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Research Paper

Synthesis of Adipic Acid via Eco-Friendly Oxidation of Cyclohexanone-Cyclohexanol over B_2O_3-SiO_2 Catalyst

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Abstract

The synthesis of adipic acid through the oxidation reaction of a cyclohexanone-cyclohexanol mixture was investigated using 30% hydrogen peroxide (H_2O_2) as the oxidizing agent and a B_2O_3–SiO_2 catalyst synthesized via heat treatment at various temperatures. This study aimed to evaluate the catalytic performance of B_2O_3–SiO_2 in facilitating the oxidation reaction and to determine the optimum reaction conditions for achieving the maximum yield of adipic acid. The effects of reaction time and temperature on the oxidation process were systematically studied. The reactions were conducted for 5, 6, 7, and 8 hours at temperatures of 60°C, 70°C, 80°C, and 90°C. The reaction products were analyzed using Gas Chromatography (GC) and Fourier-transform infrared spectroscopy (FT-IR). The results showed that the B_2O_3–SiO_2 catalyst exhibited the highest catalytic activity at a reaction temperature of 90°C, producing adipic acid with a maximum yield of 2.36% after 7 hours of reaction. Based on FT-IR characterization, it was observed that the B_2O_3–SiO_2 catalyst became unstable after the reaction, indicated by the reduction of Brønsted acid sites. This decrease in acidity led to less effective catalytic performance, resulting in a suboptimal oxidation process and a relatively low yield of adipic acid.

Keywords

Adipic Acid, Cyclohexanol, Cyclohexanone, B_2O_3–SiO_2, H_2O_2

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1. INTRODUCTION

Adipic acid (hexanedioic acid) is a vital dicarboxylic acid used extensively in the manufacture of nylon-6,6, plasticizers, lubricants, insecticides, and polymer precursors (e.g. polyesters) (Liang et al., 2025; Ribeiro et al., 2020). Traditionally, adipic acid is produced via the oxidation of cyclohexane to a cyclohexanone/cyclohexanol mixture, followed by oxidation using nitric acid, a process that emits nitrous oxide (N_2O) as a major greenhouse gas by product Lisicki et al. (2023a); Yan et al. (2020). To mitigate environmental impact, greener synthetic routes employing hydrogen peroxide (H_2O_2) as an oxidant have been explored, because H_2O_2 decomposes to water and avoids nitrogen oxide byproducts (Dutta, 2025; Qin et al., 2025). In fact, 30 wt% H_2O_2 has been demonstrated as a practical "clean" oxidant for converting cyclohexane, cyclohexanol, cyclohexanone, or their derivatives to adipic acid in prior studies (Lesbani and Mohadi, 2015; Mouanni et al., 2019; Sativa and Lesbani, 2017). Moreover, using a mixture of cyclohexanone and cyclohexanol often gives a higher yield of adipic acid than stoichiometric use of each substrate individually, due to synergistic oxidation interactions and radical chain propagation between ketone and alcohol moieties (Lisicki et al., 2023a; Moudjahed et al., 2022).

Catalysts are essential in such oxidative syntheses, as they activate hydrogen peroxide (or peroxo species) and guide selective oxidation pathways toward adipic acid instead of over-oxidation or undesired byproducts. For example, Dawson-type polyoxometalates have shown high conversion of cyclohexanone/cycloh exanol mixtures with H_2O_2 under mild conditions (Moudjahed et al., 2022). Other catalysts reported include WO_3/SiO_2 for green oxidation of cyclohexanone to adipic acid (Yan et al., 2020), and various heteropoly acids or supported metal catalysts.

Various solid acid catalysts have been employed in the oxidation of cyclohexanone and cyclohexanol to adipic acid to activate hydrogen peroxide and enhance selectivity toward the desired product. Among them, polyoxometalates (POMs) with the Keggin structure are frequently utilized in acid catalysis because of their simple synthesis procedure, strong redox ability, and excellent chemical stability (Hanifah and Amri, 2023; Qin et al., 2023; Zhao et al., 2023). However, their high cost and potential leaching in liquid-phase reactions limit their large-scale applications. Therefore, the development of alternative, stable, and low-cost catalysts remains essential. Among boron-based catalysts, boric acid (H_3BO_3) has often been applied because boron centers can confer both Lewis and Bronsted acidity, which facilitates peroxidic activation and proton transfers during oxidation cycles, although specific studies combining boron and silica in adipic acid synthesis remain limited (Deary, 2023; Walia et al., 2014; Yang et al., 2014).

In this study, a catalyst composed of boric acid (B_2O_3) and silica (SiO_2) was employed for the oxidation of a cyclohexanone–cyclohexanol mixture to adipic acid using hydrogen peroxide as the oxidant. The catalytic performance was evaluated through product analysis using Fourier Transform Infrared Spectroscopy (FTIR) and Gas Chromatography (GC). To obtain the highest possible adipic acid yield, the effects of reaction time and temperature on the oxidation process were systematically investigated.

2. EXPERIMENTAL

2.1 Chemicals and Instruments

All chemicals used in this study were of analytical grade (p.a). Cyclohexanone, cyclohexanol, hydrogen peroxide (H_2O_2), distilled water, boric acid (H_3BO_3), and silica (SiO_2) were used without further purification. The instruments employed in this research included a 250 mL reflux flask equipped with a magnetic stirrer, reflux apparatus, desiccator, filter paper, beakers, thermometer, and capillary tubes. The catalysts and products were characterized using a Fourier Transform Infrared (FT-IR) spectrophotometer (Shimadzu Prestige-21) and analyzed using a gas chromatograph (GC) Shimadzu 2010 Plus.

2.2 Preparation and Characterization of B_2O_3–SiO_2 Catalyst

The B_2O_3–SiO_2 catalyst was prepared following the method reported by Ni et al. (2024) with slight modifications. A total of 0.162 g of boric acid (H_3BO_3) was dissolved in 30 mL of distilled water in a 100 mL beaker. The mixture was stirred magnetically for 10 minutes at 80°C, followed by the addition of 1.458 g of SiO_2. The resulting suspension was refluxed for 5 hours at 90°C. After refluxing, the mixture was evaporated to obtain a solid precursor of B_2O_3–SiO_2. The obtained solid was then dried at 95°C for 48 hours to remove residual moisture. Subsequently, the dried solid was calcined at different temperatures of 300°C, 400°C, 500°C, 600°C, and 700°C. The resulting B_2O_3–SiO_2 catalyst was characterized by FT-IR spectroscopy to identify its structural and functional group features.

2.3 Conversion of Cyclohexanone–Cyclohexanol Mixture to Adipic Acid

2.3.1 Synthesis of Adipic Acid Using B_2O_3–SiO_2 Catalyst

The oxidation of the cyclohexanone–cyclohexanol mixture into adipic acid was carried out according to a modified procedure by Lisicki et al. (2023b). A mixture consisting of 1 mL cyclohexanone and cyclohexanol in a 1:1 molar ratio was placed into a flask along with 0.02 g of B_2O_3–SiO_2 catalyst (calcined at 600°C). The mixture was stirred magnetically for 10 minutes at room temperature, followed by the addition of 8 mL of 30% H_2O_2 solution. The reaction mixture was then refluxed for 6 hours at 90°C. After the reaction, the mixture was cooled and kept at 5°C for 24 hours before being analyzed by gas chromatography.

2.3.2 Effect of Reaction Time

The schematic illustration of the oxidation of the cyclohexanone–cyclohexanol mixture to adipic acid using 30% H_2O_2 as the oxidant and B_2O_3–SiO_2 as the catalyst is shown in Figure 1. The influence of reaction time on the conversion of cyclohexanone-cyclohexanol mixture into adipic acid was investigated using the same procedure as described in Section 2.3.1. The reactions were carried out for different durations of 5, 6, 7, and 8 hours. After each reaction, the solution was filtered, and the obtained product was stored in a desiccator prior to analysis using gas chromatography.

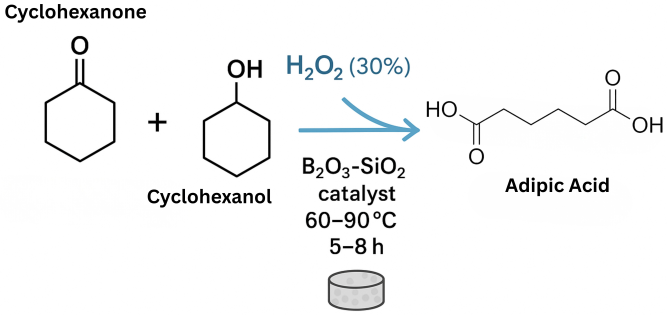

Figure 1. The Schematic Illustration of the Oxidation of the Cyclohexanone-Cyclohexanol Mixture to Adipic Acid

2.3.3 Effect of Reaction Temperature

The effect of temperature on the conversion process was studied using the optimized reaction time. The oxidation was conducted at various temperatures: 60°C, 70°C , 80°C , and 90°C . After each experiment, the solution was filtered and stored in a desiccator for subsequent gas chromatographic analysis.

2.4 Characterization of the Catalyst After Reaction

After the oxidation reaction, the B_2O_3–SiO_2 catalyst was filtered, dried at room temperature, and then treated with ammonia vapor for 24 hours. The dried catalyst was subsequently characterized by FT-IR spectroscopy to determine any changes in its acidic functional groups after the conversion reaction.

2.5 Data Analysis

The characterization of the B_2O_3–SiO_2 catalyst and the reaction products was carried out using FT-IR spectroscopy. Adipic acid formation was confirmed by gas chromatographic analysis of the oxidation products from the cyclohexanone–cyclohexanol mixture. The influence of reaction parameters such as temperature and time on conversion efficiency was evaluated. The acidic nature of the B_2O_3–SiO_2 catalyst before and after reaction was also analyzed using FT-IR.

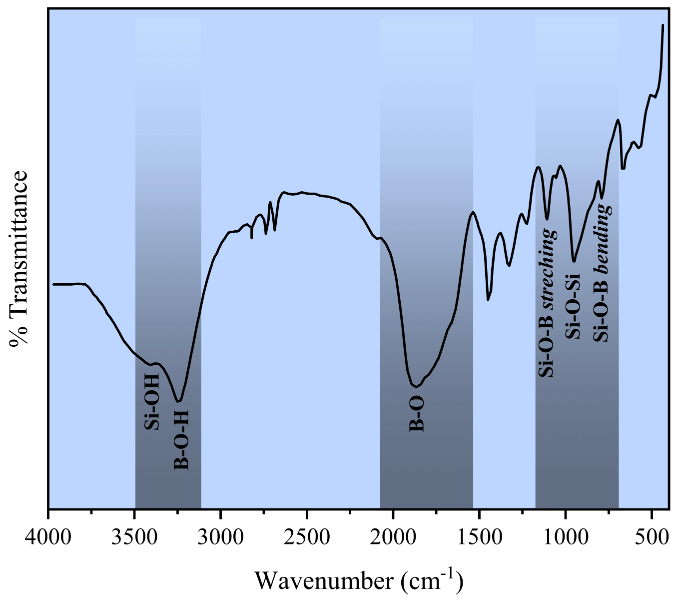

Figure 2. FT-IR Spectrum of B_2O_3–SiO_2 Compoun

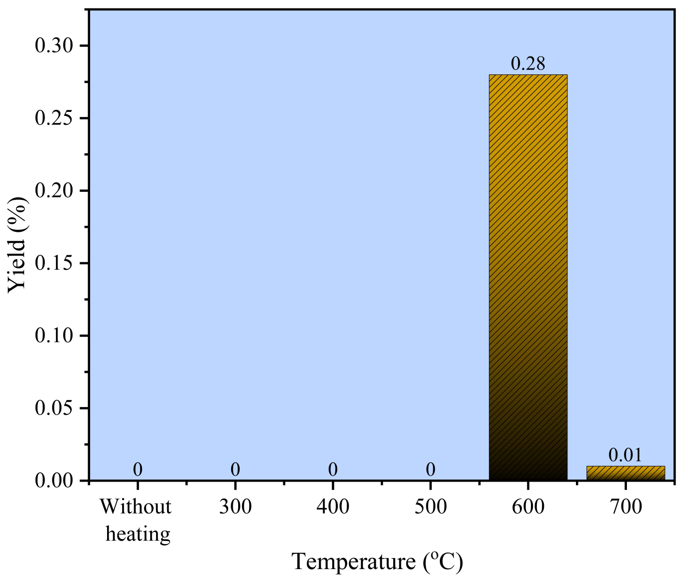

Figure 3. The Conversion Results in the Formation of Adipic Acid Using B_2O_3–SiO_2 Catalyst Synthesized at Different Calcination Temperatures

3. RESULTS AND DISCUSSION

3.1 Identification of B_2O_3–SiO_2 Compound

The B_2O_3–SiO_2 compound was prepared through the impregnation process of boric acid with silicon dioxide. The impregnation of B_2O_3–SiO_2 represents an inorganic synthesis process aimed at enhancing the acidity properties of the resulting compound, combined with silica. The characterization of B_2O_3–SiO_2 was performed using a Fourier Transform Infrared (FT-IR) spectrophotometer with the KBr pellet method to identify the functional groups present in the material.

The FT-IR spectrum of the B_2O_3–SiO_2 compound is shown in Figure 2. In Figure 2, several absorption bands corresponding to the functional groups of B_2O_3–SiO_2 were observed in the range of 4000–300 cm^{-1}. The absorption band around 3371 cm^{-1} corresponds to the stretching vibration of Si−OH, while the broad peak at 3217 cm^{-1} is attributed to the B–O–H stretching vibration (Invernizzi et al., 2018; Kostova et al., 2024). The symmetric stretching vibration of Si–O–Si was detected at 779 cm^{-1}. The bands appearing near 925 cm^{-1} and 648 cm^{-1} are associated with the stretching and bending vibrations of Si–O–B bonds, respectively. Furthermore, a distinct band was observed at 1442 cm^{-1} corresponding to the B–O vibration (Jamalaiah and Rasool, 2015).

3.2 Oxidation of Cyclohexanone–Cyclohexanol Mixture Using B_2O_3–SiO_2 Catalyst

The oxidation of cyclohexanone and cyclohexanol mixtures in a 1:1 ratio was carried out using 30% H_2O_2 as the oxidizing agent at 90°C for 6 hours. Hydrogen peroxide (H_2O_2) is an environmentally friendly oxidant, producing only water as a by-product. The B_2O_3–SiO_2 catalyst synthesized at different calcination temperatures (300°C, 400°C, 500°C, 600°C, and 700°C) was tested to evaluate its effect on adipic acid formation. The oxidation products were analyzed using gas chromatography (GC), and the conversion results are summarized in Figure 3.

The formation of adipic acid was observed when using the B_2O_3–SiO_2 catalyst calcined at 600°C and 700°C, with the highest yield of 0.28% obtained at 600°C. This result suggests that the catalytic activity of B_2O_3–SiO_2 is strongly influenced by the calcination temperature. At 600°C B_2O_3–SiO_2, the surface acidity and porosity of the catalyst are optimal, promoting the conversion of cyclohexanol–cyclohexanone to adipic acid. However, at higher temperatures (700°C), the decreased yield is likely due to the collapse of pores and partial destruction of the active sites caused by sintering, leading to catalyst deactivation (Grams and Ruppert, 2021; Lyu et al., 2023). These findings indicate that the B_2O_3–SiO_2 catalyst synthesized and calcined at $600 V \circ$C provides the most favorable conditions for the oxidation process, balancing structural stability and surface reactivity.

3.3 Effect of Reaction Time on the Formation of the Adipic Acid

The effect of reaction time on the oxidation of a mixture of cyclohexanone and cyclohexanol using H_2O_2 was studied to determine the optimal duration that produced the highest yield of adipic acid. The oxidation reactions were carried out for 5, 6, 7, and 8 hours under the same procedure as described in Section 3.2. The catalyst used was B_2O_3–SiO_2 synthesized and calcined at 600°C. The conversion of the cyclohexanone and cyclohexanol mixture into adipic acid at 600°C was obtained with varying reaction times, as summarized in Figure 4.

The conversion to adipic acid was relatively low at a reaction time of 6 hours, yielding only 0.28%. When the reaction time

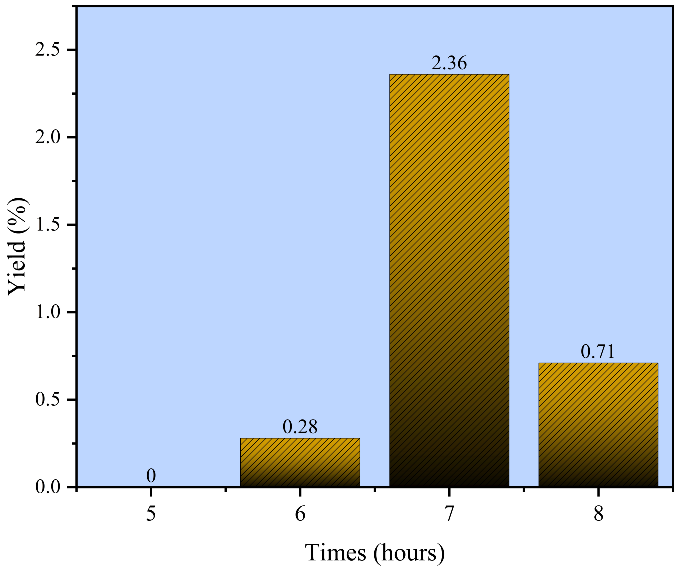

Figure 4. The Effect of Reaction Time on the Percentage Yield of Adipic Acid

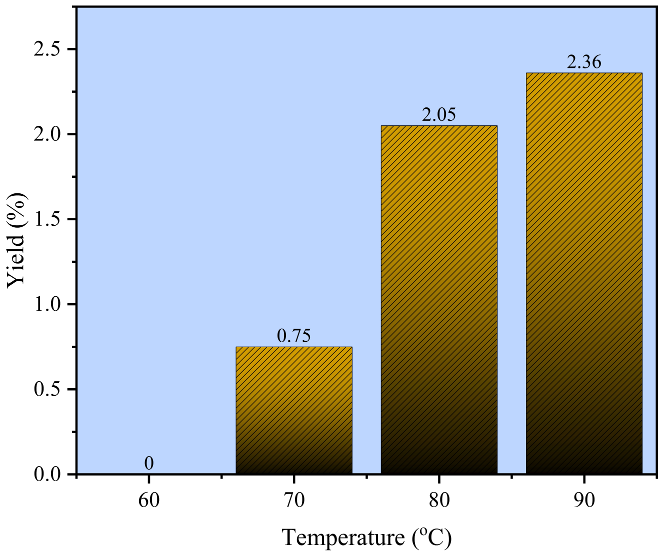

Figure 5. The Effect of Reaction Temperature on the Percentage Yield of Adipic Acid

was extended to 7 hours, the yield of adipic acid increased significantly to 2.36%. However, at a longer reaction time of 8 hours, the amount of adipic acid produced decreased to 0.71%. This decrease may be attributed to the degradation of adipic acid or the occurrence of side reactions that reduced product selectivity (Lisicki et al., 2023b; Liu et al., 2023).

The longer reaction time beyond the optimum led to product degradation and a lower yield. Moreover, excessive heating for extended durations could cause partial decomposition of adipic acid or overoxidation, producing by-products rather than crystalline adipic acid. Based on these results, the optimal reaction time for the oxidation of a cyclohexanone–cyclohexanol mixture using the B_2O_3–SiO_2 catalyst calcined at 600°C was determined to be 7 hours, yielding 2.36% adipic acid.

3.4 Effect of Reaction Temperature on the Formation of Adipic Acid

The effect of reaction temperature on the oxidation of the cyclohexanone–cyclohexanol mixture using the B_2O_3–SiO_2 catalyst was investigated to determine the optimum temperature that yields the highest adipic acid conversion. The oxidation reaction was conducted at various temperatures of 60°C, 70°C, 80°C, and 90°C. The results are summarized in Figure 5, showing that the adipic acid yield increased with rising temperature. At a reaction temperature of 60°C, no adipic acid was detected. When the temperature was increased to 70°C, the conversion of cyclohexanone–cyclohexanol to adipic acid reached 0.75%. Further increasing the reaction temperature to 80°C resulted in a yield of 2.05%, while the highest conversion of 2.36% was achieved at 90°C.

The data indicate that the reaction temperature plays a significant role in promoting the oxidation of cyclohexanone and cyclohexanol to adipic acid. Higher temperatures increase the kinetic energy of the molecules, enhancing the reaction rate and the formation of the desired product. However, temperatures above 90°C were not tested, as excessively high temperatures may lead to undesirable side reactions or catalyst deactivation (Ahmad et al., 2022; Barrientos et al., 2017; Zhao et al., 2018).

The optimal temperature for adipic acid formation under these conditions was therefore determined to be 90°C, which produced the maximum yield of 2.36%. These findings are consistent with previous research by Mouanni et al. (2019), who reported that higher temperatures facilitate the conversion of cyclohexanone–cyclohexanol mixtures into adipic acid. The increase in temperature improves the reactivity of the catalyst surface and the oxidation efficiency of the B_2O_3–SiO_2 catalyst.

3.5 Identification of Catalyst Acid Groups After Conversion Reaction

The acidity of the cyclohexanol and cyclohexanone mixture after conversion was identified qualitatively using FT-IR analysis. The FT-IR spectra of the catalyst before and after reaction are presented in Figure 6. The spectrum (a) represents the B_2O_3–SiO_2 catalyst before the reaction, while spectrum (b) corresponds to the catalyst after the oxidation of the cyclohexanone–cyclohexanol mixture. Based on the FT-IR spectrum, it can be observed that the absorption band corresponding to the functional group at a wavelength of 1458 cm^{-1} is missing, indicating that the Brønsted acid sites, typically found around 1400 cm^{-1}, have diminished. This reduction in Brønsted acidity suggests that the B_2O_3–SiO_2 catalyst becomes less stable, leading to decreased catalytic activity and, consequently, a lower yield of adipic acid (Aihara et al., 2020; Hieu et al., 2022).

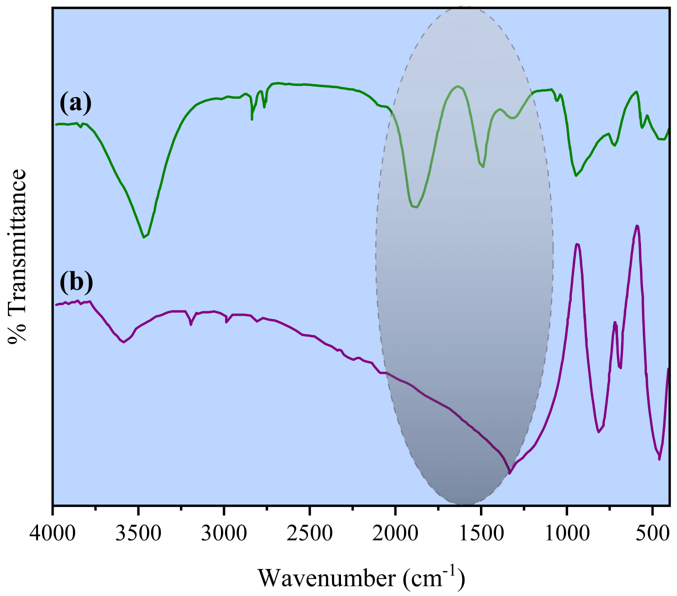

Figure 6. FTIR Spectra of B_2O_3–SiO_2 Catalyst Before (a) and After (b) the Reaction

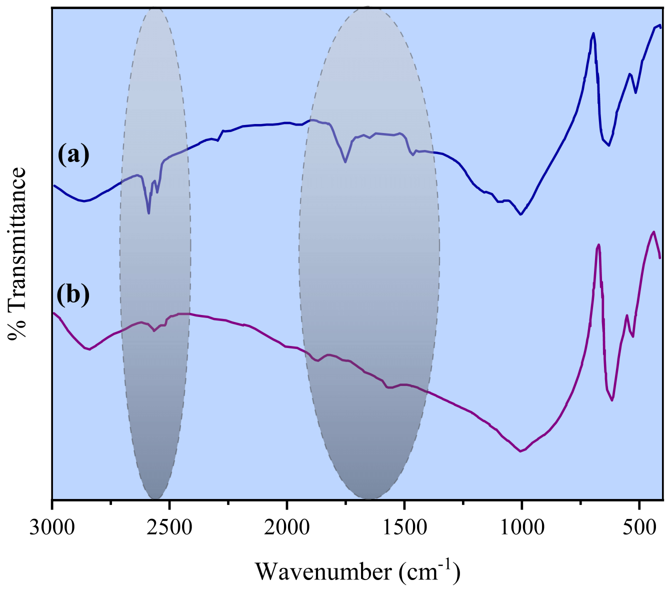

Figure 7. FTIR Spectra of B_2O_3–SiO_2 Catalyst After the Conversion Reaction Saturated with Ammonia (a) and After the Oxidation Reaction (b)

3.6 Identification of Acid Sites in B_2O_3–SiO_2 Catalyst After Reaction

The acidity characteristics of the B_2O_3–SiO_2 catalyst after the conversion reaction were also examined using FT-IR spectroscopy with ammonia as a probe molecule. The resulting spectra are displayed in Figure 7, where spectrum (a) represents the B_2O_3–SiO_2 catalyst after the reaction the conversion reaction saturated with ammonia, and spectrum (b) corresponds to the catalyst after the oxidation reaction.

The FT-IR spectrum of the pristine B_2O_3–SiO_2 catalyst (a) shows absorption bands around 1040 cm^{-1}, which are attributed to Si–O–Si stretching vibrations, and a broad band near 1400–1600 cm^{-1}, indicating the presence of surface hydroxyl groups. After ammonia adsorption (spectrum), a new absorption band appeared at approximately 1600 cm^{-1}, which corresponds to the N–H bending vibration of coordinated ammonia on Lewis acid sites. This observation confirms that the B_2O_3–SiO_2 catalyst possesses Lewis acid sites.

4. CONCLUSIONS

The oxidation of the cyclohexanone–cyclohexanol mixture using 30% hydrogen peroxide (H_2O_2) and the B_2O_3–SiO_2 catalyst successfully produced adipic acid, with the yield of 2.36% obtained at 90°C and a reaction time of 7 hours. The FT-IR analysis revealed that the B_2O_3–SiO_2 catalyst became less stable after the reaction, as indicated by the disappearance of Bronsted acid sites. This reduction in acidity contributed to the low catalytic activity and limited adipic acid formation.

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