperature to 303 K resulted in an average volume of 23.1 mL generated. Based on these results, the equilibrium law, and Equation (1) this reaction was determined to be endothermic.

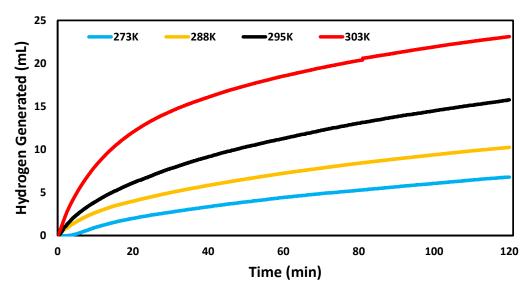


Figure 6. Volume of hydrogen generated versus time in minutes with the Pd-MCM catalyst when varying temperatures.

Thermodynamic Outcomes of Catalysis

In Equation (2), the variables are as follows: k is the reaction rate, A is the preexponential constant, Ea is the activation energy, R is the universal gas constant, T is the temperature the reaction was tested at. Based on the equation of the line from the Arrhenius plot (Figure 7) the activation energy of the reaction as catalyzed by Pd-MCM was calculated to be 27.9 kJ mol⁻¹. This activation energy was then compared against other catalysts for this reaction as seen in Table 1.

 $k = Ae^{-\frac{Ea}{RT}} \tag{2}$

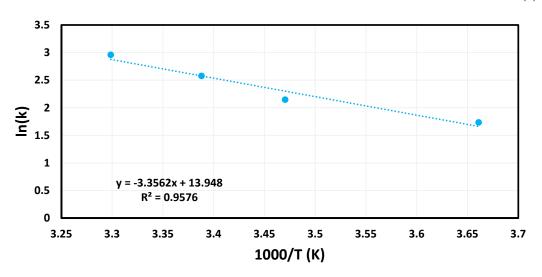


Figure 7. Arrhenius plot for calculating the activation energy of a hydrogen generation reaction for the Pd-MCM catalyst from the reaction rate calculated from the Arrhenius Equation (2).

Table 1. Comparison of	f reported activatior	n energies for catal	yzed NaBH₄ hydrolysis.

Catalyst	E _a (kJ mol ⁻¹)	Temperature (K)	Reference
Ni	71	273–308	[32]
Со	75	273–308	[32]
Co-B/MWCNTs	40	298–333	[33]
Со-В	64.9	283–303	[34]
BCD-AuNP	54.7	283–303	[35]
PtNPs	39.2	283–303	[36]
PtMWCNTs	46.2	2833–303	[37]
AuMWCNTs	21.1	273–303	[38]
Ag/MWCNTs	44.5	273–303	[39]
Pd/MWCNTs	62.7	273–303	[28]
CuGLM	46.8	283–303	[40]
AuFGLM	45.5	283–303	[41]
PtFCS	53.0	283–303	[42]
AgNP-FCS	37.0	283–303	[43]
PdFGLM	45.1	283–303	[44]
Pd-MCM	27.9	273–303	This Work

When compared to similar work, this Pd-MCM shows a competitive advantage. It showed a much lower activation energy when compared to non-precious metal catalysts, with only the cobalt composite Co-B/MWCNTs coming close. Non-composite precious

metal catalysts such as BCD-AuNP and PtNPs have relatively low activation energies but not as low as Pd-MCM. Similar precious metal composite catalysts also showed low activation energies yet only AuMWCNTs had an activation energy lower than Pd-MCM. More recent catalysts explored by this team include precious metals supported on different carbon materials that all had higher activation energies than this study's material. Even compared to other palladium composites like Pd/MWCNTs and PdFGLM, this Pd-MCM catalyst was significantly lower. This suggests that this new catalyst holds an advantage over non-composite materials, and even other composite materials like MWCNTs. This material has an environmental advantage however, as the MCM backbone is made from sustainable corn starch.

3.3. Reusability Tests

The reusability of the Pd-MCM catalyst was investigated through a series of trials to assess its performance over multiple cycles. These trials involved subjecting the catalyst to five consecutive reaction cycles under consistent conditions, including a reactant concentration of 952 μ mol of NaBH₄, a neutral pH, and a temperature of 295 K.

Figure 8 illustrates the results of these reusability trials, revealing the consistency in the volume of hydrogen produced throughout the consecutive trials. There was a slight increase observed in hydrogen generation after the second trial, followed by a stable average hydrogen yield of 17.9 mL per trial. This trend suggests that the catalyst may have undergone activation after the initial trials, leading to enhanced catalytic performance. Such activation phenomena are consistent with previous findings reported by the research team of Deraedt et al. in 2014. Their studies suggested that the bonds present on the surface of palladium nanoparticles undergo hydrolysis over time, thereby increasing their catalytic activity [42–45].

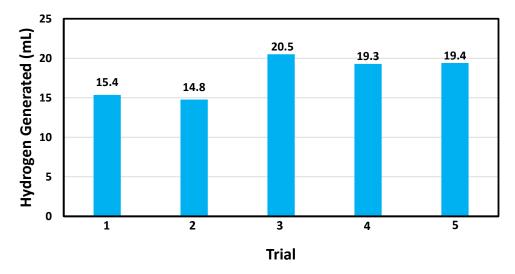
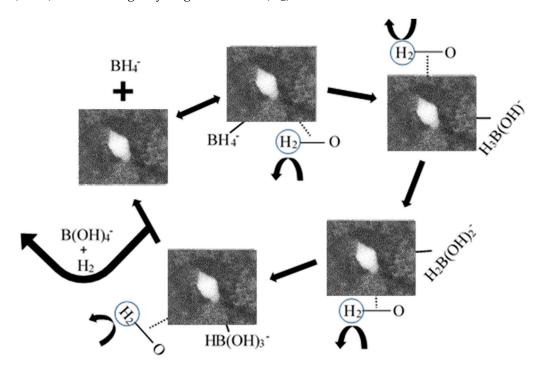


Figure 8. Testing reusability of the Pd-MCM catalyst after five consecutive hydrogen generation reactions.

The observed stability of the Pd-MCM catalyst across all five trials implies its robustness and resilience to degradation. This stability underscores the catalyst's potential for repeated use, highlighting its effectiveness as a sustainable and cost-efficient catalyst option. By demonstrating its ability to maintain catalytic activity over multiple cycles, the Pd-MCM catalyst solidifies its position as a viable and practical solution for sodium borohydride hydrolysis (Table 1), further supporting the advancement of sustainable catalysis technologies.

The proposed method of hydrogen generation via the hydrolysis of sodium borohydride (NaBH₄) catalyzed by Pd-MCM is illustrated in Scheme 1. The reaction sequence initiates when a borohydride ion (BH₄ $^-$) interacts with a Pd-NP embedded within the

MCM structure. This interaction anchors the borohydride ion to the platinum surface. Subsequently, a nearby water molecule (H_2O) is attracted to the bound borohydride ion. The oxygen atom in the water molecule coordinates with the borohydride, which facilitates the attack of the water molecule on the borohydride. This attack results in the transfer of a hydrogen atom from the borohydride to the water molecule, producing a hydroxyl group (OH^-) and releasing a hydrogen molecule (H_2) .



Scheme 1. Proposed mechanism of hydrogen generation vis Pd-MCM.

The process described above is repeated three additional times, each time producing one hydrogen molecule and resulting in the formation of a tetrahydroxoborate ion $(B(OH)_4^-)$ as the final product. After the fourth cycle, the $B(OH)_4^-$ ion dissociates from the Pd nanoparticle, freeing the catalytic site for a new borohydride ion to bind, thus allowing the cycle to repeat.

For each complete catalytic cycle involving one borohydride ion yields four molecules of hydrogen gas. The high efficiency of this process, facilitated by the MCM-supported palladium nanoparticles, highlights the potential of this method for sustainable hydrogen production [45].

4. Conclusions

The synthesis of the Pd-MCM composite was successfully accomplished and confirmed through a comprehensive characterization process. Transmission electron microscopy (TEM) provided detailed images revealing the uniform dispersion of palladium nanoparticles within the mesoporous carbon matrix. Additionally, Scanning Electron Microscopy/Energy Dispersive Spectroscopy (SEM/EDS) analysis confirmed the elemental composition of the composite, further validating its structure. X-Ray diffraction (XRD) analysis elucidated the crystalline nature of the palladium nanoparticles, affirming the successful synthesis of the Pd-MCM composite.

In catalytic testing, the Pd-MCM composite exhibited its highest reaction rate during the hydrogen evolution reaction at a temperature of 303 K, a pH of 6, and a reactant concentration of 1057 µmol of NaBH₄. By exploring a range of temperatures from 273 K to 303 K, the activation energies for the Pd-MCM catalyst were determined, yielding a relatively low value of 27.6 kilojoules per mole. This low activation energy indicates the catalyst's high efficiency in facilitating the reaction process. Furthermore, the durability of

the Pd-MCM catalyst was demonstrated through its performance in the hydrolysis reaction. Despite repeated use, the catalyst maintained its effectiveness, showcasing its robustness and stability under challenging conditions.

These findings strongly suggest that the Pd-MCM composite is not only an effective catalyst but also holds great promise for applications in the hydrogen economy. Its ability to efficiently catalyze hydrogen-related reactions, coupled with its durability and low activation energy, positions it as a valuable asset in advancing hydrogen production and utilization technologies. As such, the Pd-MCM composite represents a significant step forward in the quest for sustainable and efficient catalysts for a hydrogen-driven future.

Author Contributions: Methodology, T.M.A.-F.; Validation, T.M.A.-F.; Formal analysis, E.B., Q.Q. and T.M.A.-F.; Investigation, Q.Q.; Resources, T.M.A.-F.; Data curation, E.B.; Writing—original draft, E.B.; Writing—review & editing, Q.Q. and T.M.A.-F. All authors have read and agreed to the published version of the manuscript.

Funding: This research received no external funding.

Data Availability Statement: The raw data supporting the conclusions of this article will be made available by the authors on request.

Acknowledgments: The corresponding author acknowledges Lawrence J. Sacks' professorship in chemistry.

Conflicts of Interest: The authors declare no conflicts of interest.

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