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MICROWAVE INDUCED SYNTHESIS OF ANTHRAQUINONE COMPOUNDS : A SOLVENT FREE PATH FOR SOME DYES

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ABSTRACT

Synthesis of anthraquinone derivatives was carried under microwave irradiations. The reaction was found to follow condensation mechanism with removal of water molecule. Formation of product was confirmed through TLC, melting point and spectral analysis. The reaction was carried out in a single pot without using solvent in a domestic microwave oven.

Key words : Microwave irradiations, One pot synthesis, Dyes, Solvent free

INTRODUCTION

Microwave dielectric heating is widely exploited for acceleration of organic reactions[1] during the last one decade. There is an increasing interest in this method because of following advantages.

(i) One pot synthesis

(ii) Shorter time required for completion of reaction.

(iii) Higher yield

(iv) Purity of the products

(v) Solvent free reactions

(vi) Energy is not wasted even on bulk heating of the material.

In past few years due to numerous advantages[2], microwave technology has been used widely in household cooking as well as in the field of synthesis of organic compounds like dyes, drugs, carbohydrates etc.

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This non-conventional method has various advantages over conventional methods. A lot of work has been done in past decades. Reactions are generally of three types; (i) reaction between neat reactants, (ii) reaction of reactants, which are supported on a solid base in dry media like silica, alumina etc. and (iii) reactions in solvents.

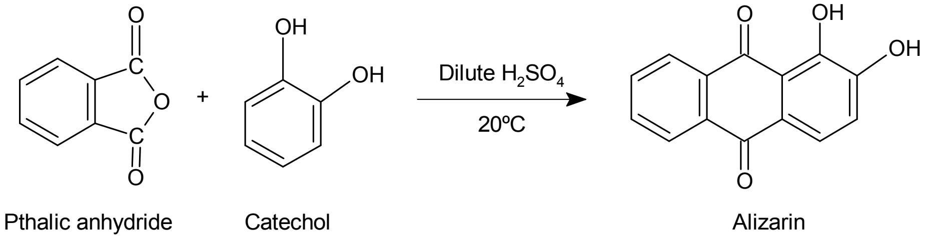
A solvent free synthesis of 2-hydrazinobenzothiazole derivative using microwave has been reported[3]. Some flavones have been synthesized under microwave irradiation. Microwave assisted solvent free synthesis of substituted cromenes was carried out by Meenakshi et. al[4]; Solid state induced heterocyclization under microwave irradiation for synthesizing of 2-phenyl-3-hydroxy-quinoline-4(H)-1 was reported by Heravi et al.[5]; Solvent free improved synthesis of some substituted 1-3-diaryl propones and 3, 5-diaryl-6-carbethoxy cyclohexenione under microwave irradiation was carried out by Jhala et al.[6]; Solvent free synthesis have also been carried out by other workers[7,8].

EXPERIMENTAL

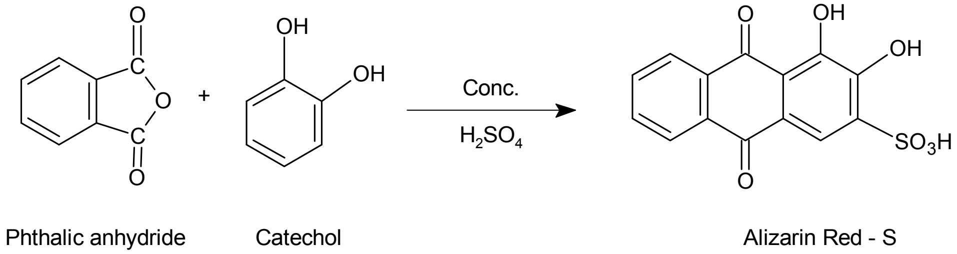
Compounds were prepared by taking phthalic anhydride, catechol, dilute H_2SO_4 or conc. H_2SO_4 in separate vessels and the mixture was irradiated in microwave at low intensity. After formation, it was recrystallisted with alcohol, where red-orange crystals and orange crystals were obtained. Then their melting points were recorded.

RESULTS AND DISCUSSION

Synthesis of anthraquinone derivatives was carried out in a single pot without using solvents in a domestic oven. The product formed was confirmed through TLC and by spectral studies. Formation of compounds viz. Alizarin and Alizarin red-S followed the reaction pathway as shown in **Schemes 1** and **2**.



Scheme 1



Scheme 2

Table 1. Physical properties of (1) and (2)

Compound	Molecular formula	Molecular weight	Melting point (°C)	Reaction time (minutes)	% Yield
Alizarin	$C_{14}H_8O_4$	240.2	289	5 -7 min.	62.50
Alizarin Red-S	$C_{14}H_8O_7S$	320.6	283	35 min.	80.00

Analytical data of compounds

Compound 1 :

Elemental analysis – Carbon – 69.0%, Hydrogen – 26.50 %, O – 3.50%

IR Spectrum : ν_{max}, [Nujol (cm^{-1})]

1587 – 1455	(Aromatic system)
1587 – 1455	(C = C, s)
1631	(C = O, m)
2922 – 2854	(C – H, s)
1296	(C – O, s)
3372	(C – OH, br, s)

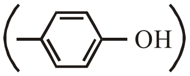
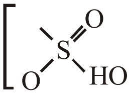
Mass spectrum : m/z

Molecular ion peak – 239.2, base peak of first fragment – 165.1, peak of second major fragment 121.1.

Compound 2 :

Elemental analysis – Carbon – 53.50%, H – 3.50 %, O – 32.00%, S – 9.50%.

IR spectrum : ν_{max}, [Nujol (cm^{-1})],

1768.80 – 1683.32	(C = O),
1501.76	(Aromatic system),
1587.81 – 1453.87	(C = C) aromatic stretching,
2904.74	
3737.39 – 3673.96	
3100.73 – 3007.23	(C – H Stretching)

Mass spectrum : m/z

Molecular ion peak – 319.1, base peak of first fragment – 165.1, peak of second major fragment 121.1.

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