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Effect of Hydrothermal Treatment Temperature and Time on Carbon-Supported Pd Catalysts for Rosin Disproportionation

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ABSTRACT: Hydrothermal modification of activated carbon has been recognized as an effective strategy to improve catalyst performance through pore structure limitation and metal dispersion enhancement. In this study, the effects of hydrothermal treatment temperature and time on carbon-supported palladium (Pd/C) catalysts were investigated for rosin disproportionation. Activated carbon derived from coconut shells was treated hydrothermally at temperatures of 180 - 200 °C for 3 - 5 h. The modified carbons were subsequently impregnated with palladium and evaluated in abietic acid disproportionation. The highest abietic acid conversion is 99.2% by catalyst prepared at 200 °C for 5 h. The nitrogen adsorption–desorption (BET) revealed that hydrothermal treatment significantly enhanced surface area and mesoporous structure, promoting uniform Pd dispersion. Improved catalytic activity was attributed to enhanced crystallinity, pore accessibility, and mass transfer. This work demonstrates that hydrothermal treatment is an efficient approach for developing high-performance Pd/C catalysts for biomass-derived resin acid valorization.

KEYWORDS: Abietic acid; Activated carbon; Hydrothermal treatment; Mesoporous structure; Pd/C catalyst; Rosin disproportionation.

I. INTRODUCTION

The growing global demand for sustainable and environmentally responsible chemical processes has intensified research efforts toward the valorization of renewable resources to produce high-value chemicals and materials. Gum rosin, a naturally abundant product obtained from pine resin, is primarily composed of abietic acid, a resin acid containing conjugated double bonds and carboxylic functional groups. These structural features impart high chemical reactivity, enabling abietic acid to undergo hydrogenation and disproportionation reactions to form more stable derivatives. Such products are widely applied in coatings, adhesives, inks, lubricants, and chemical intermediates, highlighting the importance of developing efficient and selective catalytic routes for rosin upgrading especially for dihydroabietic acid [1,2], and dehydroabietic acid [3,4].

The disproportionation of abietic acid generally occurs at temperatures of approximately 270 °C under inert atmospheres and involves intermolecular hydrogen transfer and structural rearrangement produces a mixture of dehydroabietic, dihydroabietic and tetrahydroabietic acid as illustrated in Figure 1 [5–7]. Owing to the simultaneous occurrence of dehydrogenation and hydrogenation pathways, catalyst performance plays a decisive role in determining conversion efficiency, product distribution, and energy consumption. Therefore, the rational design of highly active, stable, and selective catalysts is essential for improving process sustainability and economic viability.

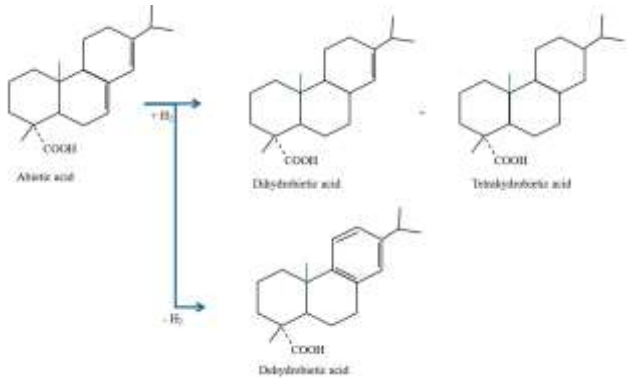

Figure 1 The disproportionation of abietic acid into dehydroabietic acid by dehydrogenation, and hydrogenation into dehydroabietic acid, and tetrahydroabietic acid.

Palladium supported on activated carbon (Pd/C), have been extensively employed in hydrogenation and disproportionation reactions owing to their high intrinsic activity, chemical stability, and resistance to deactivation [5,8–10]. However, the high cost and limited availability of

palladium considerably restrict its large-scale application, making the improvement of metal utilization efficiency a critical issue. Enhancing palladium dispersion and stabilization on suitable supports is therefore essential to maximize catalytic performance while minimizing noble metal loading. Carbon materials are widely recognized as attractive catalyst supports due to their high surface area, tunable surface chemistry, and adjustable pore architectures. Ordered mesoporous–macroporous structures facilitate mass transfer, reduce diffusion limitations, and improve accessibility of active sites, thereby enhancing catalytic performance [11–13].

Biomass-derived carbons, such as those obtained from coconut shells, offer sustainable and low-cost alternatives to conventional carbon supports. Their physicochemical properties can be effectively custom-made through hydrothermal treatment by reconstructing pore structures, removing unstable components, and reorganizing surface functional groups, resulting in increased surface area, enhanced mesoporosity, and improved surface homogeneity. Previous studies have demonstrated that hydrothermally modified carbons exhibit superior performance in diverse catalytic systems by promoting uniform metal dispersion and improving mass transport characteristics. Nevertheless, the influence of hydrothermal treatment parameters on support structure and catalytic behavior remains highly dependent on the specific reaction system and preparation conditions. Therefore, many researchers investigated to minimize the use of Pd without reducing its catalytic activity [5,8–11,13,14].

In this study, the effects of hydrothermal treatment temperature and duration on coconut shell–derived carbon supports and their corresponding Pd/C catalysts are investigated for abietic acid disproportionation. By controlling hydrothermal conditions in the range of 180–200 °C and 3–5 h, this work aims to elucidate the relationship between pore structure evolution, and catalytic performance. Comprehensive characterization using XRD and nitrogen adsorption–desorption analyses is employed to correlate physicochemical properties with catalytic activity. The results are expected to provide insights into the rational design of efficient catalysts for abietic acid disproportionation.

II. MATERIALS AND METHOD

A. Catalyst Preparation

Coconut shell-derived carbon was obtained from abundant commercial source in Indonesia. Palladium chloride ($PdCl_2$), and gum rosin (grade X), and other analytical-grade reagents were obtained and used without further purification. Activated carbon was prepared by hydrothermal treatment. Coconut shell-derived carbon was dispersed in deionized water and subjected to hydrothermal treatment in a Teflon-lined stainless-steel. The weight ratio of coconut shell-derived carbon and deionized water is equal (50% w). The treatment was conducted at temperatures of 180 - 200 °C for 3 - 5 h. After cooling to room temperature, the samples were filtered, washed thoroughly, dried, and calcined at 650°C for 1 hour. The catalysts were prepared by impregnating 0.1 wt.% Pd onto the hydrothermally treated carbon, followed by drying and calcination at 550°C for 1 hour. The catalyst was pelletized to a dimension of 3mm in diameter and 1cm in length using 0.4 g of starch and 2 mL of water as binders for 5 g of catalyst. The pellets were dried and calcined at 550°C for 1 hour.

B. Catalytic Activity Test

Hydrogenation reactions were performed in a batch reactor under Argon atmosphere. As much as 60 g of gum rosin and 6 wt.% Pd/C catalyst were loaded into the reactor and heated to 270°C for 2 hours with stirring at 100 rpm. Upon completion of the reaction, the products were separated from the catalysts, molded and cooled in air. Analyses were performed using UV–Vis spectroscopy (Analytik Jena Specord 200 Plus) over the wavelength range of 200–1100 nm, Fourier Transform Infrared Spectroscopy (FT-IR) (Jasco FT/IR-6800 with ATR) under ambient air, and acid number determination. The abietic acid content in gum rosin was determined using the formula (1) as proposed by Gu *et al.* (2020) based on UV–Vis spectrophotometric measurements [5].

$$Abietic\ Acid: W(\%) = \frac{E_{241}-E_{250}}{ckl} \times 100\% \qquad (1)$$

where E_{241}, and E_{250} represent the absorption at the troughs (241 nm, and 250 nm), c stands for the concentration of samples (g/L), l is the thickness of cuvette (cm), and k is specific absorption coefficient of pure abietic acid which is equal to 28.

C. Characterization

Characterization of catalysts included X-ray diffraction (XRD) with Cu Kα radiation (40 kV, 30mA, PANalytical), and Brunauer–Emmett–Teller (BET) surface area measurements under nitrogen atmosphere (Quantachrome Nova 1200e).

III. RESULTS AND DISCUSSION

A. Catalytic Performance

Table 1 summarizes the effect of hydrothermal treatment temperature and duration on the residual abietic acid content and conversion in rosin disproportionation using 0.1% Pd/C catalysts. The untreated gum rosin initially contained 25% abietic acid. After applying hydrothermally modified carbon-supported catalysts, the residual abietic acid content decreased to below 1% under all treatment conditions. As a result, high conversion values ranging from 96.11% to 99.09% were achieved across the investigated temperature

and time variations. These data demonstrate that hydrothermal modification of carbon supports significantly improves the catalytic performance of Pd/C in promoting abietic acid conversion.

Table 1 Catalytic activity of 0.1% Pd on activated carbon treated by hydrothermal

Hydrothermal Treatment	Abietic acid (%)	Abietic acid conversion (%)
180 C, 3h	0,41	98,38
180 C, 4h	0,31	98,75
180 C, 5h	0,30	98,79
190 C, 3h	0,25	98,99
190 C, 4h	0,28	98,87
190 C, 5h	0,25	98,96
200 C, 3h	0,33	98,67
200 C, 4h	0,97	96,11
200 C, 5h	0,23	99,09

Note: Abietic acid composition on gum rosin before reaction is 25%.

Raw gum rosin exhibits a strong absorption band at 240–250 nm, which is characteristic of abietic acid and originates from π–π* transitions of its conjugated double-bond system, indicating a high initial concentration of this compound. After reaction using untreated Pd/C, the absorbance intensity in this region decreases, reflecting partial conversion of abietic acid. In contrast, products obtained with hydrothermally treated Pd/C catalysts display a much lower absorbance intensity, particularly for longer treatment times (3–5 h at 180 °C), demonstrating more effective conversion (Supplementary Figure 1). This trend suggests that hydrothermal modification improves the surface properties and metal–support interaction, thereby enhancing the accessibility and activity of Pd active sites toward abietic acid molecules. The progressive reduction in absorbance with increasing treatment time indicates more efficient formation of disproportionation products, such as dehydroabietic and dihydroabietic acids, which exhibit weaker UV absorption in this region. Moreover, the absence of strong new absorption bands at higher wavelengths implies minimal formation of polymeric or aromatic by-products, indicating good reaction selectivity.

Hydrothermal treatment temperature has a significant influence on abietic acid conversion in the rosin disproportionation reaction. As the temperature increased from 180°C to 200°C, the conversion generally showed an increasing trend. Higher treatment temperatures promoted more effective activation of the Pd/C catalyst, resulting in improved catalytic performance during the reaction. At elevated temperatures, the catalyst exhibited greater ability to facilitate hydrogen transfer and molecular rearrangement. Consequently, higher hydrothermal treatment temperatures contributed to enhanced abietic acid conversion.

The duration of hydrothermal treatment also affects the conversion of abietic acid. An increase in treatment time from 3 to 5 h generally led to higher conversion values. Longer treatment durations improved the stability and effectiveness of the catalyst during the reaction process. This condition allowed the catalyst to maintain consistent activity throughout the disproportionation reaction. As a result, extended hydrothermal treatment time contributed to higher and more stable abietic acid conversion.

The interaction between hydrothermal treatment temperature and time plays an important role in determining abietic acid conversion. The highest conversion was achieved at 200°C for 5 h, indicating that an appropriate combination of high temperature and sufficient treatment duration is required for optimal catalytic performance. In contrast, the lower conversion observed at 200°C for 4 h suggests that suboptimal combinations may reduce catalytic effectiveness. This interaction demonstrates that temperature and time must be carefully controlled simultaneously to achieve maximum conversion. Therefore, balanced optimization of both parameters is essential for improving abietic acid conversion in rosin disproportionation.

The data in Table 1 indicate that both hydrothermal treatment temperature and duration strongly influence the catalytic conversion of abietic acid, with higher temperatures generally yielding greater conversion, likely due to enhanced activation of the carbon support and improved dispersion of palladium species, which in turn facilitate hydrogen transfer and disproportionation reactions—a phenomenon consistent with studies showing that support modification can significantly enhance catalytic performance by improving active site accessibility and metal dispersion (e.g., well-dispersed Pd on tailored carbon supports improves hydrogenation and related reactions) [15] . Increasing the treatment time from 3 h to 5 h tended to maintain or slightly improve conversion, reflecting the effect of extended exposure in stabilizing surface properties and maintaining catalyst efficacy throughout reaction conditions, aligning with literature that longer hydrothermal processing can refine pore architecture and stabilization of active phases [16–20]. The interaction between temperature and time is highlighted by the exceptionally high conversion (99.09%) at 200 °C for 5 h compared to the somewhat lower conversion at 200 °C for 4 h, indicating that an optimal combination of sufficiently high thermal energy and duration is necessary to achieve maximum catalytic effectiveness, which parallels reports in heterogeneous catalysis where both preparation temperature and temporal conditions must be optimized to maximize performance due to synergistic effects on surface chemistry and metal–support interactions.

B. Effect of Hydrothermal Treatment Temperature and

Time

The influence of hydrothermal treatment temperature and duration on abietic acid conversion was further investigated through structural and surface characterization using X-ray diffraction (XRD) and Brunauer–Emmett–Teller (BET) surface area analysis. XRD was employed to examine changes in crystallinity and palladium dispersion that may affect catalytic activity under different treatment conditions. Meanwhile, BET analysis provided quantitative information on specific surface area and textural properties that are closely related to catalyst performance. By correlating these physicochemical characteristics with the observed conversion values, the effects of hydrothermal temperature and time on catalytic efficiency could be systematically evaluated. This integrated approach enables a comprehensive understanding of how preparation conditions influence the activity of Pd/C catalysts in abietic acid disproportionation.

The XRD patterns of carbon samples under hydrothermal treatment display a dominant diffraction peak at approximately $2\theta = 24°$, which corresponds to the (002) plane of graphitic carbon, indicating the predominantly amorphous and semi-crystalline nature of the materials (Supplementary Figure 2). In addition, a weak diffraction peak appears near $2\theta = 43°$, attributed to the (100)/(101) planes associated with the initial development of hexagonal carbon layers. While the main peak at around 24° shows no significant change before and after hydrothermal treatment, the intensity of the peak near 43° increases noticeably in all treated samples. With increasing treatment time and carbon–water concentration, this peak becomes sharper and more pronounced, reflecting improved structural organization. Moreover, the diffraction signal at 43–44° is consistent with the hexagonal graphite lattice (JCPDS 75-1621), confirming the presence of ordered graphitic domains. These results demonstrate that hydrothermal treatment promotes structural ordering of the carbon material, particularly through the formation and growth of hexagonal carbon layers.

Table 2 Surface area S_{BET} and pore structure activated carbon by hydrothermal treatment.

Hydrothermal Treatment	S_{BET} m^2/g	V_{meso}/V_T (%)	Dp (nm)	Main Pore Structure
Raw Carbon	11	21	4.91	Macropore
180 C, 3h	483	51	3.43	Mesopore
180 C, 4h	477	67	3.40	Mesopore
180 C, 5h	483	61	3.80	Mesopore
200 C, 5h	503	53	4.39	Mesopore
180 C, 4 h, and 0,1% Pd	479	51	3.39	Mesopore

Hydrothermal treatment temperature and time significantly influences the pore structure and textural properties of activated carbon as shows in Table 2. Hydrothermally treated samples showed a substantial increase in surface area (477 – 503 m^2/g) and a transition to mesoporous structures, compared with raw carbon, which exhibited a low surface area of 11 m^2/g and a macroporous structure. In addition, the specific surface area of the hydrothermally treated carbon support remains constant after palladium incorporation. However, the main pore structure remained mesoporous, and no significant change in pore type was observed after Pd incorporation. These results indicate that palladium loading did not alter the fundamental pore architecture of the carbon support, while preserving its suitability for catalytic applications.

Hydrothermal treatment temperature significantly influences the pore structure and textural properties of activated carbon as shows in Table 3.2. Compared with raw carbon, which exhibited a low surface area of 11 m^2/g and a macroporous structure, hydrothermally treated samples showed a substantial increase in surface area and a transition to mesoporous structures. Increasing the treatment temperature from 180°C to 200°C led to a slight improvement in specific surface area from approximately 480 m^2/g to 503 m^2/g. This increase was accompanied by a moderate enlargement of average pore diameter from around 3.4–3.8 nm to 4.39 nm. These results indicate that higher hydrothermal temperatures promote more intensive modification, leading to improved pore accessibility and surface development.

The duration of hydrothermal treatment plays an important role in determining the extent of pore development and surface area enhancement. At 180°C, increasing the treatment time from 3 to 5 h resulted in relatively stable surface area values, ranging from 477 to 483 m^2/g, indicating that major surface development occurred within the first few hours. However, variations in pore volume ratio and average pore diameter were observed, suggesting gradual pore restructuring with prolonged treatment. The pore diameter increased from 3.43 nm at 3 h to 3.80 nm at 5 h, reflecting progressive pore widening. These changes demonstrate that longer treatment times contribute to the stabilization and refinement of mesoporous structures.

The combined effects of hydrothermal temperature and time strongly determine the final textural properties of activated carbon. The highest surface area and relatively large pore diameter were achieved at 200°C for 5 h, indicating a synergistic interaction between elevated temperature and extended treatment duration. This condition promoted optimal pore reconstruction, resulting in well-developed mesoporous structures with enhanced accessibility. In contrast, treatment at 180°C for 4 h produced slightly lower surface area and smaller pore diameter, suggesting incomplete modification under moderate conditions. These observations highlight that temperature and time must be

optimized simultaneously to achieve desirable structural characteristics.

This structural evolution has been consistently reported in recent studies, showing that hydrothermally treated carbons typically display mesoporous type pores material [21–24]. Their non-toxicity and good biocompatibility further enhance their applicability, emphasizing the importance of hydrothermal modification in the development of functional mesoporous carbon materials [25–27].

The observed variations in pore structure and textural properties show a strong correlation with abietic acid conversion in rosin disproportionation. Catalysts supported on carbons with higher surface area and well-developed mesoporous structures generally exhibited higher conversion values, as demonstrated by the superior performance at 200°C for 5 h. Larger pore diameters and higher pore volumes facilitate reactant diffusion and improve contact between abietic acid molecules and active palladium sites. Conversely, samples with relatively lower surface area and smaller pore diameters showed slightly reduced conversion. These results indicate that optimized textural properties obtained through appropriate hydrothermal treatment play a key role in achieving high abietic acid conversion.

V. CONCLUSIONS

This study demonstrates that low-loading palladium supported on hydrothermally treated activated carbon is highly effective for catalyzing abietic acid disproportionation, achieving excellent conversion performance. Controlled hydrothermal modification of coconut shell–derived carbon at 180–200 °C for 3–5 h significantly enhances surface area and promotes the formation of well-developed mesoporous structures, which facilitate uniform Pd dispersion and efficient mass transfer. The optimal catalyst prepared at 200 °C for 5 h exhibited the highest abietic acid conversion of 99.2%, confirming the critical role of treatment temperature and duration in determining catalytic efficiency. XRD and BET analyses revealed that hydrothermal treatment improves crystallinity and textural properties of the carbon support, while Pd incorporation causes only minimal changes in surface area without altering the mesoporous structure. These physicochemical improvements contribute to enhanced accessibility of active sites and stable catalytic performance.

Furthermore, UV–Vis analysis confirmed the substantial reduction of residual abietic acid, demonstrating the high effectiveness of the Pd/C catalyst containing only 0.1 wt.% palladium. The combination of low noble metal loading and optimized support properties enables efficient disproportionation with minimal by-product formation and high operational stability. This approach provides a cost-effective and sustainable strategy for biomass-derived resin acid valorization. This work also supports Sustainability Development Goals (SDG) 7 (promoting energy-efficient biomass valorization), SDG 9 (Industry, Innovation, and Infrastructure), SDG 12 (Responsible Consumption and Production), and SDG 13 (Climate Action), and SDG 14 by promoting the utilization of renewable resources, reducing dependence on precious metals, and developing energy-efficient catalytic processes for green chemical production, and minimizing environmental impacts on aquatic ecosystems.

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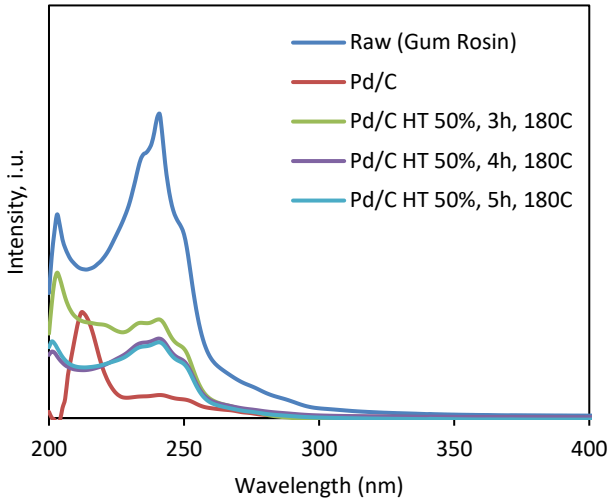
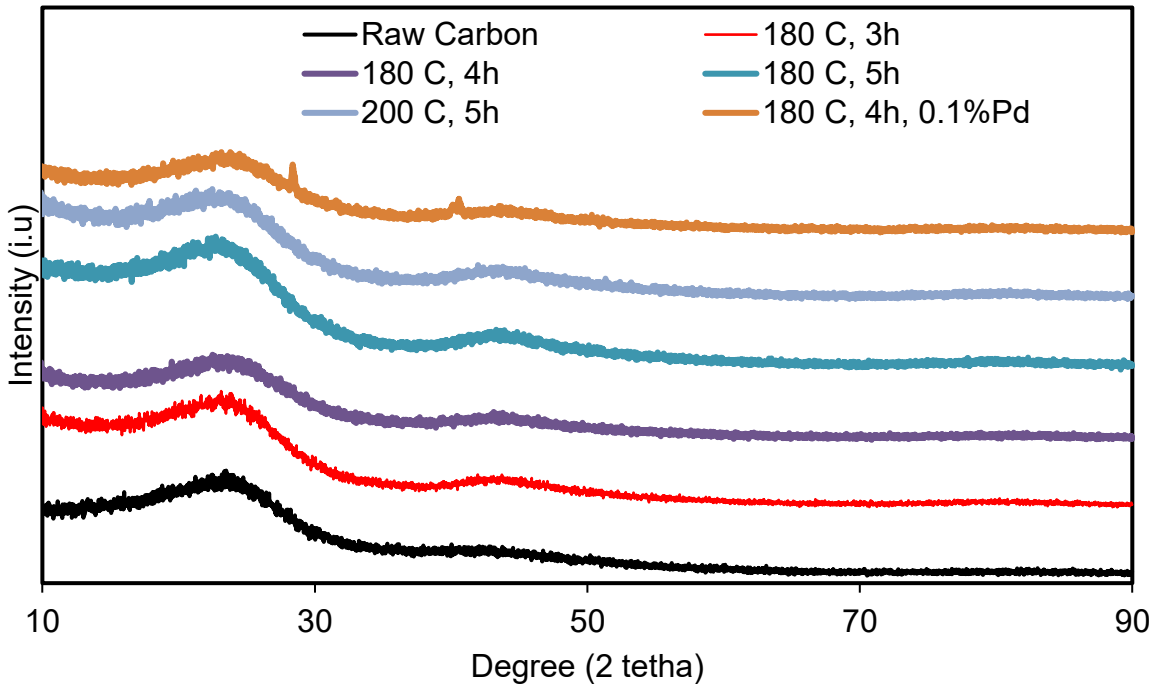
Supplementary Information



Supplementary Figure 1. UV-Vis spectrophotometric results of gum rosin as the raw material, and products obtained after 2 hours of reaction at 270 °C using a 0.1 wt.% Pd on various carbon-based catalyst with a 6% catalyst loading. The activated carbon was prepared with 50% carbon composition and subjected to hydrothermal treatment at 180 °C for durations of 3 to 5 hours, while untreated carbon (Pd/C) was used as a reference.



Supplementary Figure 2. Spectrum of XRD of activated carbon treated by hydrothermal at carbon–water composition 50% and treatment time 3–5 hours at 180 - 200 °C.