

Comparative Study on the Synthesis of Chalcone Using Conventional and Solvent-Free Methods.

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Introduction

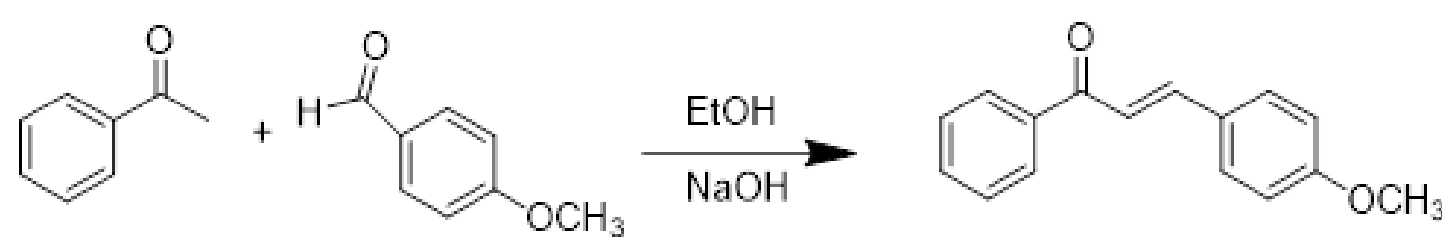
Chalcone ($C_{15}H_{12}O$) is an α,β -unsaturated aromatic compound found naturally and synthetically. It exhibits antibacterial, antioxidant, anti-inflammatory, and anticancer activities, making it important in green chemistry. This study aims to prepare the chalcone derivative 3-(4-methoxyphenyl)-1-phenylprop-2-en-1-one using both conventional and green methods.

Objectives

- To synthesize 3-(4-methoxyphenyl)-1-phenylprop-2-en-1-one using conventional and green methods.
- To compare the products using melting point (mp), thin-layer chromatography (TLC), infrared spectroscopy (IR), and nuclear magnetic resonance (NMR) analyses.
- To evaluate the efficiency and environmental impact of the green method.

Method

Reaction Equation:



Conventional method with Yield: 12.5%

- Mix 0.4 mL of Acetophenone with 10 mL of ethanol and 0.2 g of NaOH and 0.4 mL 4-methoxybenzaldehyde.
- Stir for 3h.
- Add ice-cold water.
- Liquid-liquid extraction (Et_2O).
- Dry the organic layer over ($CaCl_2$).
- Dry the residue overnight.
- Wash the precipitate with diethyl ether.

Solvent-free method with Yield: 43.13%

- grind 0.4 mL of Acetophenone with 0.2 g of NaOH and 0.4 mL 4-methoxybenzaldehyde.
- Grind for 20 min.
- dry for 2 hours.
- Add ice-cold water.
- Liquid-liquid extraction (Et_2O).
- Dry the organic layer over ($CaCl_2$).
- Dry the residue overnight.
- Wash the precipitate with diethyl ether.

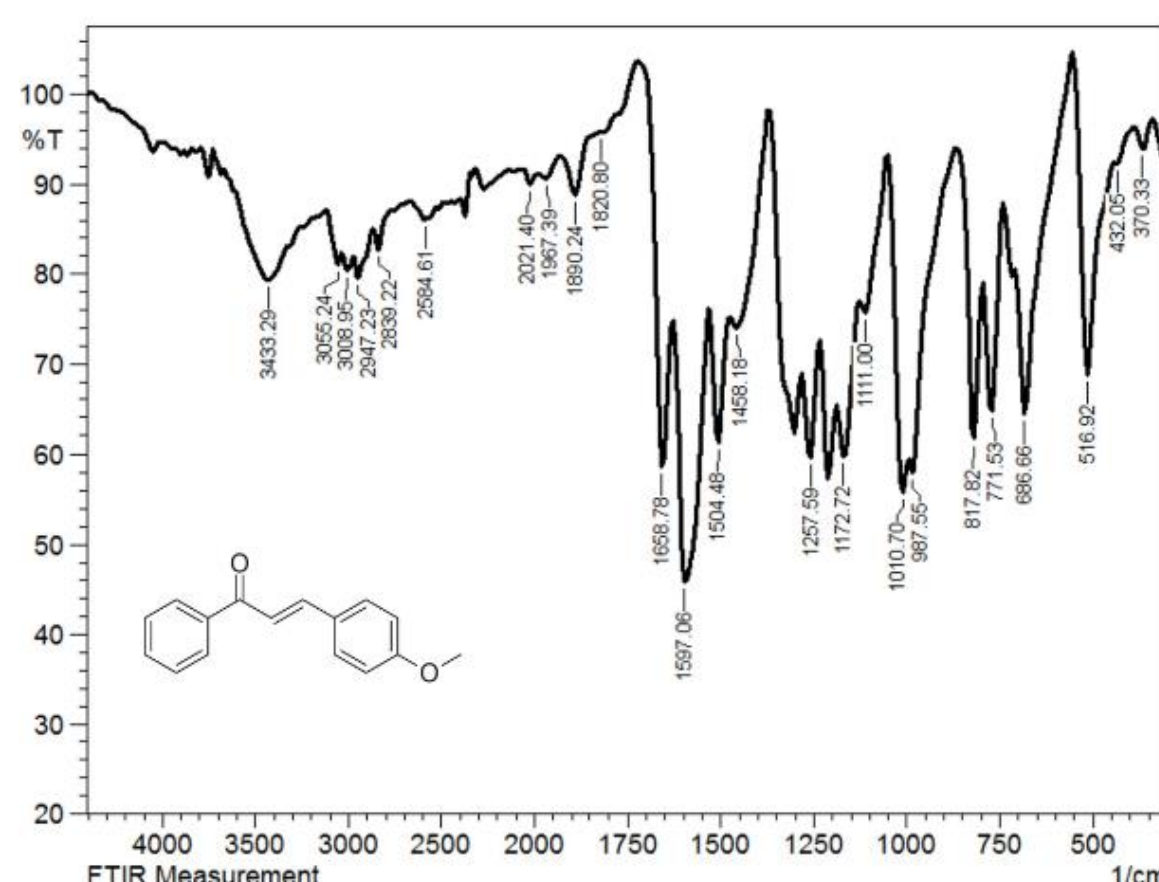
Results

The chalcone derivative was successfully synthesized by both the conventional and solvent-free methods, with melting points of $60-62^\circ C$ and $54-56^\circ C$, respectively.

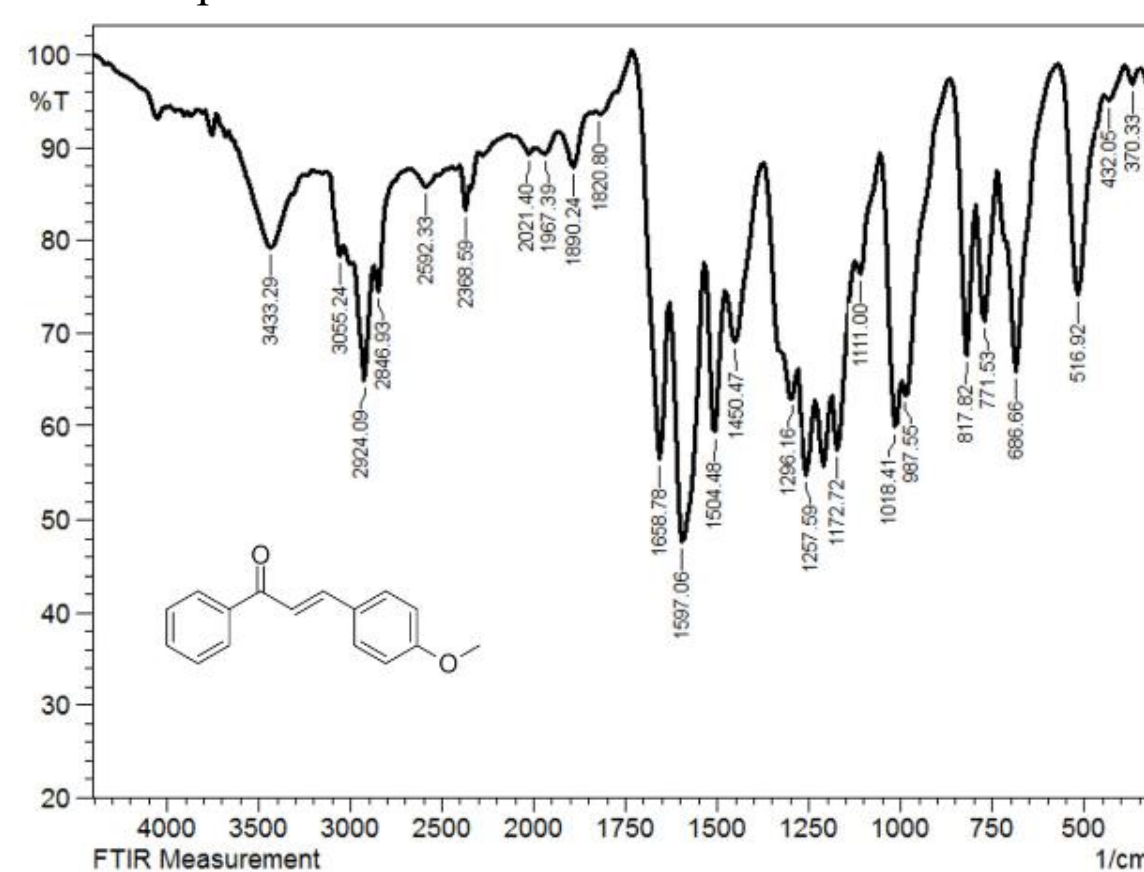
IR spectrum (cm^{-1}) (Figures 1 & 2):

The IR spectra of the product obtained from both the conventional and solvent-free methods exhibited identical characteristic absorption bands.

1658 ($C=O$), 1597 ($C=C$ alkene), 1504 ($C=C$ aromatic rings), 3055 (sp^2 C-H), 2930 and 2850 (sp^3 C-H), 1110 ($C-O$ methoxy group).



Figures 1: IR spectrum of chalcone obtained via conventional method.



Figures 2: IR spectrum of chalcone obtained via solvent-free methods.

1H -NMR spectra ($CDCl_3$), δ (ppm) (Figure 3):

3.84(s, 3H, O-CH₃), 6.9(d, $J=10$ Hz, 2H, p-OMe ring), 7.6(d, $J=10$ Hz, 2H, p-OMe ring), 8.0(d, $J=5$ Hz, 2H, H_a), 7.48(t, $J=7.5$ Hz, 2H, H_m), 7.58(t, $J=7.5$ Hz, 1H, H_p), 7.4(d, $J=15$ Hz, 1H, H-2), 7.8(d, $J=15$ Hz, 1H, H-3).

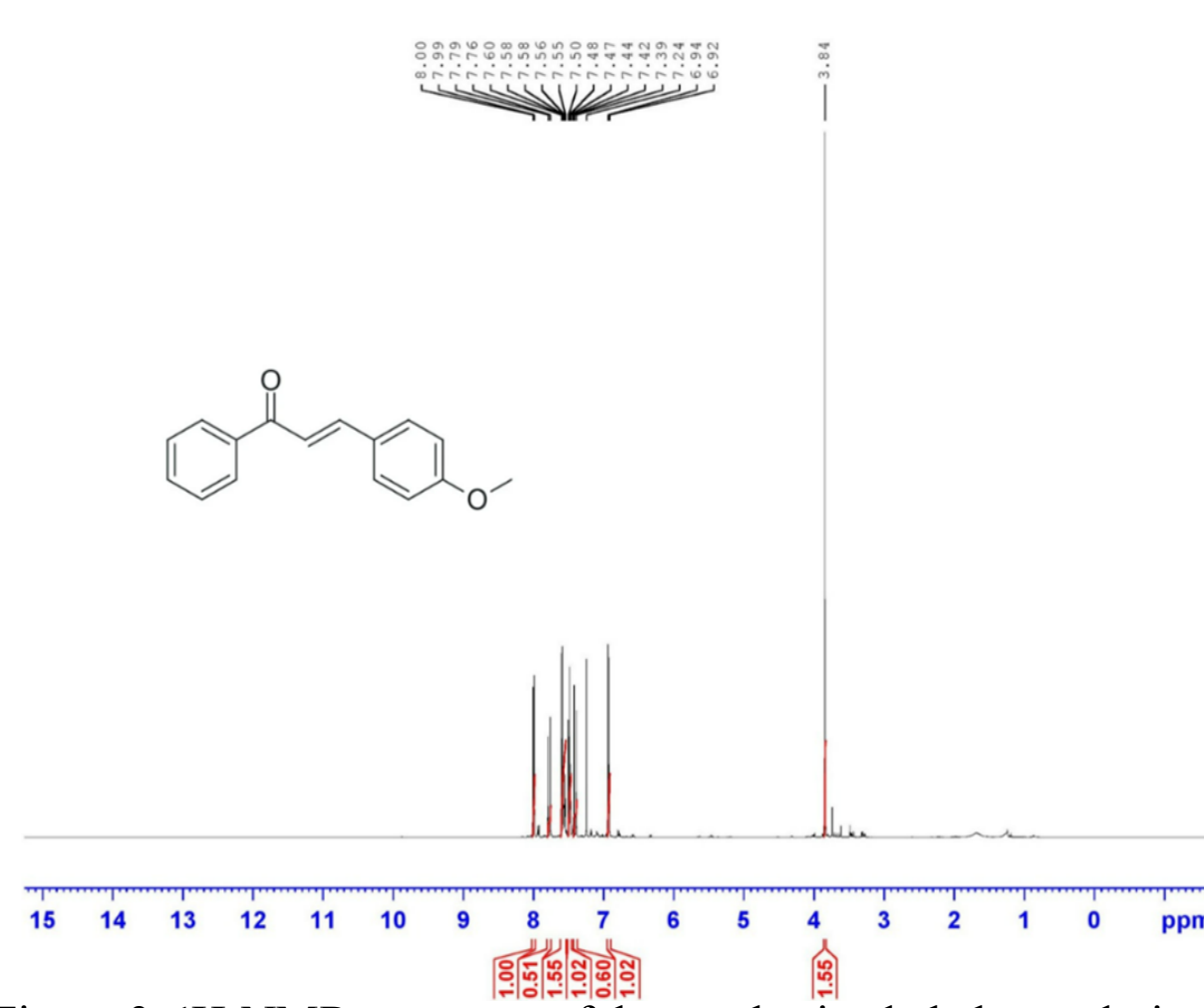


Figure 3: 1H -NMR spectrum of the synthesized chalcone derivative.

Results

^{13}C -NMR spectrum ($CDCl_3$), δ (ppm) (Figure 4):

55(methoxy carbon), 114-161(sp^2 hybridized carbon), 190($C=O$).

