

Solvent-free , One pot synthesis of Dihydropyrano [3,2-c] chromene derivatives with β -Alanine as a green catalyst

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Abstract

We reported an productive and green procedure for one pot, three-component condensation reaction of 4-hydroxycoumarine, malononitrile and appropriate aldehydes using β -alanine as a catalyst under solvent free conditions at pH 6 ,gave dihydropyrano[3,2-c] chromenes derivatives at 65⁰C. Here new compounds are synthesized and the result were compared by FT-IR, ¹H NMR, ¹³NMR as well as Mass spectra .Short reaction time, cost effective ,clean reaction profile, ,simple isolation, excellent yield and environment benign are the main advantages of this procedure.

Keywords: *Multi-component reaction; Green procedure; Solvent-free conditions; dihydropyrano[3,2-c]chromenes derivatives; Knoevenagel condensation*

1. Introduction

In modern synthetic chemistry, multi-component reactions (MCRs) have been one of the most important approaches for the preparation of highly functionalized organic compounds. Recently, multi-component reactions strategies offer significant advantages as a result they facilitate more step and time saving conversions of simple starting materials into complex targets. Therefore, they have all the characteristics that furnish to an ideal synthesis: high atom efficiency, quick and simple implementation, time and energy saving, environment-friendly, cost-effective and they offer a target and diversity-oriented synthesis.¹ In the last few decades , chemists have been aware of the environmental effect of their chemistry. Green Chemistry or environmentally

benign chemistry is the form of chemical consequence and methods that avoid the use and generation of precarious substances.² Now a days, to stop the use and production of unsafe substances in the designing, making and use of chemical products, they are trying to develop new synthetic methods which solve environmental problems involves the designing and re-designing of chemical creation and chemical products.

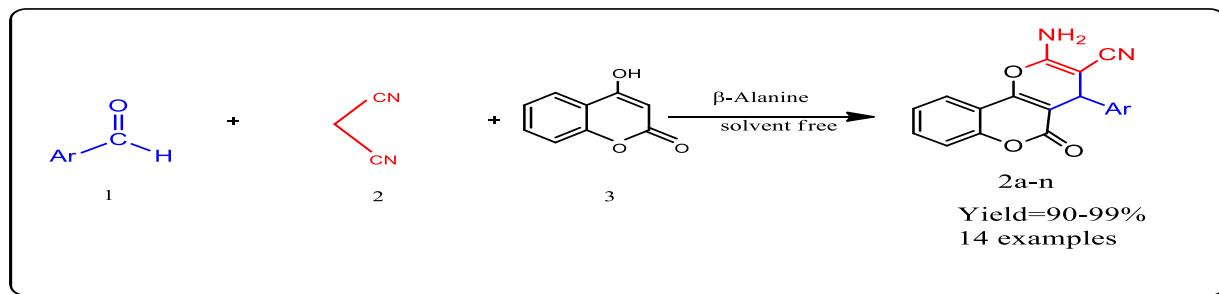
Among the heterocyclic compounds, chromene derivatives are the main and important components of numerous chemical free products. Dihydropyrano[3,2-*c*]chromenes and their derivatives manifest various interesting biological properties such as anti-coagulant,^{3,4} anticancer,⁵ anti-HIV,^{6,7} antitumor,⁸ antimicrobial,⁹ antibacterial, and antiamebic^{10,11} activities.

The synthesis of dihydropyrano[3,2-*c*] chromenes derivatives by a three component condensation reaction of 4-hydroxycoumarine, aldehydes and malononitrile has been performed under various conditions by using different catalyst Such as DABCO,¹² Mg(ClO₄)₂,¹³ CH₃OONH₄,¹⁴ DBU,¹⁵ DAHP,¹⁶ Na₂HPO₄,¹⁷ K₂CO₃,¹⁸ MgO,¹⁹ (S)-proline,²⁰ TBAB,²¹ HPAA,²² [bmim]Br,²³ potassium phthalimide-N-oxyl,²⁴ ammonium acetate,²⁵ Fe₃O₄@GO-naphthalene-SO₃H nanocatalyst,²⁶ Fe₃O₄@SiO₂-Poly acrylic acid nanocatalyst,²⁷ MNPs@Cu nanocatalyst²⁸ and ionic liquid^{29,30}. While these methods have achieved good results, they are limited by high cost, high reaction temperatures, low yields, use of toxic solvents and the requirement of special apparatus.

So in continuation of work on the synthesis of heterocyclics ,herein we report synthesis of dihydropyrano[3,2-*c*]chromenes derivatives beta-alanine as an efficient eco-friendly, non-toxic catalyst. In our body , Beta-alanine is a non-essential amino acid which is produced naturally. It also has an antioxidant, anti-aging and immune-enhancing properties.

2. Experimental Section

All the chemicals were purchased from Sigma Aldrich, CDH and Loba and were used without further purification. All reactions were monitored by TLC, using benzene as a solvent. Spots were visualized in iodine chamber. Melting points were determined in an open capillary using a Pinsky Martin flash point apparatus and are uncorrected .IR spectra were recorded on a FTIR spectrophotometer in wave number (cm⁻¹). ¹H NMR and ¹³C NMR spectra were obtained on a BRUKER AVANCE NEO (at 500 MHz) using DMSO as a solvent at SAIF, Chandigarh.



Scheme 1 The reaction of 4-hydroxycoumarin, malononitrile and aldehydes in the presence of β -Alanine under solvent free conditions at 65°C.

2.1 General procedure for the preparation of compounds 2a-n

In a 50 ml round bottomed flask, beta-alanine (16%) was added to a mixture of aromatic aldehydes (1mmol), malononitrile (1mmol), and 4-hydroxycoumarin (1mmol), then the reaction mixture was stirred at 65°C for the appropriate time at pH 6, as summarized in Table-1. To monitor the progress of the reaction TLC was used. After the completion of the reaction, the mixture was allowed to cool at room temperature. The product obtained was filtered, washed with water and then was recrystallized with ethanol.

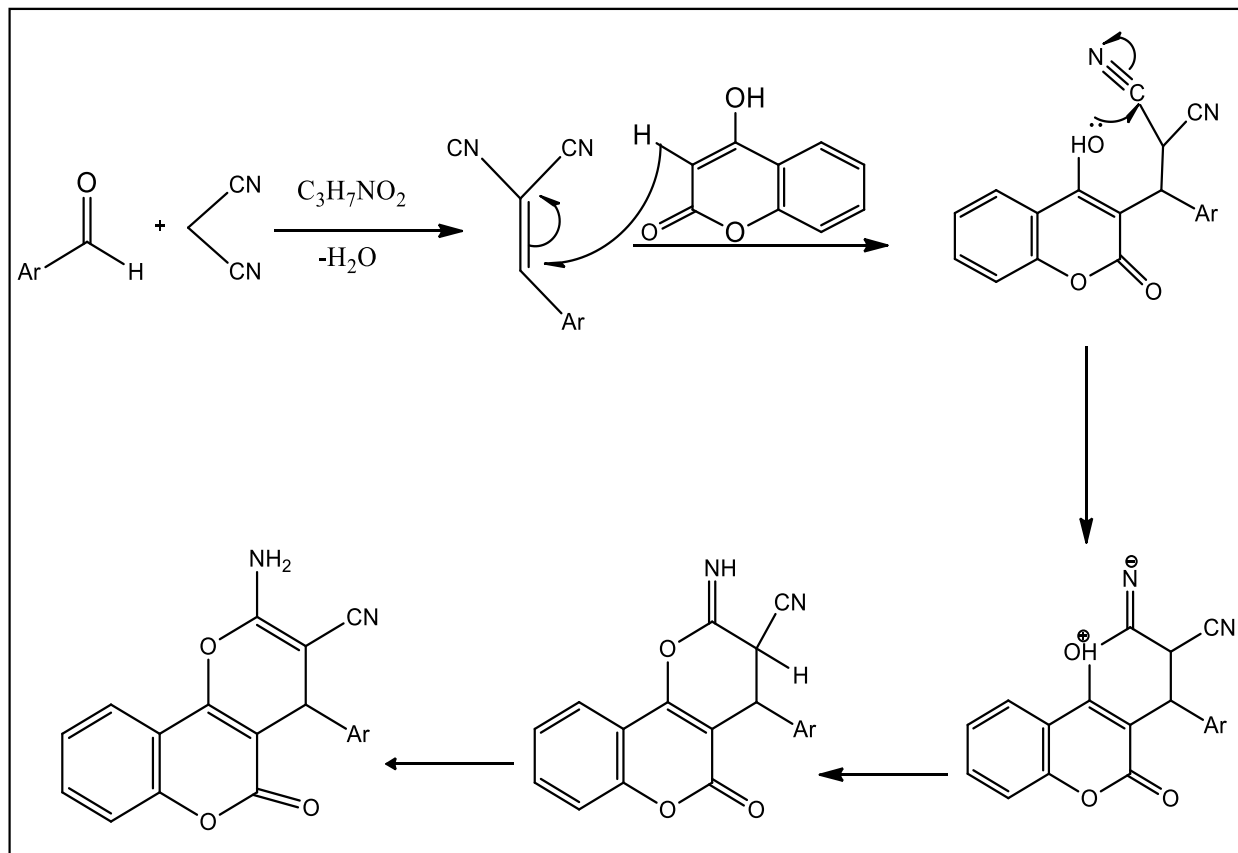
Table 1. Preparation of substituted dihydropyrano[3,2-c]chromenes derivatives.

Entry	Aldehyde	Time (min)	Yield (%)	Product	M.p.(°C)/ [Ref.]
1.	4-chlorobenzaldehyde	5	99	2a	263-264 ¹²
2.	2-chlorobenzaldehyde	7	98	2b	262-264 ²⁶
3.	4-hydroxybenzaldehyde	4	95	2c	262-263 ²⁶
4.	4-nitrobenzaldehyde	7	98	2d	260-261 ¹²
5.	4-methoxybenzaldehyde	8	96	2e	224-225 ²⁸
6.	4-bromobenzaldehyde	6	96	2f	247-250 ¹²
7.	3-nitrobenzaldehyde	6	95	2g	260-261 ²⁶
8.	3-pyridine carboxaldehyde	6	90	2h	259-253 ¹³
9.	3,4-dimethoxybenzaldehyde	7	98	2i	222-225 ³⁰
10.	4-fluorobenzaldehyde	5	97	2j	259-260 ²⁹
11.	4-fluoro-3-nitrobenzaldehyde	7	95	2k	236-238 [New Compound]
12.	Indole-3-carboxaldehyde	6	96	2l	205-206 [New Compound]
13.	4-butoxy,3-nitrobenzaldehyde	5	97	2m	238-240 [New Compound]
14.	2-bromo,4-methylbenzaldehyde	7	96	2n	240-241 [New Compound]

^a Isolated yield

^b New compounds synthesized in this work

The possible reaction mechanism of the reaction is shown in the scheme 2. In the first step of this reaction, Knoevenagel product is formed by the condensation of the aldehyde with malononitrile in the presence of β -Alanine, a green catalyst. The unsaturated nitrile then undergoes subsequent reaction with 4-hydroxycoumarin in the presence of β -alanine to give the desired product.



Scheme 2

Scheme -2 Suggested mechanism for the synthesis of dihydropyrano [3,2-c] chromene derivatives based on Knoevenagel condensation reaction

2.2 Spectral data for the products are listed below:

2.2.1 Compound (2a): 2-Amino-4-(4-chlorophenyl)-5-oxo-4H,5H-pyrano-[3,2-c]chromene-3-carbonitrile : IR (KBr, cm^{-1}): 3379 and 3310 (NH_2), 3258 (Ar-H), 2193 (CN), 1674 (C=O), 1639; ^1H NMR (500 MHz, $\text{DMSO}-d_6$) δ 4.49 (s, 1H, -CH), 6.32 (s, 2H, $-\text{NH}_2$), 7.17 (d, 1H, $J = 8.0$ Hz), 7.24-7.37 (m, 1H), 7.43-7.49 (m, 1H), 7.56 (t, 1H, $J = 7.5$), 7.69 (d, 1H, $J = 8.5$), 7.89 (t, 1H, $J = 7$), 7.93 (d, 1H, $J = 8.5$), 8.52 (d, 1H, $J = 7.3$)

2.2.2 Compound (2b) : 2-Amino-4-(ortho-chlorophenyl)-5-oxo-4H,5H-pyrano-[3,2-c]chromene-3-carbonitrile: IR (KBr, cm^{-1}): 3380 and 3282 (NH_2), 2199 (CN), 1672 (C=O), 1599; ^1H NMR (500 MHz, $\text{DMSO}-d_6$) δ : 4.98 (s, 1H, -CH), 7.25-7.29 (m, 2H, and $-\text{NH}_2$), 7.17 (d, 1H, $J = 8.0$ Hz), 7.24-7.37 (m, 1H), 7.43-7.49 (m, 1H), 7.56 (t, 1H, $J = 7.5$ Hz), 7.69 (d, 1H, $J = 8.5$ Hz), 7.89 (t, 1H, $J = 7$), 7.93 (d, 1H, $J = 8.5$ Hz), 8.52 (d, 1H, $J = 7.3$ Hz)

2.2.3 Compound (2c) : 2-Amino-4-(4-hydroxyphenyl)-5-oxo-4H,5H-pyrano-[3,2-c]chromene-3-carbonitrile: IR (KBr, cm^{-1}): 3561 (OH), 3382 and 3356 (NH_2), 2197 (CN), 1672 (C=O), 1602; ^1H NMR (500 MHz, $\text{DMSO}-d_6$) δ : 4.33 (s, 1H, -CH), 6.71-6.72 (m, 2H, $-\text{NH}_2$), 6.96 (d, 1H, $J = 8.5$ Hz), 7.06 (d, 1H, $J = 8.5$ Hz), 7.32-7.45 (m, 3H), 7.6 (t, 1H, $J = 7.5$ Hz), 7.87-8.240 (m, 3H)

2.2.4 Compound (2d): 2-Amino-4-(4-nitro phenyl)-5-oxo-4H,5H-pyrano-[3,2-c]chromene-3-carbonitrile : IR (KBr, cm^{-1}): 3382 and 3326 (NH_2), 2195(CN), 1670 (C=O), 1605; ^1H NMR (500 MHz, $\text{DMSO-}d_6$) δ : 4.20(s,1H, -CH), 6.9 (s,2H,- NH_2),7.06-7.19 (m,3H), 7.33-7.36 (m, 2H),7.7 (d, 1H, J=8.5), 7.97 -8.55(m, 2H,)

2.2.5 Compound (2e): 2-Amino-4-(4-methoxyphenyl)-5-oxo-4H,5H-pyrano-[3,2-c]chromene-3-carbonitrile: IR (KBr, cm^{-1}): 3389 and 3324 (NH_2), 2156(CN), 1668 (C=O), 1602; ^1H NMR (500 MHz, $\text{DMSO-}d_6$) δ : 3.71 (s,3H,- CH_3), 4.37(s,1H,-CH), 6.37(s,2H,- NH_2),6.83(d,1H, J=7.5), 7.104-7.59 (m, 4H),7.82-8.28(m, 3H,)

2.2.6 Compound (2f): 2-Amino-4-(4-bromophenyl)-5-oxo-4H,5H-pyrano-[3,2-c]chromene-3-carbonitrile :IR (KBr, cm^{-1}): 3380 and 3290 (NH_2), 2198(CN), 1675 (C=O), 1610; ^1H NMR (500 MHz, $\text{DMSO-}d_6$) δ : 4.47(s,1H, CH),6.26 (s,2H, - NH_2), 7.07(d, 1H, J=7 Hz), 7.26 (t, 1H, J=8 Hz), 7.34(t,1H, J=8Hz), 7.43-7.50 (m,1H), 7.70(t,1H, J=7.5), 7.80-8.51(m, 3H)

2.2.7 Compound (2g) : 2-Amino-4-(3-nitrophenyl)-5-oxo-4H,5H-pyrano-[3,2-c]chromene-3-carbonitrile: IR (KBr, cm^{-1}): 3387 and 3318 (NH_2), 2202(CN), 1669 (C=O),1604 ; ^1H NMR (500 MHz, $\text{DMSO-}d_6$) δ : 4.72 (s,1H, CH), 7.42(s,2H,- NH_2), 7.43-7.56 (m,3H), 7.62 (t, 1H, J=8Hz) , 7.65(t, 1H, J=8 Hz),7.81 (d, 1H, J=8Hz) , 8.10-8.15(m, 2H)

2.2.8 Compound (2h): 2-Amino-4-(2-formylpyridine)-5-oxo-4H,5H-pyrano-[3,2-c]chromene-3-carbonitril :IR (KBr, cm^{-1}): 3363 and 3173 (NH_2), 2201(CN), 1670 (C=O), 1603; ^1H NMR (500 MHz, $\text{DMSO-}d_6$) δ : 4.56 (s,1H, -CH), 7.34-7.35(m,2H,- NH_2), 7.40-7.77(m,4H), 7.89(d, 1H, J=8 Hz),8.30(t, 1H, J=8), 8.46-8.95 (m, 3H)

2.2.9 Compound (2i): 2-Amino-4-(3,4-dimethoxyphenyl)-5-oxo-4H,5H-pyrano-[3,2-c]chromene-3-carbonitril : IR (KBr, cm^{-1}): 3323 and 3293 (NH_2), 2196(CN), 1663 (C=O), 1609; ^1H NMR (500 MHz, $\text{DMSO-}d_6$) δ : 3.79 (s,3H, - OCH_3), 3.89 (s,3H,- OCH_3), 4.40(s,1H,-CH), 6.27 (s,2H, - NH_2), 6.68-6.80 (m,1H), 6.87(t, 1H, J=8.5 Hz), 7.2(d, 1H, J= 8.5 Hz), 7.27-7.35(m,1H), 7.54-7.62 (m,1H), 7.72 (t, 1H, J=8), 7.8 (t, 1H, J= 7.5)

2.2.10 Compound (2j): 2-Amino-4-(4-fluorophenyl)-5-oxo-4H,5H-pyrano-[3,2-c]chromene-3-carbonitril: IR (KBr, cm^{-1}): 3337 and 3190 (NH_2), 2190(CN), 1676 (C=O), 1602; ^1H NMR (500 MHz, $\text{DMSO-}d_6$) δ : 4.47(s,1H,-CH),7.09-7.13 (m,2H, - NH_2), 7.31 (t,1H, J=7.5Hz), 7.38-7.46(m, 3H) , 7.67(t, 1H, J= 7.5 Hz), 7.8(d, 1H,J=8Hz) ,8.01 (m, 1H), 8.49 (d,1H,J= 7)

2.2.11 Compound (2k): 2-Amino-4-(4-fluoro,3-nitrophenyl)-5-oxo-4H,5H-pyrano-[3,2-c]chromene-3-carbonitrile: IR (KBr, cm^{-1}): 3323 and 3189 (NH_2), 2210(CN), 1676 (C=O),1600 ; ^1H NMR (500 MHz, $\text{DMSO-}d_6$) δ : 4.69 (s,1H, CH), 7.41-7.43(s,2H,- NH_2), 7.4 (t,1H, J=7.5Hz) , 7.52-7.55 (m, 2H), 7.73(t, 1H, J=7.5 Hz),7.81 (t, 1H, J=7.5 Hz) , 7.90 (d, 1H, J=8 Hz) ,8.07-8.09 (m,1H,); ^{13}C NMR (500 MHz, $\text{DMSO-}d_6$) : 57.26, 103.09, 113.42, 116.98, 118.79,118.95, 123.01, 125.01, 125.60, 133.50, 136.30, 137.33, 141.20, 141.23,152.73, 154.37, 155.08, 158.63, 160.06; MS: m/z =380.41(M^+), 403.34(M^+ + Na), 239.48 [M^+ - $\text{C}_6\text{H}_3\text{O}_2\text{NF}$], 151.47 , 194 etc.

2.2.12 Compound (2l) : 2-Amino-4-(Indole-3-carboxyaldehyde)-5-oxo-4H,5H-pyrano-[3,2-c]chromene-3-carbonitrile: IR (KBr, cm^{-1}): 3318 and 3190 (NH_2), 2110(CN), 1673 (C=O),1610 ; ^1H NMR (500 MHz, $\text{DMSO-}d_6$) δ : 4.73 (s,1H, CH), 5.62(s, 2H, CH_2), 6.93 (t,

1H, J= 7.5 Hz), 7.05(t, 1H, J=8Hz),7.25-7.38(m,2H), 7.45 (t,1H, J=8 Hz), 7.56-7.66 (m, 1H), 7.81 (d, 1H, J=7.5 Hz),7.94 (d, 1H, J=8 Hz) , 8.01 (d, 1H, J=8 Hz),8.51-8.63 (m,1H,) ; ¹³C NMR (500 MHz, DMSO-d₆) : 57.56, 110.48, 111.88, 113.38,116.97, 119.84, 123.01, 124.45, 125.06, 127.52, 129.99, 133.42, 139.25, 148.69, 149.61, 150.81,151.81, 152.68, 154.28, 158.59, 160.09; MS: m/z = 371.48 (M⁺),423.39 (M⁺ + Na), 241.20 [M⁺-C₉H₅N], 149.45 etc.

2.2.13 Compound (2m): 2-Amino-4-(4-butoxy-3-nitroaldehyde)-5-oxo-4H,5H-pyrano-[3,2-c]chromene-3-carbonitrile: IR (KBr, cm⁻¹): 3342 and 3187 (NH₂), 2186(CN), 1670 (C=O),1605 ; ¹H NMR (500 MHz, DMSO-d₆) δ : 0.89 (t,2H, CH₂ J=6 Hz), 1.38-1.42 (m, 2H, CH₂) , 1.66 (t, 3H, CH₃, J= 6 Hz),1.67-2.50 (m, 2H,-CH₂), 4.07 (s, 1H, CH), 6.44 (s, 1H), 7.02-7.12 (m, 2H), 7.20-7.34 (m,1H), 7.80 (d, 1H, J=8 Hz), 7.44 (d, 1H, J= 7Hz), 7.64-7.74 (m, 1H) , 7.83 (t, 1H, J= 7 Hz), 8.5 (d, 1H, J= 7 Hz)) ; ¹³C NMR (500 MHz, DMSO-d₆) : 52.74, 109.11, 115.47, 116.25, 118.75, 123.40, 125.94, 126.42, 132.44, 135.07, 139.43, 150.52, 153.79, 161.56 ; MS: m/z =434.47 (M⁺), 456.42 (M⁺ + Na), 239.46 [M⁺-C₁₀H₁₂O₃N], 241.49 etc.

2.2.14 Compound (2n): 2-Amino-4-(2-bromo,4-methylbenzaldehyde)-5-oxo-4H,5H-pyrano-[3,2-c]chromene-3-carbonitrile: IR (KBr, cm⁻¹): 3321 and 3121 (NH₂), 2165(CN), 1672 (C=O),1602 ; ¹H NMR (500 MHz, DMSO-d₆) δ : 2.50-2.54(m, 3H, -CH₃), 4.37 (s, 1H, CH), 6.44(s, 1H), 7.43-7.46 (m, 2H), 8.13-8.18 (m, 3H), 8.43 (t, 1H, J= 7Hz), 8.72 (s, 1H); ¹³C NMR (500 MHz, DMSO-d₆) : 57.72, 103.53, 113.54, 115.36, 117.04,119.52, 123.06, 124.30, 125.10, 133.45, 136.14, 139.90, 152.72, 154.13, 158.47, 160.07 ; MS: m/z =411.37 (M⁺), 434.30 (M⁺ + Na), 239..46 [M⁺-C₇H₆Br], 163.46 etc.

3. Results and Discussion

Firstly the reaction of 4- hydroxycoumarine(1mmol), malononitrile(1mmol), and benzaldehyde(1mmol) was investigated in the presence of different amounts of β-Alanine in 3ml of water at different temperature. The effect of different concentration (mol %) of β-Alanine such as 8,10, 12, 14, 16, 18 and 20 on the synthesis of dihydropyrano[3,2-c]chromenes derivatives in 3ml of water at 65 °C temperature give 67,73, 79, 82, 85, 80 ,85,78 and 80% yield(Table 2). After these, the effect of different concentration of β-Alanine under solvent free condition at 65°C temperature give good yield.

Table 2. Effect of concentration of catalyst on the reaction of 4-hydroxycoumarine, malononitrile, and benzaldehyde on the synthesis of dihydropyrano[3,2-c]chromenes derivatives under solvent free conditions.

Entry	β-Alanine (concn. mol%)	Temperature (°C)	Time (min)	Yield (%)
1.	8	65	18	67
2.	10	65	18	73
3.	12	65	16	79
4.	14	65	15	82
5.	16	65	12	85

6.	18	65	12	80
7.	20	65	11	80
8.	16	65	11	85
9.	16	60	11	78

The pH of the reaction mixture was made acidic or alkaline by adding few dilute acetic acid or sodium hydroxide. The pH of the solution was checked by pH indicator litmus test paper. Effect of different pH 4,5,6,7,8,9,and 10 on the synthesis of dihydropyrano[3,2-c]chromenes was studied (Table 3). The great results were obtained at pH 6 and using 16 mol% of β -Alanine at 65°C under solvent free conditions giving 99% yield (Table 3, **entry 5**).

Table 3. Effect of pH on the reaction of 4-hydroxycoumarin, malononitrile, and benzaldehyde on the synthesis of dihydropyrano[3,2-c]chromenes derivatives under solvent free conditions in the presence of 16% β -Alanine.

Entry	pH value	Temperature (°C)	Time(min)	Yield (%)
1.	pH- 10	65	5	62
2.	pH -9	65	4	76
3.	pH -8	65	6	86
4.	pH -7	65	12	85
5.	pH-6	65	6	99
6.	pH -5	65	8	96
7.	pH-4	65	10	90

When reaction get completed, the reaction mixture was washed with water and filtered off. Then obtained crude product obtained was recrystallized with ethanol. The optimized reaction conditions were then applied to different aldehydes . All the aldehydes underwent this three-component condensation reaction with malononitrile and 4-hydroxycoumarin to produce dihydropyrano[3,2-c] chromenes with excellent yield .A variety of aryl aldehydes , bearing electron withdrawing or electron donating groups participated well in this reaction and the products were achieved in good yields (99%-90%).

4. Conclusions

In conclusion, a rapid and environment friendly method for the synthesis of dihydropyrano[3,2-c]chromenes derivatives has been developed by using β -Alanine as a non-toxic, inexpensive ,safe and green catalyst under solvent free conditions. This method offers great advantages for example clean and mild reaction profile, high conversions, easy handling and a straightforward work-up which make it a beneficial and attractive process for the synthesis of these compounds.

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Biography



Sandeepika Rana received the M.Sc degree in Chemistry from the Lovely Professional University, Jalandhar, India in 2013. At present she is a Ph.D candidate in the Department of school of studies in Chemistry and Biochemistry. Her current research is concerned with heterocyclic compounds with their biological properties.



Aarti Nagar is a lecturer on College. She has done M.Sc degree in Chemistry from Vikram University. She is also a PhD candidate at the Vikram University .Her areas of interest and research are in the nano materials and heterocyclic compounds.



Poonam Singh has done M.Sc degree in Chemistry from Govt. & Science college Ratlam (M.P) in 2013. She received the M.Phil degree from Vikram University ,Ujjain. She is also a Ph.D candidate at the Vikram University. Her area of research on nano materials and heterocyclic compounds.



Dr. Shubha Jain is a Professor in Chemistry at the Vikram University , Ujjain (M.P) where she is the Head of the Department. She has pioneered research in organic

chemistry, heterocyclic compounds, photochemistry and bioactive compounds etc. and has published more than 50 research articles.

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