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NOTE

Microwave-Assisted Solvent Free Synthesis of Some 4,6-Dicinnamoyl Resorcinols

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4,6-Dicinnamoyl resorcinols (**2a-h**) were synthesized in high yield (90-95 %) using a microwave-assisted solvent free condensation reaction of 4,6-diacetyl resorcinol (**1**) and aromatic/heteroaromatic aldehydes under alkaline medium. Products were recrystallized from benzene. Synthesis of 4,6-dicinnamoyl resorcinols under microwave irradiation is found much easier and faster than conventional method.

Key Words: 4,6-Diacetyl resorcinol, Aromatic/heteroaromatic aldehydes, Microwave irradiation, Solvent free.

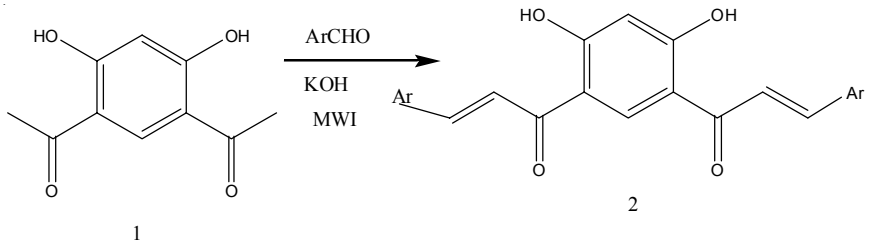
Many heterocyclic compounds show wide range of biological activities[1-4], notable among which are the antiviral, antibiotic, anticancer, antihypertensive and antiinflammatory activities. Hence they were claimed to be potential biologically active compounds. Therefore, in recent years much attention has been paid to develop novel biodynamic heterocyclic compounds.

4,6-Dicinnamoyl resorcinols (**2a-h**) are key intermediates for the synthesis of novel *bis* and mixed heterocyclic compounds. Microwave assisted synthesis has become popular in organic synthesis as it is eco-friendly[5-7]. The microwave assisted organic reactions occur more effectively and in an economical manner with enhanced product purity and chemical yields[8,9]. Microwave assisted synthesis under solvent free condition[10] is less expensive and environment friendly. This technique is more popular in library synthesis, where compounds can be rapidly synthesized by multi component single step. In view of the environmental benign synthesis under microwave irradiation and as a part of our ongoing research programme towards the Green Synthesis, the concept of Microwave assisted Organic Reaction Enhancement (MORE) chemistry has been adopted for the rapid and efficient synthesis of 4,6-dicinnamoyl resorcinols (**2a-h**). It was also decided to investigate the influence of microwave irradiation on condensation of 4,6-diacetyl resorcinol and aromatic/heteroaromatic aldehydes in alkaline medium.

All the chemicals used in the reactions are of reagent grade quality. IR spectra were performed on Perkin-Elmer spectrophotometer. Reaction proceed was routinely monitored by thin layer chromatography (TLC) on silica gel-G plates. Reactions were performed in a Samsung microwave oven with 800w output. All products were characterized by comparison of melting points and their spectra (superimpossible IR) with those of authentic samples which were prepared under conventional method.

General procedure: 4,6-Diacetyl resorcinol (0.01 mol) aromatic/hetero-aromatic aldehydes (0.022 mol) and KOH were placed in a 150 mL Borosil beaker. This beaker was zapped into the microwave oven and subjected to microwave irradiation at 100 watts for 2-3 min. The crude products were recrystallized from benzene.

4, 6-Dicinnamoyl resorcinols (**2a-h**) were prepared by condensation of 4,6-Diacetyl resorcinol (**1**) with appropriate aromatic/heteroaromatic aldehydes in alkaline medium under microwave irradiation (**Scheme-I**).



1 2

Ar: a = phenyl; b = *p*-methoxy phenyl; c = *m*-methoxy phenyl;
d = *p*-methyl phenyl; e = *o*-chloro phenyl; f = *p*-chloro phenyl
g = 2-thienyl; h = 2-furyl

Scheme-I

TABLE-1
COMPARISION OF TIME AND YIELDS IN THE FORMATION OF SOME 4,6-DICINNAMOYL RESORCINOLS UNDER MICROWAVE IRRADIATION AND CONVENTIONAL METHOD

Entry	Aldehyde	Microwave irradiation		Conventional method	
		Time (min)	Yield (%)	Time (h)	Yield (%)
2a	Benzaldehyde	2	90	12	60
2b	*p*-Methoxybenzaldehyde	2	95	10	70
2c	*m*-Methoxybezaldehyde	2	92	12	68
2d	*p*-Methylbenzaldehyde	2	94	10	71
2e	*o*-Chlorobenzaldehyde	3	90	12	72
2f	*p*-Chlorobenzaldehyde	2	92	10	65
2g	Thiophene-2-carbaxaldehyde	3	92	12	60
2h	Furfural	3	94	12	65

Conclusion

In summary, this work demonstrates a rapid, efficient and environmentally friendly method of synthesis of 4,6-dicinnamoyl resorcinols under microwave irradiation and results obtained confirm the superiority of microwave irradiation method over the conventional method.

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ERRATUM

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Dissolution Kinetics of Calcined Ulexite in Ammonium Chloride Solutions

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1. In Introduction part, line 20 (p. 2002) read **ammonium chloride** instead of ammonium sulphate.
2. p. 2003, line 1, read **colemanite** instead of magnesite.
3. Reference 11, Read G. Tekin, Bau Fen Bil. Enst. Dergisi, Turkey, p. 100 (2004).
4. Reference 12, Read C. Kum, M. Alkan, M.M. Kocakerim, *Hydrometallurgy*, **36**, 259 (1994).