Microwave Synthesis of Chalcone Using Zinc phosphate as a Catalyst

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Abstract: **A new series of chalcone containing 1,3,5-triazine substituted derivative were synthesized. Microwave induced Synthesis of substituted 1-(4-(4,6-bis(4-substituted phenylamino)-1,3,5-triazin-2-lamino)phenyl) -3 -(4-substitutedphenyl) prop-2-en-1-one derivatives using $Zn_3(PO_4)_2$ Catalyst. The entire synthesized derivative was confined using spectral data. The microwave assisted synthesis shows high yield in a limited period of time.**

Keyword: Chalcone, microwave, spectral data.

INTRODUCTION

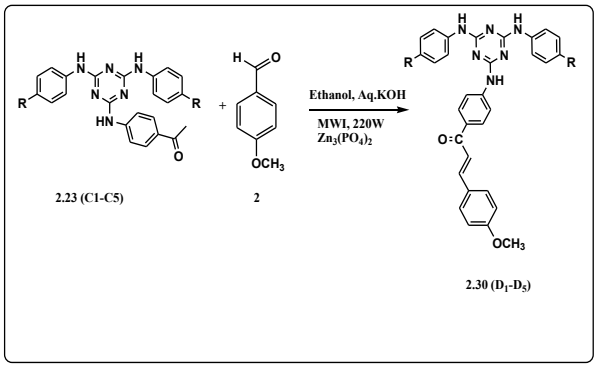
Chalcone (1,3-diaryl-2-propene-1-ones) are natural or synthetic compounds that can be normally found in a variety of plant species.[4] These chemicals have a wide range of biological properties, including anti-inflammatory[5], antipyretic[6], antimutagenic[7], antimalar- al[8], anticancer properties.[9], antiviral[10], Antifungal[11], Antibacterial[12]etc. Chalcones are made by two reactions: Claisen-Schmidt condensation, base-catalyzed aldol condensation, and acid-catalyzed aldol condensation between properly substituted aldehydes and ketones, followed by dehydration.[13,14] The chalcones are distinguished by their structural variety.[15] One or more aryl substituent's, which include halogens, alkyl, amino, nitro, acetamido, and carboxyl groups, may be included in the synthetically generated chalcones.[14,15]

In fact, many chalcone and their synthetic analogues exhibit a wide range of biological activities that are highly valued in many areas of modern life, including medicine for the treatment of cancer, cardiovascular disease, viral and parasitic infections, food industry as food additives, agriculture as a growth promoter and to control weeds and undesired pests, and food industry as food additives.[16, 17, 18] Here MWI induced Pyrenyl chalcone is formed using Zinc Nitrate is reported.[19]

EXPERIMENTAL

MATERIALS AND METHODS: - Aldrich chemicals provided all of the chemicals and solvents, and TLC-pre-coated plates were utilized to track the reaction's progress. Thermex Thermatron System Pvt. Ltd. is the manufacturer of the microwave oven. The microwave oven was used throughout the whole reaction. Recrystallization was used to purify the produced compounds, and IR and physical constants were used to describe their structures. Under UV illumination, they displayed a single spot on the TLC plate; uncorrected melting temperatures were recorded in open capillaries using a melting point equipment. The SHIMADZU IR Affinity-1 was used to perform infrared spectroscopy using the potassium bromide (KBr) pellet technique. The predicted structure was validated by analyzing the produced compounds' IR and 1HNMR spectra. Chemdraw Ultra 8.0 (Chemoffice 2004, Cambridge Soft, and Cambridge, USA) was used to verify IUPAC names.

Scheme-I



PROCEDURE: - An Equi-molar amounts of compound 2.23 (C_1-C_5) (2 mmole) and P-methoxybenzaldehyde (2 mmole) and $Zn_3(PO_4)_2$ (0.3g, 1.2mmole) were taken in closed vessel containing cap. In Microwave oven, the mixture irradiate for 10 min at power output was 220 watts. After cooling, organic layer was separated using dichloromethane. After evaporation it gives Pale yellow product.

Table-1: Optimization of Zinc Phosphate catalyst

Entry	Catalyst	Quantity (g)	Time (min)	Yield (%)
2.30.D_1	$Zn_3(PO_4)_2$	0.1	10	78
2.30.D_2		0.2	10	75
2.30.D_3		0.3	10	90
2.30.D_4		0.4	10	80
2.30.D_5		0.5	10	75

Table-2: Synthesis of tri-substituted chalcone under optimized condition using Microwave induced method

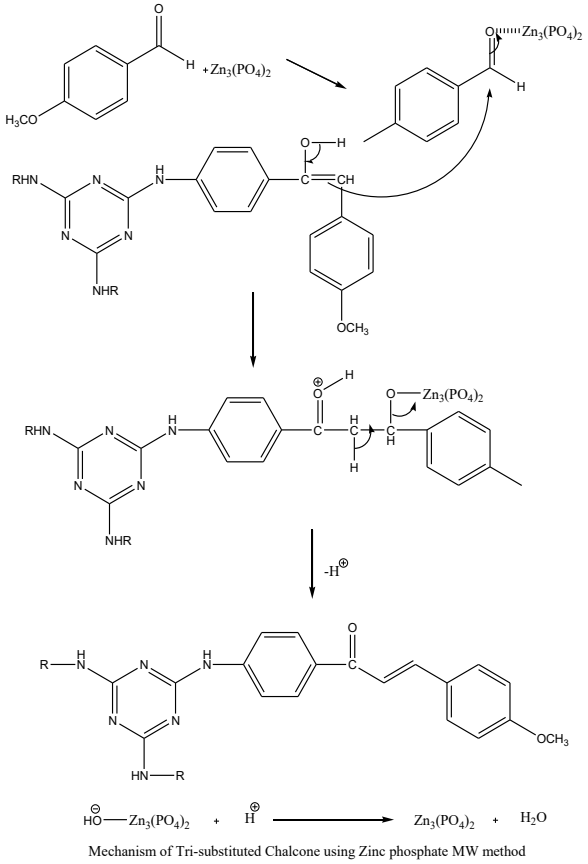
Compound	R	Observed M.P (^{0}C)	% Yield	R_f Value
2.30.D_1	H	247	85	0.95
2.30.D_2	P-OCH_3 C_6H_5	256	87	0.91
2.30.D_3	P-NO_2 C_6H_5	260	88	0.90
2.30.D_4	P-OH C_6H_5	252	89	0.93
2.30.D_5	P-Cl C_6H_5	254	90	0.92

Spectral data for CV/US/MW

Table-3: IR, NMR & CMR Spectral data for CV/US/MW synthesis of tri-substituted triazine

Comp.	IR (cm^{-1})	H-NMR	^{13}C-NMR
2.30.D_2	3310(N-H), 823 (C-N triazine), 1410 (-C=N), 3309 (Ar-OCH_3), 1595 (-CO-C=C-), 1651 (-CO-)	6.88 (d, 1H, –CO–CH=), 7.08-7.6 (M,12H,ArH), 7.8-8.3 (d,Ar-H,), 3.7-3.8 (S,9H,-OCH_3)	δ=188 (-CO), 146(C=N of s-tri.), 162(C-S-tri), 161(Ar-OCH_3), 56(-OCH_3), 113-119 (Ar-C), 122-127 (Ar-CH=CH-)
2.30.D_3	3309(N-H), 822 (C-N triazine), 1408 (-C=N), 3308 (Ar-OCH_3), 1594 (-CO-C=C-), 1651 (-CO-)	6.90 (d, 1H, –CO–CH=), 7.08-7.6 (M,12H,ArH), 7.8-8.3 (d,Ar-H,), 3.7-3.8 (S,9H,-OCH_3)	δ=189 (-CO), 145(C=N of s-tri.), 163(C-S-tri), 161(Ar-OCH_3), 55(-OCH_3), 113-119 (Ar-C), 122-127 (Ar-CH=CH-)
2.30.D_4	3308(N-H), 823 (C-N triazine), 1411 (-C=N), 3308 (Ar-OCH_3), 1596 (-CO-C=C-), 1653 (-CO-)	6.89 (d, 1H, –CO–CH=), 7.08-7.6 (M,12H,ArH), 7.8-8.3 (d,Ar-H,), 3.7-3.8 (S,9H,-OCH_3)	δ=187 (-CO), 145(C=N of s-tri.), 164(C-S-tri), 161(Ar-OCH_3), 54(-OCH_3), 113-119 (Ar-C), 122-127 (Ar-CH=CH-)
2.30.D_5	3307(N-H), 824 (C-N triazine), 1410 (-C=N), 3309 (Ar-OCH_3), 1594 (-CO-C=C-), 1652 (-CO-)	6.88 (d, 1H, –CO–CH=), 7.08-7.6 (M,12H,ArH), 7.8-8.3 (d,Ar-H,), 3.7-3.8 (S,9H,-OCH_3)	δ=188 (-CO), 146(C=N of s-tri.), 163(C-S-tri), 161(Ar-OCH_3), 55(-OCH_3), 113-119 (Ar-C), 122-127 (Ar-CH=CH-)

Mechanism of Formation of Compound



Mechanism of Tri-substituted Chalcone using Zinc phosphate MW method

Fig. 1:- Mechanism of Tri-substituted Chalcone using Zinc phosphate MW method

RESULT & DISCUSSION

The reaction of Compound 2.23(C_1-C_5) with p-methoxybenzaldehyde in presence of catal- ytic amount of zinc phosphate using MW method. We got excellent yield in terms of product 2.30 (D_1-D_5) and all the melting point, IR, NMR are exactly match with literature. Conventional method uses hazardous chemical, solvents, and reagent which can produce toxic waste and contaminants. This process may considerably contribute to environmental pollution and damage, So this method has variety of drawbacks as compare to that Green approach were used for synthesis of triazine derivative, further we proceeding with the microwave irradiation (Scheme-I) of compound 2.23 (C_1-C_5) with P-methoxybenzaldehyde in presence of zinc phosphate as a catalyst shows good result which gives compound 2.30 (D_1-D_5) compare to conventional approach. Here we used 220 Watt of power effectively for synthesis of chalcone derivative. As a result, we discovered an efficient green way to synthesizing novel derivative while reducing time using a microwave synthesizer. Zinc phosphate was utilized in microwave assisted process because it absorbs radiation from microwave readily.

Spectral analysis: - The S-triazine containing chalcone was confirmed by –C=O group at 1725cm^{-1} and 1627 cm^{-1}, also N-H stretching frequency was at 3333 cm^{-1}. The s-triazine contains C-N freq. shown at 822 cm^{-1}. The NMR value in ppm as ∂=7.5-7.9 ppm indicates the presence of chalcone group as –CO-C=CH- and Ar-CH=CH- group was confirmed. Also ∂ = 7.1-7.8 ppm indicate the presence of Aromatic protons. As a result, we will look at the proposed structure for compound 2.30 (D_1-D_5). All physical and spectroscopic properties were identical to compound published in the literature.

CONCLUSION

Here we synthesize different types of triazine derivative containing chalcone by using catalyst zinc phosphate with microwave. This is a proper green and efficient route for preparation of various chalcone derivatives. There is no study available utilizing zinc phosphate as a catalyst for preparation of 1-(4-(4,6-bis (4-substituted phenylamino)-1,3,5-triazin-2-ylamino) phenyl)-3-(4-substitutedphenyl) prop-2-en-1-one using ultrasound microwa ve method. This technique have various benefits over previous and standard reaction approaches, including ease of operation, a simpler work up procedure, increased yield, a shorter reaction time and environmentally favorable procedures with additionally MW procedures, all the product were synthesized in just a matter of minute, giving them a competitive advantage over the harsh traditional processes. Finally we can save significant amount of energy when we conduct out reaction utilizing an microwave approach instead of conventional (standard) approach.

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