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MICROWAVE INDUCED SYNTHESIS OF ANTHRAQUINONE DERIVATIVES A SOLVENT FREE PATH

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ABSTRACT

Synthesis of Anthraquinone dyes was carried out in a microwave oven under solvent free conditions. The reactions were found to follow condensation process. Formation of products was governed through tlc and were confirmed by spectral analysis. The process with lesser reaction time, single pot synthesis without using costly solvents provides a newer path for synthesis of these dyes.

Key words: Microwave irradiation, Solvent free, tlc, spectral analysis, single pot

INTRODUCTION

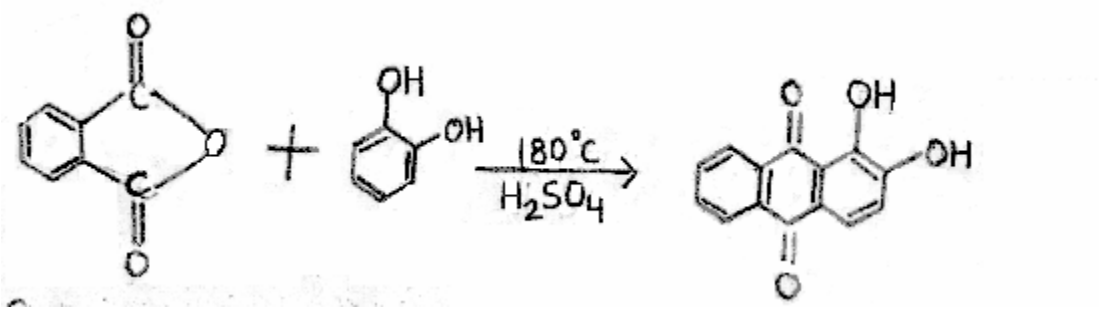
Microwave radiation lies in the electromagnetic spectrum in between the radio wave and infrared frequency. As the wave length of microwave radiation is large, these are not capable of ionizing or breaking bonds and thus are considered as heat radiations.

Microwave radiations are now widely used as unconventional source of energy for synthesis of many organic substances and have variety of applications[1-3] . Organic molecules absorb the microwave energy selectively enhancing the rate of reaction. The more chemistry was earlier restricted to the use of high boiling point polar solvents like DMSO, DMF etc. Later few low boiling point solvents like Toluene[4] were used but were found to generate serious hazards.

To overcome the problem in microwave synthesis, later on solvent free path[5-6] was developed. This dry media synthesis is now widely used. Varma[7] has carried out solvent free synthesis of thioacetones and thioamides. Bram et al[8] made use of catalysts and solid support. Pasha et al[9] carried out synthesis of amides from carboxylic acids and urea in presence of pyridine under microwave irradiation.

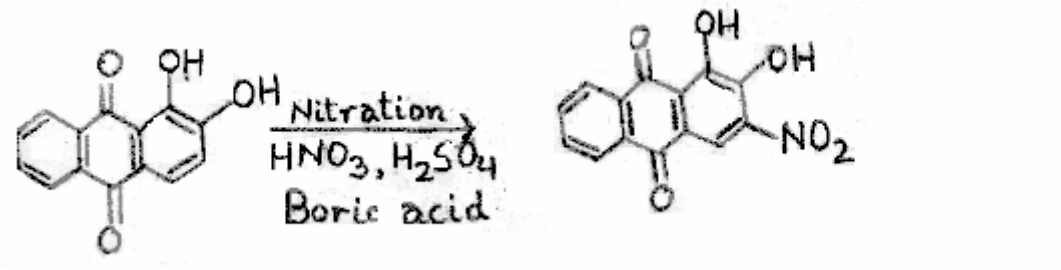
EXPERIMENTAL

Compound-1 was prepared by taking catechol and phthalic anhydride in a microwave oven in presence of concentrated H_2SO_4. Brown precipitate obtained on irradiating is then treated with dilute NaOH and recrystallized by dilute HCl. The reaction is given as-



Scheme-1

Compound-2 is prepared by nitration of Alizarin by nitrating mixture in presence of boric acid in a vessel and is irradiated in a microwave oven. Formed liquid product is cooled and to this water is added. The crystalline product is then recrystallised by adding first dilute NaOH and then precipitating by adding dilute HCl. Orange colored crystals are obtained. The reaction scheme is given as-



Scheme-2

RESULTS AND DISCUSSION

The anthraquinone derivatives are used as coloring agents in various industries like textile, plastic etc. For synthesis of these compounds through traditional method was a tedious job with lots of equipment arrangement, large amount of solvent requirement and along with all these a longer time span for completion of the reaction. The time required for these reactions may range from hours to days.

An alternate path of synthesis of these compounds in domestic microwave oven in a single pot without using solvent and with drastically reduced time span is suggested and data of analysis are given. Physical data are given in table-1 of these compounds.

Table-1:Physical data of [1]and[2]

S.No.	Molecular formula	Molecular weight	M.P.(^oC)	Reaction time(min.)	λ_{max}	% yield
[1]	$C_{14}H_8O_4$	240.21	275	3	605&569	64.2
[2]	$C_{14}H_7O_4N$	254.30	---	21	653	53.8

Spectral Analysis:

Compound-1 %C-53.84, %H-30.76 and %O-15.40, ν_{max}, nujol- 3754.3, 3480.0(–OH str), 3085.8(Ar C-H str), 2653.9, 2525.7(int mol H bonded –OH)2990.0(Ar C-H str), 1677.3(Aryl ketone >C=O str), 1591.1, 1490.3(Ar C-C multiple bond str), 1387.5(Phenolic –OH bend, C-O str), 1281.7(C-O vib), 1150.0(Tert –OH group), 1070.0(Ar C-H bending), 903.8, 767.7(Ar C-H vib), δ_H $CDCl_3$ TMS, 5.92(Ar-OH intramolecular bonded, 2H, s), 6.72(Ar-H, 1H, d), 6.86(Ar-H, 3H, d), 7.02(Ar-H,2H, d), 7.84(Ar-H, 2H,d)

Compound-2 %C-53.84, %H-26.92 and %O-15.38, ν_{max}, nujol- 3754.3, 3480.0(–OH str), 3085.8(Ar C-H str), 2653.9, 2525.7(int mol H bonded –OH)2990.0(Ar C-H str), 1677.3(Aryl ketone >C=O str), 1592.6(Ar NO_2 str) 1490.3(Ar C-C multiple bond str), 1387.5(Phenolic –OH bend, C-O str), 1361.4(Ar NO_2), 1281.7(C-O vib), 1150.0(Tert –OH group), 1070.0(Ar C-H bending), 903.8, 767.7(Ar C-H vib), δ_H $CDCl_3$ TMS, 6.23(Ar-OH intramolecular bonded, 2H, s),7.01(Ar-H, 2H, d), 7.12(Ar-H, 2H, t), 8.23(Ar-H, NO_2 influenced, 1H, s).

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