Microwave assisted esterification using Fe$_2$(SO$_4$)$_3$.4H$_2$O/concentrated H$_2$SO$_4$ as efficient catalyst

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2, 4-D 1 (0.221 g, 1 m mole), Fe$_2$(SO$_4$)$_3$.4H$_2$O (0.423 g, 1 m mole) and conc. H$_2$SO$_4$ (0.098 mL) in absolute methanol (20 mL) was taken in RBF placed in a microwave oven and irradiated (300w, 67-68°C) for 5 min [1]. Upon completion of reaction (monitored by TLC), using petroleuemether-ethylacetate (8:2) as the eluent solvent system. The reaction mixtures was allowed to attain room temperature, after the completion of reaction the solvent was removed by vacuum distillation and treated with cold water. The liquid product separated washed with water to furnish compound 2, yield 90%.

Melting point: 134-136 °C

IR (KBr) (cm$^{-1}$): 1722 (>C=O of ester), 1225, 1044 (C=O of ester), 3023 (C=H, aromatic ring), 1510 (C=C, aromatic ring), 825 (Ar-Cl).

$^1$H-NMR (CDCl$_3$-DMSO-d$_6$) (400 MHz): $\delta$= 6.70-7.90 (3H, m, Ar-H), 4.0 (3H, s, -COOCH$_3$), 4.46 (2H, s, -CH$_2$).

$^{13}$C-NMR (CDCl$_3$-DMSO-d$_6$) (62.90 MHz): $\delta$= 115.29-134.1 (aromatic carbons), 170 (>C=O of ester), 20 (-COOCH$_3$), 35 (-CH$_2$).
MS (m/z): 235 (M+), 204 (C₈H₅O₂Cl₂⁺), 176 (C₇H₅OCl₂⁺), 59 (C₂H₃O₂⁺), 31 (CH₃O⁺).

Elemental Analysis: Calculated for C₉H₈O₃Cl₂: C 45.95, H 3.40; found: C 45.98, H 3.43.

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References

Sample Availability: Available from MDPI.

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