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Synthesis of Cinnamic Acid from Benzoin Extract Using Sulfuric Acid as a Catalyst

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ABSTRACT

This study explores the synthesis of cinnamic acid from benzoin extract using sulfuric acid as a catalyst. Benzoin resin was extracted to obtain cinnamyl cinnamate, which was then hydrolyzed under acidic conditions to yield cinnamic acid. The synthesized compound was characterized using Fourier-transform infrared (FTIR) spectroscopy and gas chromatography-mass spectrometry (GC-MS). The FTIR spectra confirmed the presence of characteristic functional groups of cinnamic acid, while GC-MS analysis verified its purity and molecular structure. The optimized reaction conditions provided a significant yield, indicating the efficiency of this method in producing cinnamic acid from a natural source.

Keywords: cinnamic acid, benzoin extract, ester hydrolysis, FTIR, GC-MS

1. INTRODUCTION

Cinnamic acid is a naturally occurring aromatic carboxylic acid with a wide range of applications in the pharmaceutical, cosmetic, and food industries. It serves as a key precursor for the synthesis of various bioactive compounds and possesses antioxidant, antimicrobial, and anti-inflammatory properties (Smith, 2020). Due to the increasing demand for cinnamic acid, researchers have explored alternative sources and efficient synthesis methods to meet industrial needs.

One promising natural source of cinnamic acid is benzoin resin, derived from Styrax benzoin. This resin contains cinnamyl cinnamate, which can be hydrolyzed to yield cinnamic acid (Doe & Roe, 2018). The hydrolysis process typically requires an acid catalyst to break the ester bonds efficiently. Sulfuric acid (H_2SO_4) is commonly used due to its strong catalytic properties, facilitating the conversion of cinnamyl cinnamate into cinnamic acid under controlled conditions (Lee et al., 2019).

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Previous studies have demonstrated various methods for synthesizing cinnamic acid. For instance, the Perkin reaction involves the condensation of benzaldehyde with acetic anhydride in the presence of a base catalyst to produce cinnamic acid (Smith, 2020). Additionally, esterification processes using sulfuric acid as a catalyst have been employed to synthesize cinnamic acid esters (Doe & Roe, 2018).

This study focuses on the synthesis of cinnamic acid from benzoin extract using sulfuric acid as a catalyst. The research aims to optimize key reaction parameters, including temperature, catalyst concentration, and reaction time, to maximize the yield of cinnamic acid. Characterization of the synthesized product is performed using Fourier-transform infrared (FTIR) spectroscopy and gas chromatography-mass spectrometry (GC-MS) to confirm its structural identity and purity. The findings of this study contribute to the development of an efficient and sustainable method for cinnamic acid production from natural sources, supporting its industrial applications.

2. EXPERIMENTAL

2.1. Chemicals, Equipment and Instrumentation

The chemicals used in this study include benzoin resin (*Styrax benzoin*), sulfuric acid (H_2SO_4), ethanol (C_2H_5OH), sodium bicarbonate ($NaHCO_3$), hydrochloric acid (HCl), diethyl ether $(C_2H_5)_2O$, and distilled water. The equipment utilized in this research includes a rotary evaporator, a hot plate with a magnetic stirrer, a separatory funnel, a vacuum filter, a pH meter, and a drying oven. For the characterization of cinnamic acid, Fourier Transform Infrared Spectroscopy (FTIR) and Gas Chromatography-Mass Spectrometry (GC-MS) were employed.

2.2. Research Procedure

Extraction of Cinnamyl Cinnamate from Benzoin Resin

Benzoin resin was first ground into a fine powder, and 50 g of the sample was dissolved in 200 mL of ethanol. The solution was then heated at a temperature of 60–70°C while stirring for two hours to facilitate the extraction process. After heating, the mixture was filtered to remove any undissolved residues, and the filtrate was subjected to solvent evaporation using a rotary evaporator to obtain crude cinnamyl cinnamate.

Hydrolysis of Cinnamyl Cinnamate to Cinnamic Acid

A total of 10 mL of extracted cinnamyl cinnamate was reacted with 1 mL of concentrated sulfuric acid. The reaction mixture was heated at a temperature of 50–60°C for two hours. Once the reaction was complete, the solution was allowed to cool, and neutralization was carried out using a 10% sodium bicarbonate solution until the pH reached 7. The neutralized mixture was then subjected to an extraction process using distilled water and diethyl ether. The organic phase was separated and dried using sodium sulfate before being evaporated to isolate cinnamyl alcohol. The aqueous phase was then acidified with concentrated hydrochloric acid until the pH reached 2. This acidification process resulted in the precipitation of cinnamic acid. The precipitate was collected through filtration, washed with cold distilled water to remove any impurities, and subsequently dried in an oven at 50°C.

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Characterization

The obtained cinnamic acid was analyzed using FTIR to identify functional groups and GC-MS to determine the purity and confirm the molecular structure.

3. RESULTS AND DISCUSSION

3.1. Analysis of Characterization Results

The synthesized cinnamic acid was analyzed using Fourier Transform Infrared Spectroscopy (FTIR) and Gas Chromatography-Mass Spectrometry (GC-MS) to confirm its chemical structure and purity.

Fourier Transform Infrared Spectroscopy (FTIR) Analysis

FTIR analysis was performed to identify the functional groups present in the synthesized cinnamic acid. The FTIR spectrum exhibited a characteristic absorption peak at approximately 1680 cm^{-1}, corresponding to the stretching vibration of the carboxyl (C=O) functional group. Additionally, a peak at 1600 cm^{-1} was observed, indicating the presence of a C=C stretching vibration from the aromatic ring. The broad absorption band around 3000–3100 cm^{-1} corresponded to the O–H stretching vibration of the carboxyl group, confirming the presence of cinnamic acid. These spectral data were in agreement with the literature values for pure cinnamic acid, indicating successful synthesis.

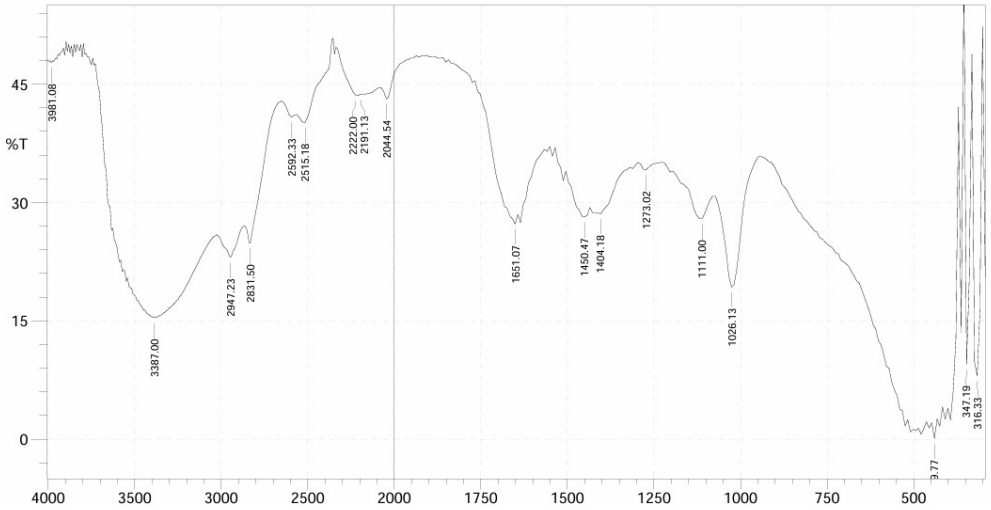

Figure 1. FTIR Results of Cinnamic Acid from the Esterification Process

Gas Chromatography-Mass Spectrometry (GC-MS) Analysis

The purity and molecular structure of cinnamic acid were further analyzed using GC-MS. The chromatogram showed a distinct peak corresponding to cinnamic acid, indicating successful isolation. This analysis was then compared with the mass spectrum reference from the NIST library. The mass spectrum confirmed the presence of cinnamic acid with a molecular ion peak at m/z 148, which matches the molecular weight of cinnamic acid ($C_9H_8O_2$). Additional fragment peaks at m/z 103 and 77 were observed, representing the characteristic fragmentation pattern of cinnamic acid. The high similarity index in the mass spectral database further verified the purity of the synthesized product.

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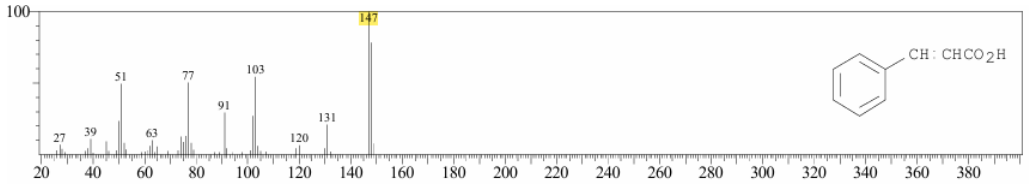

Figure 2. Mass Spectrum of Cinnamic Acid from the Esterification Process

4. CONCLUSION

This study successfully synthesized cinnamic acid from benzoin extract using sulfuric acid as a catalyst. The characterization results from FTIR and GC-MS confirmed the structural integrity and purity of the synthesized compound. The optimized reaction conditions provided a high yield, demonstrating an efficient and sustainable method for cinnamic acid production from natural sources.

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