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CITRUS LEMON JUICE MEDIATED A COST EFFECTIVE ONE POT EFFICIENT SYNTHESIS OF 1, 4 DIHYDROPYRIDINES

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Abstract: Citrus lemon juice is found to be an efficient natural organo catalyst for the one pot three component synthesis of 1, 4 dihydropyridines using aromatic aldehydes, ethyl acetoacetate and ammonium acetate at 80°C under solvent-free condition. The crucial features of this protocol are use of non-toxic and readily available natural catalyst, good to excellent yield, green synthesis, solvent-free condition and shorter reaction time with no by-products.

Keywords: 1, 4 dihydropyridines, citrus lemon juice, green synthesis, natural catalyst, solvent-free condition.

Introduction

Nowadays, the pollution related issues have become a major concern. In this context the green approach for the synthesis of organic molecules of biological significance has gained importance. In multicomponent reaction (MCR) three or more reactants combine to form a product[i]. The product molecule is obtained in one step and one pot. The multicomponent reactions have advantages such as atom economy, selectivity, efficiency and no by-products[ii]. The organic Bronsted acids and amines were used as homogeneous catalyst in multicomponent reactions. The Lewis acid supported on solid materials is also used in multicomponent reactions[iii].

The naturally occurring catalyst becomes important due to features such as green, cheap, easily availability and safe. The fruit juice is employed in to execute the multicomponent organic reactions due to its catalytic properties[iv, v]. One of them is lemon juice which contains citric acid (5-7%), ascorbic acid (0.5%) and other organic acids having pH in acidic region[iv]. The lemon juice is useful in the treatment of arthritis and rheumatism, heart disease, cancer prevention, high blood pressure, prevents kidney stone, asthma and etc[iv]. The citrus lemon juice has properties such as antioxidant and antimicrobial, pharmacological, anti-inflammatory, anti-cancer, antiallergy, cardiovascular protection, neuroprotective and hepato protective[vi-ix]. Recently, the juice of citrus lemon is also used as a catalyst in different organic reaction[x-xiii].

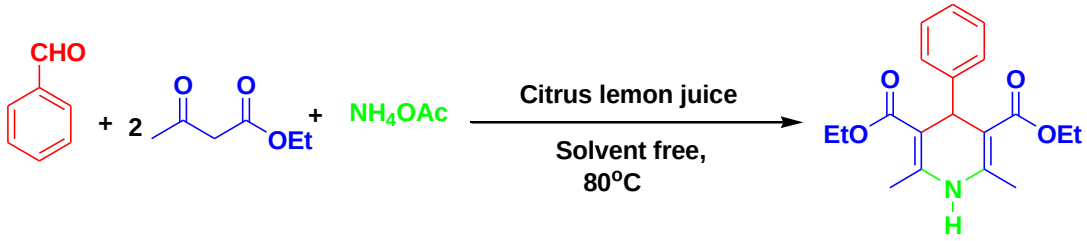
The dihydropyridine derivatives have medicinal properties such as antidiabetic, geroprotective, hepatoprotective, antitubercular agents, bronchodilator, antiatherosclerotic and anticancer[xiv-xx]. The dihydropyridines are also used in the treatment of tumour therapy and Alzheimer's disease[xxi, xxii]. 1, 4 Dihydropyridines is a source of drug molecule because it possess anti-aggregation and neuroprotective properties[xxiii-xxiv].1, 4 Dihydropyridines are also exhibit the activities such as anti-inflammatory[xxv], anticonvulsant[xxvi], antithrombotic[xxvii], analgesic[xxviii], antibacterial[xxix], antioxidant[xxix], antifertility agent[xxx], cystic fibrosis trans membrane conductance regulator activity[xxxi], vasodilation[xxxii], anti-leishmanial agents[xxxiii], stress protective effect[xxxiv], mineralocorticoid receptor antagonist activity[xxxv], cerebral anti-ischaemic agents[xxiv] and chemosensitizers[xxxvi]. The dihydropyridines are used in the treatment of hypertension[xxxvii], cardiovascular disorder[xxxviii], anti-HIV drugs[xxxix] and Rho-kinase inhibitors[xl].

Hantzsch first reported the synthesis of 1, 4 dihydropyridine[xli] but the yield was low. In literature it found that many catalytic systems have been used for the synthesis of 1, 4 dihydropyridines such as bismuth nitrate[xlii], silica-supported perchloric acid[xliii], iodobenzene diacetate[xliv], ZnO nanoparticles[xlv],tin dioxide nanoparticles[xlvi], chitosan[xlvii], Cu-doped ZnO nanocrystalline powder[xlviii], MgO nanoparticles[xlix], mesoporous vanadium ion doped titania nanoparticles[l], nano-crystalline solid acid catalyst[li], uranyl nitrate hexahydrate[lii], melamine trisulfonic acid[liii], graphene oxide nanoparticles[liv], tetrabutyl ammonium hexatungstate[lv], glycine nitrate[lvi], phosphosulfonic acid[lvii], aminated multiwalled carbon nanotubes[lviii], trichloroisocyanuric acid[lix], *p*-toluenesulfonic acid[lx], poly (vinylimidazolum acetic acid)-entrapped nanozeolite[lxi] and Fe_2O_3/ZrO_2[lxii]. All these methods find some important features but at the same time suffer from one or more limitations such as long reaction time, toxic reagents and low yields. In continuation to our research[lxiii-lxviii] to develop green protocols, herein we wish to report application of naturally available citrus lemon juice as a non-toxic and low-cost catalyst towards the synthesis of 1, 4 dihydropyridine derivatives at 80°C under solvent-free condition.

Results and discussion

In order to optimize the suitable condition a model reaction of benzaldehyde, ethyl acetoacetate and ammonium acetate with citrus lemon juice was chosen. In our primary study the model reaction was carried out using solvents like methyl alcohol, ethyl alcohol, acetonitrile, dichloromethane, toluene and without solvent in order to optimize the effect of solvent medium (Table 1). The various solvent afforded the reaction in lower yields (57-79 %). The results obtained suggest that the reaction occurs smoothly under solvent-free condition. The time required to complete the reaction was less and the yields of the product was also more under solvent-free condition.

Next, we carried out model reaction at different temperature at solvent-free condition (Table 2). The amount of product increases with rise in temperature up to 80°C. Beyond these no pronounced rise in yield of the product was observed. The results shows that 80°C is the optimum temperature for the reaction which requires less time and gives more yield of the product.



Scheme 2: Model reaction

Table 1: Effect of solvent on reaction

Entry	Solvent	Time (min)	Temp (°C)	Yields[a] (%)
1	Methyl alcohol	30	80	73
2	Ethyl alcohol	26	80	79
3	Acetonitrile	37	80	75
4	Dichloromethane	28	80	73
5	Toluene	34	80	57
6	Solvent free	25	80	93

[a]Isolated yields

Table 2: Effect of temperature on reaction

Entry	Temp (°C)	Time (min)	Yields[a] (%)
1	50	35	54
2	60	34	62
3	70	30	78
4	80	25	93
5	90	25	93
6	100	25	91
7	110	25	90

[a]Isolated yields

A blank reaction of benzaldehyde, ethyl acetoacetate and ammonium acetate without catalyst was carried out at 80°C under solvent-free condition. After 12h no product was observed in reaction mixture. The model reaction was studied with 0.010, 0.020, 0.030, 0.040, 0.050, 0.060 and 0.070 g of citrus lemon juice to optimize the amount of catalyst required (Table 3). We observed that reaction of benzaldehyde, ethyl acetoacetate and ammonium acetate with 0.050 g of citrus lemon juice gives 93 % yield of the product. We also observed that further increase in the amount of citrus lemon juice to 0.070 g there was no significant improvement in the yield of the desired product.

These results increased our interest to check the scope, generality and application of this protocol for the synthesis of 1, 4 dihydropyridine derivatives. The series of 1, 4 dihydropyridines were synthesized using different aldehydes under above optimized conditions. The reaction was monitored by thin layer chromatographic technique and it indicates that the reaction completed in short time (9-30 min) with good to excellent yield of products (82-97 %). Both electron deficient and electron rich aromatic aldehydes worked very well. We also observed that aldehydes with electron with-drawing groups give high yield as compared to aldehydes with electron-donating groups (Table 4).

To check the merits of citrus lemon juice catalysed synthesis of 1, 4 dihydropyridine derivatives it was compared with literature reported protocols (Table 5). It was observed that application

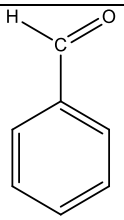
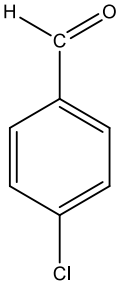
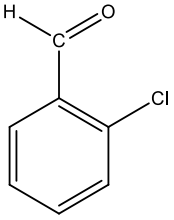
of naturally occurring, low cost of lemon juice good to excellent yields in short time are some features of this protocol. The merits of citrus lemon juice catalysed synthesis of 1, 4 dihydropyridine derivatives over the reported catalytic system. In most of the cases comparative yields of the expected product were obtained but the reported procedure required long reaction time (except Entry 7, 12 and 13). Most of the reactions worked out in presence of solvents (except Entry 7 and 13). These results reveal that citrus lemon juice is more efficient biodegradable catalyst for the synthesis of 1, 4 dihydropyridines.

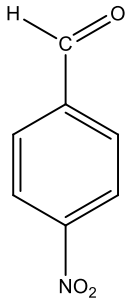
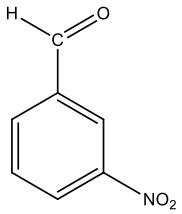
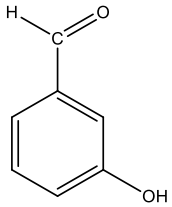
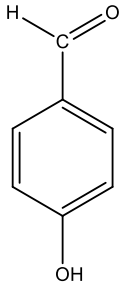
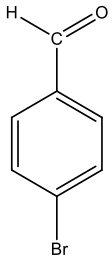
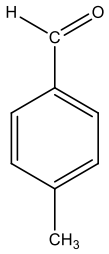
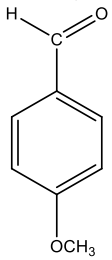
Table 3: Effect of catalyst amount

Sr. No.	Amount (g)	Time (min)	Yields[a] (%)
1	0.010	50	58
2	0.020	41	62
3	0.030	35	67
4	0.040	29	72
5	0.050	25	93
6	0.060	25	93
7	0.070	25	93

[a]Isolated yields

Table 4: Synthesis of 1, 4 dihydropyridines with citrus lemon juice

Entry	Reactant	Product	Time (min)	Yields[a] (%)	M. P (ºC) (M. P. lit) [Reference]
1		**4a**	25	93	155 (154-156)[lx]
2		**4b**	26	82	150 (149-151)[lx]
3		**4c**	14	84	127 (126-127)[lxii]

4		**4d**	9	97	125 (124-126)[lx]
5		**4e**	17	90	164 (163-165)[lx]
6		**4f**	30	83	180 (178-179)[liii]
7		**4g**	26	90	229 (228-230)[lx]
8		**4h**	27	86	162 (162-163)[xxii]
9		**4i**	30	95	132 (131-133)[lx]
10		**4j**	24	94	154 (154-156)[lx]

[a]Isolated yields

Table 5: Comparison of catalyst to synthesize 1, 4-dihydropyridine derivatives

Entry	Catalyst	Condition	Time	Yields[a, b] (%) [References]
1	Triphenylphosphine	EtOH/reflux	5 h	72[xxii]
2	Nano-crystalline sulphated zirconia	EtOH/reflux	40 min	92[liii]
3	Nano ZnO	EtOH/reflux	45 min	79[liii]
4	Nano-γ-alumina	EtOH/reflux	40 min	80[liii]
5	Nano-ZSM-5 zeolite	EtOH/reflux	45 min	85[liii]
6	Aminated multiwalled carbon nanotubes	EtOH/reflux	4 h	85[lx]
7	*p*-toluenesulfonic acid	Reflux	15 min	90[lxii]
8	Trichloroisocyanuric acid	EtOH-H_2O/reflux	55 min	92[lxi]
9	Cu doped ZnO nanocrystalline powder	H_2O/reflux	1.5 h	97[l]
10	MgO nanoparticles	EtOH/reflux	110 min	85[li]
11	Uranyl nitrate	Sonication	15 min	96[liv]
12	Tetrabutylammonium hexatungstate	reflux in oil bath/110^oC	20 min	93[lvii]
13	Phosphosulfonic acid	Reflux	15 min	98[lix]
14	Trichloroisocyanuric acid	Water-ethanol/reflux	55 min	92[lxi]
15	Graphene oxide nanoparticles	EtOH/reflux	3 h	92[lvi]
16	Citrus lemon juice	Reflux	25 min	93 [Present method]

[a]Isolated yields

[b]Reaction with benzaldehyde, ethyl acetoacetate, ammonium acetate and citrus lemon juice heated at 80^oC.

Experimental

All chemicals required were procured commercially and used without purification. The melting points were noted by an open capillary method. The products obtained were matched with known compounds using their spectral data. The IR spectra were recorded on Perkin-Elmer FT-IR spectrophotometer using KBr pellets. The ^1H NMR were recorded on Bruker Avance II (400 MHz) using DMSO as the solvent and TMS as an internal standard. Mass spectra were determined on a Varian-Saturn GC/MS instrument.

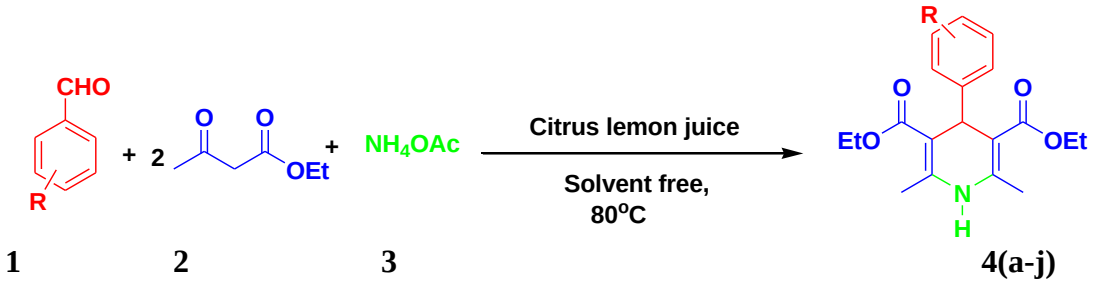
2.1. Preparation of juice from citrus lemon

The citrus lemon fruits were collected from garden and were washed by using distilled water and dried at room temperature. The juice was extracted mechanically and centrifuged. The separated juice was used as catalyst in organic reactions.

2.2. General procedure for synthesis of 1, 4 dihydropyridines

A mixture of aromatic aldehyde (1 mmol), ethyl acetoacetate (2 mmol), ammonium acetate (1 mmol) and citrus lemon juice (0.050 g) were taken in a round-bottomed flask and was heated at 80^oC in an oil bath under solvent-free condition. The formation of product during the reaction was checked by thin layer chromatography. The solid products achieved were cooled at room temperature and hot ethanol was added to it. The hot solution was filtered to remove any

insoluble impurity, the filtrate was allowed to attain room temperature. The solid product was filtered and dried to afford pure 1, 4 dihydropyridine derivatives (**Scheme 1**).



1 **2** **3** **4(a-j)**

Scheme 1: Synthesis of 1, 4 dihydropyridines

Selected spectral data:

Compound (4a):IR (KBr) (ν_{max}, cm^{-1}): 3324, 2978, 1677, 1127, 1024. ^{1}H NMR (400 MHz, DMSO-d_6) δ_H: 1.15 (t, 6H), 2.24 (s, 1H), 2.34 (s, 6H), 4.10 (q, 4H), 5.49 (s, 1H), 7.22-7.37 (m, 5H). MS (m/z): 329.16 (M^+).

Compound (4b):IR (KBr) (ν_{max}, cm^{-1}): 3321, 2979, 1688, 1645, 1120, 1024, 756. ^{1}H NMR (400 MHz, DMSO-d_6) δ_H: 1.19 (t, 6H), 2.23 (s, 1H), 2.33 (s, 6H), 4.12 (q, 4H), 5.52 (s, 1H), 7.17-7.29 (m, 4H). MS (m/z): 363.12 (M^+).

Compound (4d):IR (KBr) (ν_{max}, cm^{-1}): 3424, 1712, 1637, 1435, 1139, 801. ^{1}H NMR (400 MHz, DMSO-d_6) δ_H: 1.17 (t, 6H), 2.25 (s, 1H), 2.32 (s, 6H), 4.08 (q, 4H), 5.57 (s, 1H), 8.09-7.37 (m, 4H). MS (m/z): 374.15 (M^+).

Compound (4j):IR (KBr) (ν_{max}, cm^{-1}): 3362, 2966, 1664, 1123, 1022. ^{1}H NMR (400 MHz, DMSO-d_6) δ_H: 1.15 (t, 6H), 2.23 (s, 1H), 2.29 (s, 6H), 3.65 (s, 3H), 4.08 (q, 4H), 5.67 (s, 1H), 6.76-7.17 (m, 4H). MS (m/z): 395.17 (M^+).

Conclusion

We have established a green method for the synthesis of 1, 4 dihydropyridine derivatives by condensation of aromatic aldehyde, ethyl acetoacetate and ammonium acetate using natural organocatalyst such as biodegradable citrus lemon juice under solvent-free condition. The use of naturally found green and non-toxic citrus lemon juice is the advantage of present method. The high yield, easy work-up, cost effective and solvent-free reaction are the significant features of this method.

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