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Microwave Assisted and Eco-friendly synthesis of pyridine based chalcone and its derivatives

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ABSTRACT-

Now in a current scenario green approach in organic synthesis covers a precious value over its traditional one. Green synthesis evolving in this decade got an appreciation from many researchers. This method for the synthesis of derivative of Prop-2en-1one from 2- acetyl pyridine and different aromatic aldehyde at room temperature by using PEG-400 solvent under microwave irradiation yields chalcone within short of time with excellent yield. This method is suitable alternative path for the green approach. In this method may consider as convenient route for synthesis of Chalcone derivative.

Keywords- 2-acetyl pyridene, PEG-400, microwave irradiation, chalcone

Introduction-

In the last decade Chalcone derivative got enormous value due to its pharmaceutical importance.It may find the application as precursor for the synthesis of many organic compounds. Chalcone is useful for its pharmacological activities. Many organic compounds such as flavonoid[1], isoflavonoid[2], pyrazole, pyrimidine etc are synthesized from chalcone derivatives. Chalcone find its importance in various biological applications comprising anti-inflammatory[3-4], antiviral[5],anti depressant[6] , anti-bacterial[7],anti-tumor[8], anticancer [9-12], antileishmanial[13] , immunomodulatory[14] , ant hyperglycemic [15],antiangeogenic activities[16]. In this study the synthesis formation of prop-2-en-1-one from 3-acetyl pyridine and different aromatic aldehyde in presence of strong alkali with PEG -400 as a green solvent under microwave irradiation shows extraordinary result with excellent yield of product. In the recent time microwave

irradiation was spontaneously getting importance in the field of organic synthesis. The process is simple and convenient and the use of polyethylene glycol -400 proved to be eco friendly. We attempt to synthesize Prop-2en-1-one derivative by using principles of green chemistry. The solvent taken PEG- 400 considered as environmental benign reaction solvent. It is easily available, water soluble, non toxic, cheap in cost from which separation of reaction product facilate.

In addition to their numerous biological activities, chalcones find a pronounced application in synthetic organic chemistry. Many heterocyclic compound are synthesized by using chalcone and as synthons for the synthesis of many pharmaceuticals . Having such a varied pharmacological activity and synthetic utility, chalcones have attracted chemists to develop a large number of synthetic methodologies for their synthesis around the world.

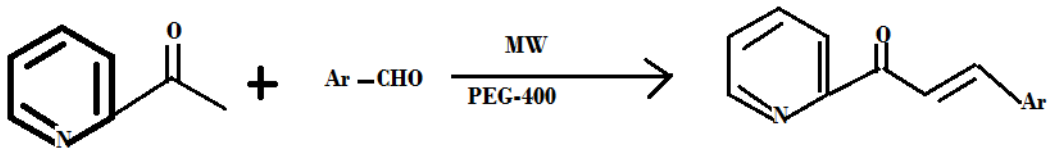
Material and Methods-

Experimental-

The analytical grade chemicals are purchased. The synthesized product is crystallized with ethyl alcohol and the purity of synthesized compound is checked by thin layer chromatography. The melting point of compound is taken in an open capillary and is uncorrected. Infrared spectra were recorded on Perkin Elsmeir spectrometer. The 1H NMR spectra were recorded on 400 MHz spectrometer with TMS as an internal standard. The purification of compound is done by recrystallisation in ethyl alcohol.

Procedure-

A mixture of 2-acetyl Pyridene (0.01 mole) and aromatic aldehyde (0.01 mole) were mixed well in 20 ml of PEG-400 and then Aq. 30% KOH was added drop wise with continuous stirring and shaken well. The reaction mixture was put under microwave irradiation for 2-3 minutes. The completion of reaction was checked by thin layer chromatography. After confirmation of completion of reaction, complete reaction mixture was poured into crushed ice and acidified with HCl if necessary in order to maintain PH. The solid precipitate obtained was filtered and recrystallised from ethanol. The characterization of synthesized organic compound is completed.



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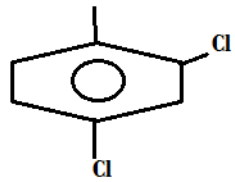

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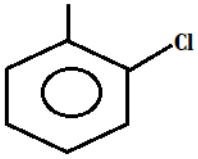

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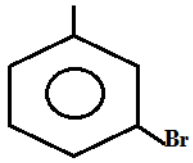

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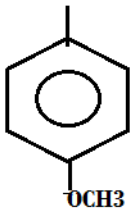

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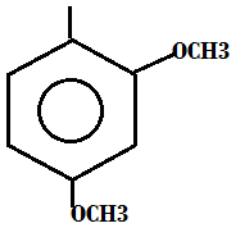

The Analysis of molecular formula, Melting Point, Percentage Yield is shown below

Sr. No	Molecular Formula	M.P	Percentage Yield
1	$C_{14}H_9NOCl_2$	118	82
2	$C_{14}H_{10}NOCl$	95	85
3	$C_{14}H_{10}NOBr$	106	80
4	$C_{15}H_{13}NO_2$	122	85
5	$C_{16}H_{15}NO_3$	128	78

IR –data (CM^{-1}) :-

1 - 1721 (C=O), 1645 (CH=CH), 855 (C-Cl), 1512 (C=N)

2- 1724 (C=O), 1648 (-CH=CH), 851 (C-Cl), 1536 (C=N)

3- 1730 (C=O), 1652 (-CH=CH), 860 (C-Br), 1530 (C=N)

4- 1732 (C=O), 1651 (-CH=CH), 1174 (-OCH3), 1584 (C=N)

5- 1735 (C=O), 1649 (-CH=CH), 1170 (-OCH3), 1412 (C=N)

1H NMR (in ppm)

Compound 1-

7.31 (1H, d, C-3' -H), 7.47 (1H, d, -CO-CH=), 7.57 (1H, d, C-6-H), 7.88 (1H, s, C-3-H), 7.92 (2H, m, C-4' -H, C-5' -H), 8.23 (1H, d, C-5-H), 8.32 (1H, d, =CH-Ar), 8.78 (1H, d, C-6' -H)

Compound 2-

7.40 (1H, d, -CO-CH=), 7.55 (1H, m, C-4' -H), 7.69 (1H, d, =CH-Ar), 7.89 (1H, d, C-3' -H), 7.96 (1H, m, C-5' -H), 8.22 (2H, d, C-2-H,C-6-H), 8.34 (2H, d, C- 3-H,C-5-H), 8.78 (1H, d, C-6' -H)

Compound 3-

7.21 (1H, d, -CO-CH=), 7.29 (1H, d, C-3' -H), 7.39 (1H, s, C-2-H), 7.60 (1H, d, C-4' -H), 7.68 (1H, m, C-5' -H), 7.85 (1H, d, C-6-H), 8.18 (1H, m, C-5-H), 8.29 (1H, d, =CH-Ar), 8.62 (1H, d, C-4-H), 8.77 (1H, d, C-6' -H)

Compound 4-

7.39(1H, d, J=16 Hz), 7.56(1H,d,J=16Hz), 7.86(1H,d,J=9Hz),7.69(2H,d), 7.58(1H,d,J=8Hz),7,23(2H,d), 7.19(1H,m)

Compound 5-

7.68(1H,d,J=16Hz), 7.84(1H,d,J=16 Hz), 7.20(1H.m), 7.87(1H,d, J=9 Hz), 7.44 (1H,m), 7.42(1H,d,J=9Hz), 7.40(2H,d), 7.66(2H,m)

Result and Conclusion-

The synthesis of chalcone from 2-acetyl pyridene with different aromatic aldehyde under microwave irradiation by using PEG-400 solvent emerges out to be an eco-friendly and a clean product is obtained during synthesis with good percentage of yield.

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