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CHARACTERISATION AND SPECTRAL ANALYSIS OF COUMARIN DERIVATIVE

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ABSTRACTS

A clean coumarin derivative were synthesized using recent technology of microwave irradiation, a comparative study was done by using conventional method and microwave irradiation technique, the study includes comparison between yield and time. The present study reveals the synthesis of coumarin derivative in one step in which 2-oxo-2H-1-benzopyran-4-carbaldehyde is treated with hydrazine hydrate to give desired product conventionally and also by microwave irradiation technique, the target compounds were Characterized to deduce the structure.

KEYWORDS: Coumarin, NMR, Microwave Irradiation, Convetional.

INTRODUCTION

Coumarin belongs to heterocyclic ring system which includes aromatic moiety benzene ring fused to a pyran ring. Coumarin contains the basic backbone of various types of phenols which is present in alkaloids, tocopherols, anthocyanins.[1] Uptill recent advances in the research development coumarin molecule is reported to be having many such activities such as antihelmenthic[2], antihepatits[3], antidengue[4], anti-malarial[5], herbicidal, analgesic and anticonvulsant[6] activity. Here in this we aim to synthesize new Coumarin derivatives and to investigate their antimicrobial and anti inflammatory activities.

METHODS AND MATERIALS

Raw materials used in this experiment was obtained from M/S Fluka AG (Bachs, Switzerland) and M/S Sigma-Aldrich chemicals and Co. Inc. (Milwoukee, WI,USA). are not corrected. IR spectra were established by using IR-Affinity, Shimadzu using DRS system.

^{1}H-NMR spectra was established on a JEOL AL-300 FT-NMR spectrometer (300 MHz, JEOL Ltd., Tokyo, Japan), using TMS as internal standard. Mass data has been recorded on Agilent GC-MS carried on GC7890 MS 200 of Agilent, Microwave oven for synthesis Monowave 300 Anton Parr.

EXPERIMENTAL

Synthesis of ethyl (pyrazin-2-yloxy)acetate (Compound 2) Conventional method coumarin derivative (0.01 mol) and hydrazinehydrate was dissolved in acetone stirred for 3 hours. Reflux the reaction mixture leading to formation of title compound. Reaction monitoring was done by TLC. Product was isolated by drowning into cold water. Aqueous layer was extracted with10 mL Diethyl ether for three times to extract product. Recrystallized the product with acetone. Yield 48%; Colourless solid; mp:234^{0}C, ^{1}H NMR(400 MHz, DMSO-δ6) δ (ppm) 5.9 (s, 1H), 3.1(s, 2H), 7.06-8.08 (m, 4H, Ar-H).IR (KBr) cm^{-1}: 1279(C-N), 3486 (- N=) MS (m/z): 202 [M^{+}] ($C_{11}H_{10}N_2O_2^{+}$),187 ($C_{11}H_9NO_2$), 146($C_9H_6O_2$).

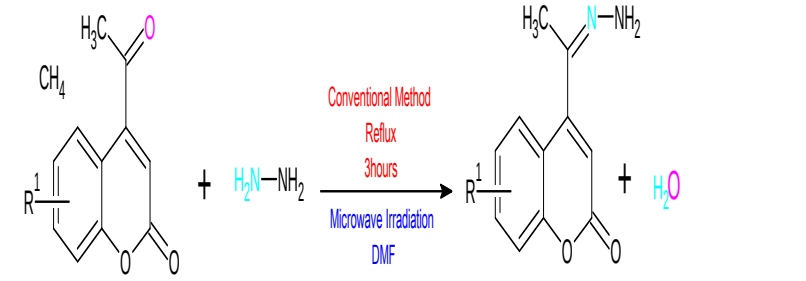
Synthesis of ethyl (pyrazin-2-yloxy) acetate (Compound 2) Microwave-Irradiation method

Coumarin Derivative (0.01mol) was dissolved in DMF. To this solution Hydrazine hydrate solution was added (0.01mol). The reaction mixture was irradiated for 210seconds. The crude product was cooled to room temperature, pour the reaction mixture to ice cold water, product was obtained. Filtered the product; wash with water. Recrystallized the product with Ethyl alcohol**,** Yield 87%; Colourless solid; mp:232^{0}C, ^{1}H NMR(400 MHz, DMSO-δ6) δ (ppm) 5.6 (s, 1H), 2.3(s, 2H), 7.11-8.28 (m, 4H, Ar-H).IR (KBr) cm^{-1}: 1210(C-N), 3456 (- N=) MS (m/z): 202 [M^{+}] ($C_{11}H_{10}N_2O_2^{+}$),187 ($C_{11}H_9NO_2$), 146($C_9H_6O_2$).

Observation

Compound	Substituent	Conventional		Microwave		Melting Point
		Time in (Hours)	Yield in (%)	Time in (Seconds)	Yield in (%)	(in ^{0}C) M.P/B.P
2a	H	3	48	210	87	232
2b	-OCH_3	9	65	209	85	244
2c	-Cl	5	66	216	89	237
2d	NO_2	5.5	52	234	92	349
2e	-COOH	6	67	189	77	277

SCHEME



4-acetyl-2*H*-1-benzopyran-2-one

Compound 1

4-[(1*Z*)-1-hydrazinylideneethyl]-2*H*-1-benzopyran-2-one

Compound 2

RESULT AND DISSCUSSION

Compound 2d has shown yield of 92% when synthesized by using microwave-technique where as conventional method has produced yield of 52%, same in case for 2c yield from conventional method is low as 66% then in microwave technique with 89%.

CONCLUSION

The microwave method was found to be better then conventional method in terms of reaction time, yield and relatively simple method to perform synthesis.

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