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Synthesis & Characterization of some Dihydropyrimidinone Derivatives by using Mango Juice as a Green Catalyst

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Abstract

Raw mango juice is used as a green catalyst for the Biginelli reaction. Substituted aldehyde 1 Ethyl acetoacetate 2 and urea 3 were stirred/refluxed using ethanol as a solvent and raw mango juice as a green catalyst. After completion of reaction the mixture was poured in cold water, extracted with ethyl acetate, evaporated to dryness and purified by crystallization using methanol to give corresponding dihydropyrimidinone 4 in higher yield.

Key Words: Raw mango juice, Biginelli, Dihydropyrimidinone, Green catalyst

INTRODUCTION

Dihydropyrimidinones are important heterocycles containing a pyrimidine moiety in the ring nucleus. In recent days, have aroused interest in medicinal chemistry due to its versatile biological activity. The main activities associated with this class of compounds are antitumor, antibacterial and calcium channel antagonism/inhibition[1].

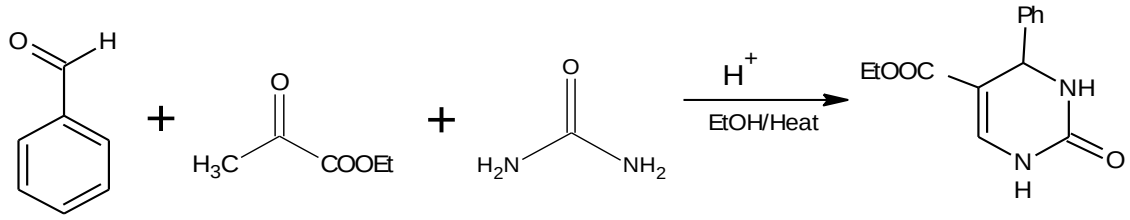
Dihydropyrimidinones are prepared by Biginelli reactions, have greater attention due to its important antimicrobial activities[2, 3]. The synthesis and utilization of these multi-disciplined moieties is in progress from the last few decades, which is clearly evident from the increasing number of publications and patents.

Although the most straightforward route to synthesize DHPMs is the one-pot acid-catalyzed Biginelli condensation , this protocol using ethanol and catalytic amounts of HCl often provides only low to moderate yields of the desired target molecules, in particular, when substituted aromatic aldehydes or thioureas are used [4].

In recent years, many greener routes are developed to carry out environmentally benign reactions in organic synthesis. The number of organic transformations is made by using fruit juice as a catalyst.[5] The mango water is also used as an efficient natural catalyst for the synthesis of amino Schiff bases.[6]

RESULTS AND DISCUSSION:

Equimolar amount of Aldehyde, ethyl acetoacetate and urea were taken in a 100 ml round bottom flask with ethanol as a solvent (10mL). Raw mango juice (1mL) was added to the reaction mixture. The reaction mixture was stirred / refluxed till completion of the reaction monitored by TLC. After completion of reaction the mixture was poured in cold water and extracted with ethyl acetate and evaporated to dryness. The crude product was purified by crystallization using methanol to give corresponding dihydropyrimidinone in higher yield

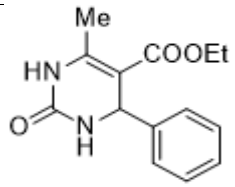
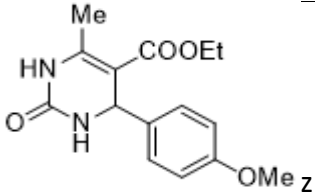


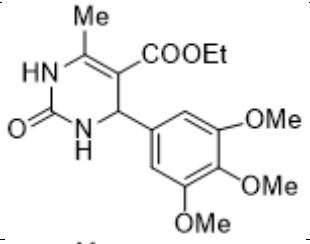
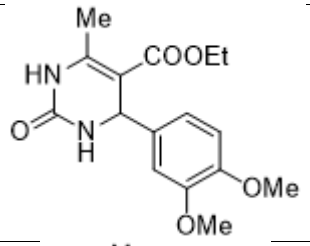
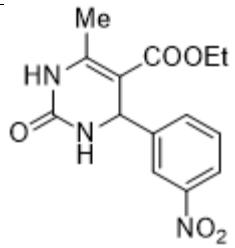
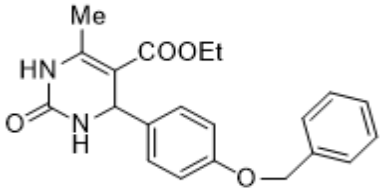
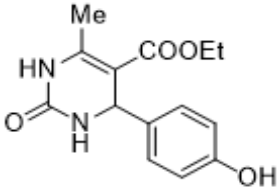
1 **2** **3** **4**

EXPERIMENTAL

Melting points were determined in open capillary tubes and are uncorrected. IR spectra were recorded on a Perkin-Elmer FT spectrophotometer in a KBr disc. ^{1}H NMR spectra were recorded on a Bruker Avance II 300 MHz spectrophotometer DMSO-d6 as a solvent and TMS as an internal standard (chemical shift in δ values). Mass spectra were obtained on a Finnigan mass spectrometer. Purity of the compounds was checked by TLC on silica gel G plates.

Table 1: Characterization data of synthesized compounds

Comp .	Product	Time (hr)		Yield (%)		MP (^{0}C)
		Stirring	Reflux			
		A	B	A	B	
4a		6	5	86	93	203
4b		6	4.5	91	97	202

4c		5	4	93	98	218
4d		5	4	94	98	198
4e		7	6	82	89	227
4f		5	3.5	88	96	240
4g		6	3.5	91	93	231

General Procedure

Aldehyde (1mmol), ethylacetoacetate (1mmol) and urea (1mmol) were taken in a 100 ml round bottom flask with ethanol as a solvent (10mL).

Raw mango juice (1mL) was added to the reaction mixture. The reaction mixture was stirred/refluxed till the completion of the reaction, monitored by TLC. After completion of reaction the mixture was poured in cold water and extracted with ethyl acetate and evaporated to dryness. The crude product was purified by crystallization using methanol to give corresponding dihydropyrimidinone in higher yield.

1a-Ethyl-6-methyl-2-oxo-4-phenyl-1,2,3,4-tetrahydropyrimidine-5-carboxylate.

IR (cm^{-1}): - 3236.39 (NH), 1723.20 (COOEt), 1699.02 (NHCONH).

^{1}H (ppm): - (DMSO, 300MHz), 1.075-1.123 (3H, t, -OCH_2**CH_3**), 2.264(3H, s, -CH_3), 3.952-4.023(2H, q, -O**CH_2**CH_3), 5.146-5.156 (1H, d, -CH), 7.230-7.254 (3H, d, -ArH), 7.307-7.330 (2H, d, -ArH), 7.747 (1H, s, -NH), 9.194 (1H, s, -NH).

1b-Ethyl 4-(4-methoxy phenyl)-6- methyl -2–oxo-1,2,3,4-tetrahydro- pyrimidine-5-carboxylate.

IR (cm^{-1}):- 3234.0-3105.58 (NH), 1720.28 (COOEt), 1702.36 (NHCO).

^{1}H (ppm):- (DMSO, 300MHz), 1.073-1.120 (3H, t, OCH_2**CH_3**), 2.233(3H, s, CH3), 3.712 (3H, s, O**CH_3**), 3.960-3.984 (2H, q, O**CH_2**CH_3), 5.078-5.088 (1H, d, -CH), 6.85-6.883 (2H, d, -ArH), 7.124-7.153 (2H, d, -ArH), 7.669(1H, s, -NH), 9.151(1H, s, -NH).

1c-Ethyl 6-methyl-2-oxo-4- (3,4,5 -trimethoxyphenyl)-1,2,3,4 –tetra hydro pyrimidine -5- carboxylate.

IR (cm^{-1}):- 3227.39-3097.70 (NH), 1721.90 (COOEt), 1706.08 (NHCO).

^{1}H (ppm):- (DMSO, 300MHz), 1.177-1.224 (3H, t, OCH_2**CH_3**), 2.356 (3H, s, CH_3), 3.828 (9H, s, O**CH_3*3**), 4.100-4.146 (2H, q, O**CH_2**CH_3), 5.374-5.700 (1H, d, -CH), 6.534-6.883 (2H, d, -ArH), 7.260 (1H, s, -NH), 7.954 (1H, s, -NH)

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