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SYNTHESIS AND CHARACTERIZATION OF 7-HYDROXY-4-METHYLCOUMARIN DERIVATIVES VIA SOLVENT-FREE MODIFIED PECHMANN CONDENSATION

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This Final Year Project Report entitled **"Synthesis and Characterization of 7-Hydroxy-4-Methylcoumarin Derivatives via Solvent-Free Modified Pechmann Condensation"** was submitted by Nur Fatini Binti Abd Halim in partial fulfillment of the requirements for the Degree of Bachelor of Science (Hons.) Chemistry with Management, in the Faculty of Applied Sciences, and was approved by

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ABSTRACT

SYNTHESIS AND CHARACTERIZATION OF 7-HYDROXY-4-METHYLCOUMARIN DERIVATIVES VIA SOLVENT-FREE MODIFIED PECHMANN CONDENSATION

Coumarins represent an important group of naturally occurring heterocyclic compounds that have attracted considerable interest due to their wide range of biological activities, including antioxidant, anti-inflammatory, and antibacterial effects. These compounds are commonly synthesized via the Pechmann condensation reaction involving activated phenols and *β*-keto esters. Conventional approaches to coumarin synthesis typically employ strong liquid mineral acids such as sulfuric acid, which are highly corrosive, environmentally hazardous, and associated with waste management and disposal concerns. In response to these limitations, the present study focuses on the development of a greener and more sustainable synthetic approach for 7-hydroxy-4-methylcoumarin. The efficiency of solid organic acid catalysts, namely *p*-Toluenesulfonic acid (*p*-TsOH) and oxalic acid, was investigated under solvent-free conditions. The target compound was synthesized through a solvent-free Pechmann condensation reaction between resorcinol and ethyl acetoacetate, with both reactions conducted within 80-100 °C using catalytic quantities of the respective acids. The synthesized products were characterized to confirm their structure and purity using Ultraviolet–Visible (UV-Vis) spectroscopy, Fourier-Transform Infrared (FT-IR) spectroscopy, and Gas Chromatography–Mass Spectrometry (GC-MS). FT-IR spectra verified the formation of the coumarin ring system, with characteristic lactone carbonyl (C=O) stretching bands observed at 1664.97 cm^{-1} for the *p*-TsOH-catalyzed product and 1665.60 cm^{-1} for the oxalic acid-catalyzed product. UV-Vis analysis showed a maximum absorption wavelength (λ_{max}) at 320 nm, consistent with the extended π-conjugated benzopyrone structure. Among the catalysts studied, *p*-TsOH exhibited higher catalytic efficiency, yielding a greater product amount and producing spectra with improved sharpness compared to oxalic acid. GC-MS analysis further confirmed the identity of 7-hydroxy-4-methylcoumarin by the presence of a molecular ion peak at *m*/*z* 176. Overall, the results demonstrate that solid organic acids serve as effective and environmentally benign alternatives to conventional mineral acids, enabling a rapid, high-yield, and atom-economical synthesis of coumarin derivatives in accordance with green chemistry principles.

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