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Research Article

DEVELOPMENT OF MICROWAVE ASSISTED SYNTHESIS FOR COMMON MOLECULES

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ABSTRACT

Microwave synthesis are attractive in offering reduce pollution, low cost and high yields with shorter reaction time together with simplicity in processing and handling. In this study, the compounds like β-Resorcylic acid & Benzocaine were synthesized by microwave irradiation. The compounds were synthesized rapidly with more yields as compared to the conventional method. The synthesized compounds were subjected to physico-chemical studies like melting point, TLC, partition coefficients, dissociation constants and % ionization. The structures of synthesized compounds were characterized by UV, FT-IR, MASS and NMR spectroscopy.

Keywords: Microwave irradiation, β-Resorcylic acid, Benzocaine, Physicochemical

INTRODUCTION

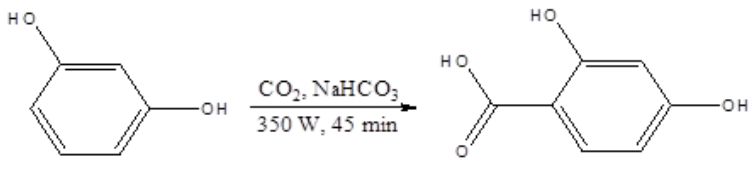
Practical's in organic chemistry include the synthesis and evaluation of organic and pharmaceutical compounds which require more than 2-3 h heating for synthesis. Further purification, drying and evaluation of the synthesized compound is not possible within a single practical. To overcome these problems, microwave assisted synthesis was developed for organic compounds that require time in minutes only. Microwave heating can offer a rapid and efficient alternative to conventional oil-bath, sand-bath, or steam bath technology. It offers reduced reaction time, lower cost, simplicity in processing, reduced pollution, high yield, no contact required between the energy source and the reaction vessels.[1-3]Keeping the above in view, β-Resorcylic acid & Benzocaine were synthesized by microwave irradiation. The synthesized compounds were subjected to physico-chemical studies like melting point, TLC, partition coefficients, dissociation constants and % iosnization. The structures of synthesized compounds were characterized by UV, FT-IR, MASS and NMR spectroscopy.[4-8]

MATERIALS AND METHODS

All the chemicals used in the reaction were of AR grade. For synthesis, CATA's Scientific Microwave Synthesis System was used. Melting point was determined by open capillary method using DBK programmed melting point apparatus and uncorrected. The purity and homogeneity of the compounds as well as completion of reaction times was checked by thin layer chromatography. The spots were visualized by iodine vapours and visualized with U. V. light. All the compounds were purified by preparative TLC. The IR spectra of all the compounds were recorded in FT-IR (Model: ShimadzuFT-IR - 8400 S) using KBr pellets in the region of 4000-500 cm^{-1}. The NMR spectra were recorded in Bruker Avaze II at a frequency of 400 MHz and the Mass spectra were recorded on QP- 2010 PLUS GC-MS.

Synthesis of β- Resorcylic acid

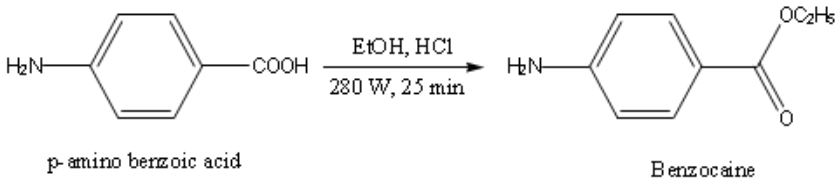
In 50 ml two necked flask,0.55 g of resorcinol, 2.77 g of sodium hydrogen scarbonate and 5.55 ml water were placed. The mixture was refluxed by microwave irradiation in scientific microwave oven for 45 min (Power input: 350 W, 5 P). Then the solution was refluxed over a flame for 30 min while passing a rapid stream of CO_2. The solution was acidified with 2.5 ml conc. hydrochloric acid. The solution was cooled in ice bath, collected the acid by filtration and recrystallized from hot water using a little decolorizing carbon.[9] The purity of product was checked by thin layer chromatography using acetonitrile: n-hexane as mobile phase in the ratio of 8:2.



Resorcinol ß- Resorcylic acid

Synthesis of Benzocaine

In 10 ml two necked flask, 0.6 g p-amino benzoic acid and 4 ml absolute ethanol (previously saturated with dry hydrogen chloride) were placed. The mixture was refluxed by microwave irradiation for 25 min (power input: 280 W, 4 P). The hot solution was poured into excess of water and sodium carbonate was added until it was neutral to litmus. The product was filtered off and dried. The recrystallization was carried out by rectified spirit.[9] The purity of product was checked by thin layer chromatography using chloroform: ethyl acetate as mobile phase in the ratio of 9:1.



p- amino benzoic acid Benzocaine

Reaction time of products by Conventional and MWI method

Compound	Conventional method[9]		Microwave method		
	Time (min)	Yield (%)	Time (min)	Output (Watt)	Yield (%)
β- Resorcylic acid	240	64	45	350	70.32
Benzocaine	120	69	25	280	77.73

Characterization

Formation of β-Resorcylic acid & Benzocaine were confirmed by IR, NMR and Mass spectral data. The characterization data of the synthesized compounds has been given as below:-

β- Resorcylic acid:

IR (KBr) (cm^{-1}): 3250-2500 cm^{-1}(-OH stretching of carboxylic acid), 1650.95 cm^{-1}(C=O stretch). GC-MS (m/z) = 154(M^+). ^{1}H NMR (400 MHz, DMSO) : δ 11.39 &11.42 (s, 1H of OH), 10.42 (s, 1H of COOH), 7.26 (s, 1H), 7.37-7.41 (m, Ar-H), 7.84-7.86 (m, Ar-H).

Benzocaine:

IR (KBr) (cm^{-1}): 3000-2800 cm^{-1}(C-H stretching of CH_2& CH_3), 1685.67 cm^{-1} (C=O stretching of ester), 3425 & 3340.48 cm^{-1} (asymmetric & symmetric N-H stretching of aromatic amine), 1635.52 cm^{-1}N-H deformation, 3050 cm^{-1} (aromatic C-H stretching). GC-MS (m/z) = 165 (M^+).

^{1}H NMR (400 MHz, $CDCl_3$) : δ 6.62-6.65 (d, J = 2.01, 1H), 7.84-7.87 (d, J = 1.96, 1H), 4.28-4.34 (q, J = 2.02, 2H), 1.34-1.37 (t, J = 3.00, 3H).

RESULTS AND DISCUSSION

The β-Resorcylic acid & Benzocaine were prepared by microwave irradiation method. The reaction yield, melting point, R_f value[10] and other physicochemical data of synthesized compounds are given in table.

Compound	Molecular formula (Mol.Wt.)	Nature	Melting point (^{0}C)	R_f value	Partition coefficient[11](P)	Dissociation constant[12](pKa)	% Ionization
β- Resorcylic acid	$C_7H_6O_4$ (154)	Faint yellow crystals	218-220	0.61	5.84	3.70	20.45%
Benzocaine	$C_9H_{11}NO_2$ (165)	Brown crystals	89-90	0.60	2.57	2.45	40.89%

CONCLUSION

A convenient microwave assisted synthesis of organic compounds was developed. The method offers several advantages including good to high yields and an easy experimental work-up procedure. The reaction time has brought down from hours to minutes. Overall decrease in heating time effectively reduces the cost of fuel or electricity. The physico-chemical parameters and spectral analysis of all the compounds were studied. Thus, the newly developed procedure in this study is economic, rapid and simple. Microwave irradiation facilitates the polarization of molecules causing rapid reaction to occur. In conclusion, we have successfully adopted the use of microwave technique for routine practical classes in chemistry laboratories.

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