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# Aqueous extract of *Acacia Concinna* pods: an efficient, eco-friendly catalyst for the synthesis of 3-Acetyl/carboxylate quinolines at room temperature

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**ABSTRACT:** A simple, efficient and environmentally benign protocol for the synthesis of 3-acetyl quinolines has been achieved using aqueous extract of *Acacia Concinna* pods as a green surfactant type catalyst. We found for the first time, the aqueous extract of *Acacia Concinna* pods could be effectively catalyze the condensation of  $\alpha$ -aminoaryl ketone with active methylene compounds to yield 3-acetyl/carboxylate quinolines in excellent yields (90–98%), under mild conditions. The low cost, easy availability of the catalyst and simple reaction conditions suggest the possible use of present method for large scale preparations.

**KEYWORDS:** Quinoline, *Acacia* pods, green catalyst etc

## I. INTRODUCTION

New catalytic synthetic methods in organic chemistry that satisfy increasingly stringent environmental constraints are in great demand by the pharmaceutical and chemical industries. In addition, novel catalytic procedures are necessary to produce the emerging classes of organic compounds that are becoming the targets of molecular and biomedical research. Several types of substances such as enzymes, surfactants, ionic liquids, clays and supercritical solvents are now widely recognized as practical alternatives to traditional organic synthesis, and as convenient solutions to certain intractable synthetic problems. This is due to problems associated with prevailing catalysts like hazardous nature, expensive, difficult to handle, tedious work up, requirement of hazardous organic solvents and elevated temperature conditions, and above all, their adverse effects on environment. In an attempt to circumvent these disadvantages, we looked to nature for help. Nature provides a fantastic array of catalysts extremely well suited to supporting life such as, intact plant systems represent a unique class of potential biocatalysts for the reactions of various organic substrates.<sup>1</sup> The synthetic transformations using these materials are more efficient and generate less waste than the conventional chemical methods. More recently, the plant cell culture of *Daucus Carota* root,<sup>2</sup> soaked *Phaseolus aureus* (green grams),<sup>3</sup> coconut juice (*Cocos nucifera*)<sup>4</sup> were used as biocatalyst for selective reduction of ketones.

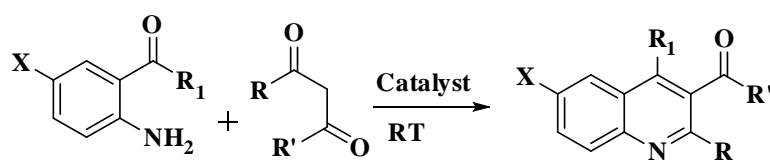
Quinolines are very well known natural products which exhibit a broad range of applications in the pharmaceutical, perfume and cosmetic industries.<sup>5</sup> Many synthetic Quinolines have shown interesting properties such as anticancer,<sup>6</sup> anti-inflammatory and antioxidant.<sup>8</sup> Many well known marketed drugs such as Warfarin, novobiocin, tiocloamarol contains Quinolines nucleus as a basic scaffold and recently, Quinolines were investigated as novel anti-proliferative agents (Fig. 1). They are also exploited as intermediates and building blocks in organic synthesis.<sup>9</sup> This has enthused us to search new and more convenient methods for the preparation of Quinolines derivatives.

*Acacia Concinna* is a medicinal plant (commonly known as Shikakai, Leguminosae family) grows in a tropical rainforests of southern Asia. The fruit is known as "fruit for hair" in its use as a traditional shampoo. Various properties of *Acacia Concinna* fruit are due to the presence of saponins in it, which are foaming agent. Saponins from the plant's pods have been traditionally used as a detergent. Specifically, they are amphipathic glycosides grouped

phenomenologically by the soap-like foaming they produce when shaken in aqueous solutions, and structurally by their composition of one or more hydrophilic glycoside moieties combined with a lipophilic triterpene derivative. The fruit is known to contain 10-11.5 % saponins. The structures of different saponins present in the fruit have been recently established.<sup>16</sup> These saponins have surfactant properties similar to dodecyl benzene sulphonates. These interesting properties of aqueous extract of *Acacia Concinna* pods allow us to use it as an ecofriendly surfactant type catalyst for organic synthesis.

## II. RESULTS AND DISCUSSION

In continuation of our work following the principles of green chemistry,<sup>12</sup> we have developed a simple, efficient and green protocol for the preparation of 3-Acetyl quinolines using aqueous extract of pods of *Acacia Concinna* as a green and inexpensive catalyst (Scheme 1). Our approach reduces the use of hazardous organic solvents and uses simple and mild conditions, with inherently lower costs.



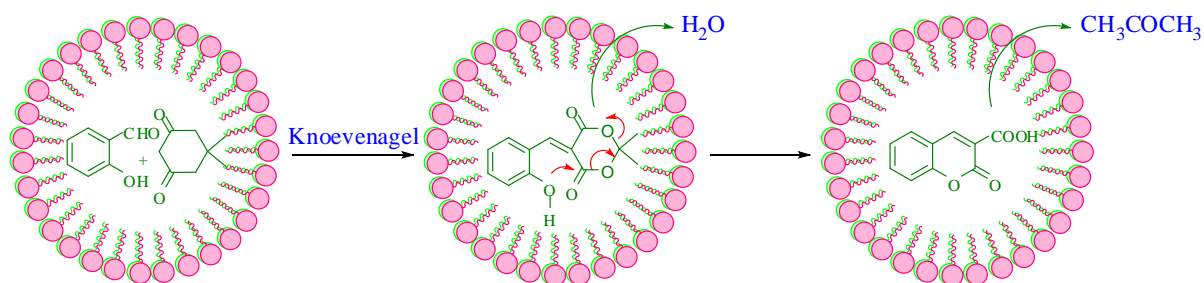
X= H, Cl

R1= -CH3, -Ph

Scheme 1. Synthesis of 3-acetyl/carboxylate quinolines

In the beginning a test reaction using 2-amino ketone (1 mmol) and acetyl acetone (1 mmol) in 5 ml 10% (W/V) aqueous extract of *Acacia Concinna* pods at room temperature was performed in order to establish the real effectiveness of the catalyst. And we are fortunate to get the excellent yield of the product **3a** (92%) after 3 hours. In order to optimize the reaction conditions same reaction was carried out using different concentrations of catalyst. It was found that 20% of catalyst shows maximum yield in minimum time (98%). Higher concentration of the catalyst (30%, 50%) neither increases the yield nor lowers the conversion time (Table 1). Thus, 20% (W/V), 5 ml aqueous extract was found to be the optimal quantity and sufficient to push the reaction forward.

In order to compare the strength of aqueous extract of *Acacia Concinna* pods as catalyst with aqueous solutions of various cationic, anionic and non-ionic surfactants, a model reaction was carried out between 2-hydroxy benzaldehyde (**1**) and Meldrum's acid (**2**) using various surfactants as catalyst and results are summarised in table 2. The solutions in consideration were taken well above their critical micellar concentrations (CMC). In the presence of acidic surfactant dodecylbenzenesulphonic acid (DBSA), the desired product was obtained in moderate yield (Table 2, entry 2). All other surfactants sodium dodecyl sulphate (SDS), Triton X-100, cetylpyridinium bromide (CPB), cetyltrimethylammonium bromide (CTAB) afforded the desired product **3a** in low yields (Table 2, entries 3-6). However, in the presence of aqueous extract of *Acacia Concinna* pods (a natural catalyst), the desired product was obtained in excellent yield within a short period of time (table 2, entry 7). When the reaction was carried out in water, without any surfactant no product formation was observed after 12h (Table 2, entry 1). Thus, aqueous extract of *Acacia Concinna* pods was found to be most obvious choice for further exploration of the methodology with other 2-hydroxy benzaldehyde derivatives. The advantageous effect of aqueous extract of *Acacia Concinna* pods may be attributed due to the aggregation of the substrates through hydrophobic interactions, which squeeze water molecules out of the organic phase (Figure 3)



**Figure 3.** Aggregation of the substrates through hydrophobic interactions.

The efficiency of this aqueous approach was studied for the synthesis of wide variety of 3-carboxycoumarin derivatives using the similar reaction conditions and results are summarized in table 3. The nature (electron withdrawing or electron donating) and position of the substituent on the aromatic ring did not show any marked differences in the yield of 3-carboxycoumarin derivatives. Mechanistically, the reaction was proceeding *via* Knoevenagel condensation followed by cyclization to obtain 3-carboxycoumarins.

**Table 1.** Optimization of the concentration of catalyst

Entry	% conc. of aqueous extract (W/V)	Time (min)	Yield (%) <sup>a</sup>
1.	5	300	20
2.	10	190	92
3.	20	120	98
4.	30	120	98
5.	50	120	96

<sup>a</sup> isolated yield

**Table 2.** Effect of catalyst on the yield of 3-acetyl/Carboxylate quinolines

Entry	Surfactant	Time (hr)	Yield (%) <sup>a</sup>
1.	None	13	42
2.	DBSA	11	68
3.	SDS	9	60
4.	Triton X-100	8	52
5.	CPB	7	50
6.	CTAB	5	65
7.	Malonic acid	6	78
8.	Citric acid	5	94
9.	<i>p</i> -anisic acid	8	69
10.	Proline	6	70
11.	Catalyst (20%)	2	98

<sup>a</sup>Isolated yield

To further explore the potential of this aqueous approach, we have successfully applied this approach for the synthesis of cinnamic acid derivatives by the condensation of various aromatic aldehydes other than salicylaldehydes with Meldrum's acid followed by ring opening. Initially, when the reaction of benzaldehyde and Meldrum's acid was carried out at room temperature using aqueous extract of *Acacia Concinna* pods, only condensation product **5a** was observed. But, when the same reaction was carried out at 50-60°C, formation of cinnamic acid **6a** was observed in excellent yield (Scheme 2). With the optimized procedure in hand, an investigation into the scope of the methodology, using a range of aldehydes was conducted (Table 4,5).

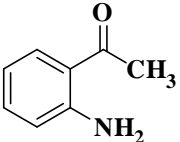
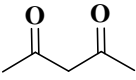
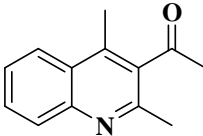
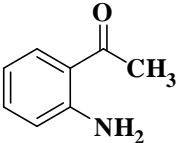
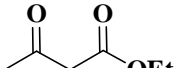
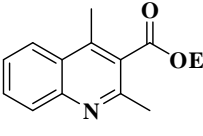
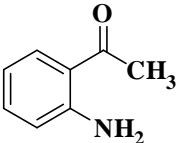
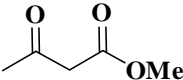
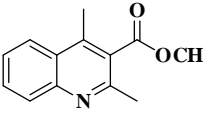
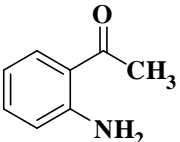
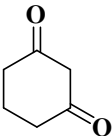
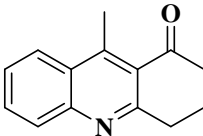
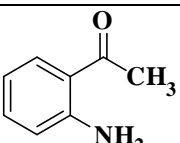
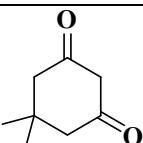
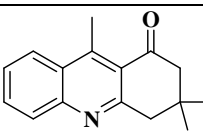
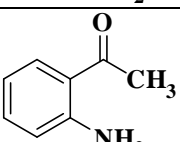
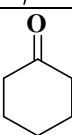
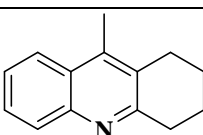
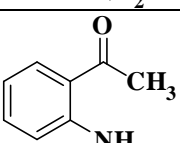
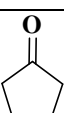
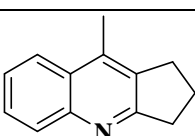
Perkin reaction is the most frequently used method for the preparation of the cinnamic acid and its derivatives but the main disadvantage of this reaction is aldehydes in the presence of base lead to formation of unwanted side products formation. As well as in the presence of electron-donor substituent, the yield of the target product markedly decreases; in such systems the Perkin reaction is not employed for preparative purposes. For the electron donating groups,

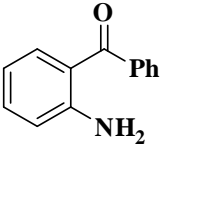
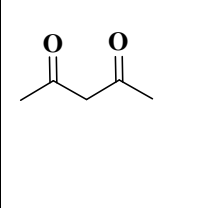
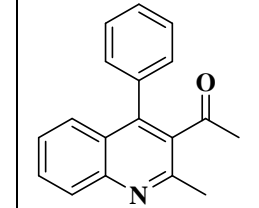
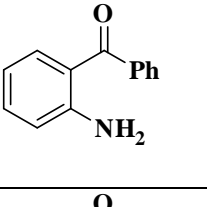
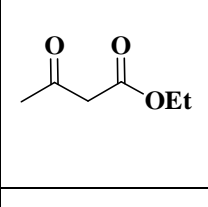
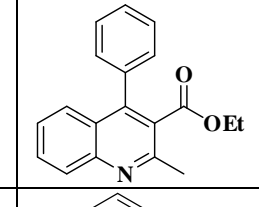
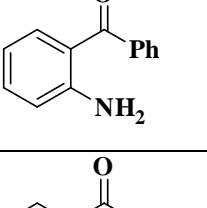
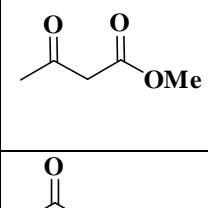
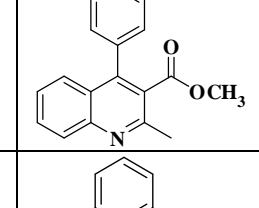
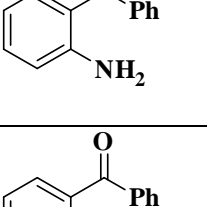
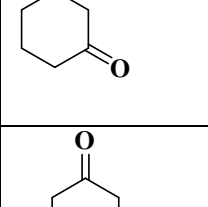
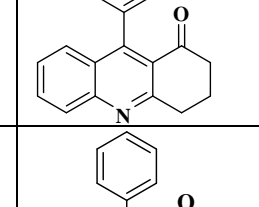
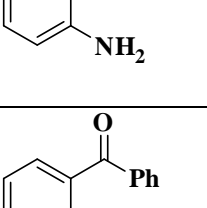
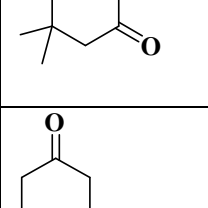
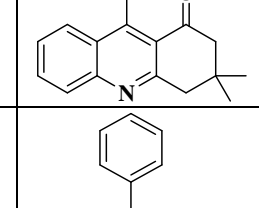
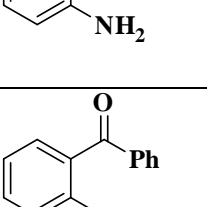
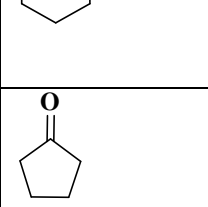
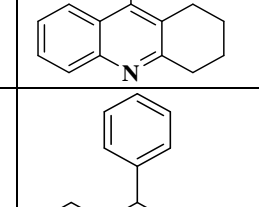
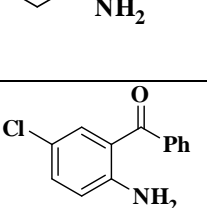
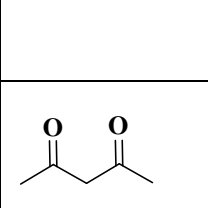
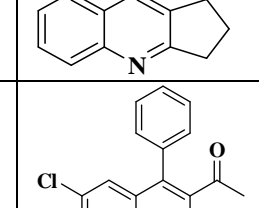
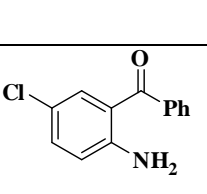
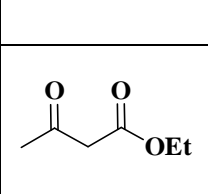
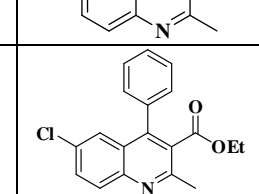

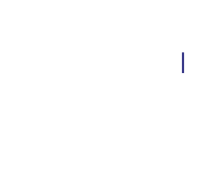



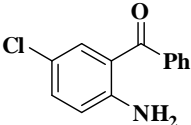
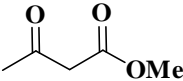
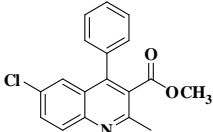
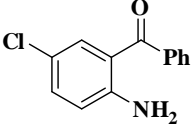
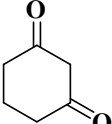
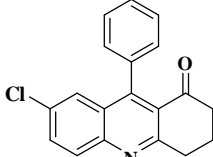
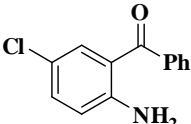
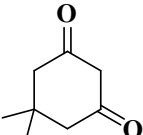
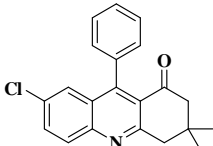
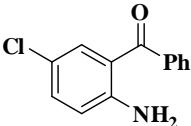
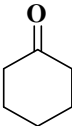
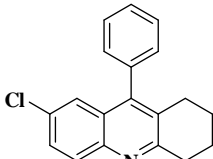
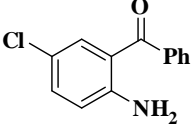
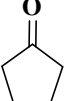
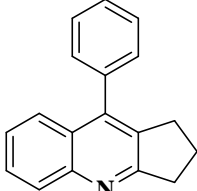
Knoevenagel and Debner modification reactions lead to the good yields of the final product but the main drawback is that reaction needs long duration of time.

In recent years, various other methods have been reported for the preparation of cinnamic acid and its derivatives such as microwave irradiation of aryl aldehydes and malonic acid with polyphosphate ester (PPE) as the mediator catalyst in solvent-free condition,<sup>17</sup> using POCl<sub>3</sub> as an acid catalyst.<sup>18</sup> But, the long reaction time, elevated temperature, unsatisfactory yields and only worked very well for electron donating substituent's are the major disadvantage of these methods.

This aqueous protocol overcomes all these drawbacks and proceeds smoothly for substrates containing both electron donating as well as withdrawing groups within a very short period of time under mild condition resulting in excellent yields of substituted cinnamic acids.

Entry	$\alpha$ - amino aryl ketone	$\alpha$ - methylene ketone	product	Time Hr	Yield	M.P.
1				3		
2				3		
3				3		
4				3		
5				3		
6				3		
7				3		

8				2			
9				2			
10				2			
11				2			
12				2			
13				2			
14				2			
15				2			
16				2			

17				2			
18				2			
19				2			
20				2			
21				2			

### III. EXPERIMENTAL

#### General remarks

All chemicals were obtained from commercial suppliers and were used without further purification. Melting points were determined in open capillaries and were uncorrected. IR spectra were recorded on FT-IR Nicolet iS 10 spectrophotometer in KBr pellet and  $^1\text{H}$  NMR spectra were recorded on a Bruker 400 MHz spectrometer in  $\text{CDCl}_3$  using tetramethylsilane as internal standard and chemical shifts are reported in  $\delta$  units and the coupling constants (J) are reported in Hertz. Mass spectra were obtained with a Shimadzu LCMS-2010EV. TLC was performed on aluminum-backed silica plate with visualization by UV-light and column chromatography using silica gel (mesh size 100–200). The purity of the compounds was checked by TLC on precoated  $\text{SiO}_2$  gel (HF254, 100–200 mesh) aluminum plates (E. Merck) using *n*-hexane: EtOAc (9:1) as mobile phase and visualized by iodine vapors. Dry *Acacia concinna* pods were purchased from local market and authenticated from School of Life Sciences, SRTMU, Nanded (India).

#### General procedure for the preparation of catalyst

Powdered pods of *Acacia Concinna* fruit (20 g) were taken into a 250 ml conical flask; to it water (100 ml) was added and allowed to boil for 15 min. The material was then filtered off till hot and discard the residue. The aqueous extract was employed as a catalyst (20%) for the synthesis of 3-carboxycoumarins.

#### General procedure for the synthesis of 3-acetyl quinolines

A mixture of *o*-amino-aryl ketone (1.0 mmol),  $\alpha$ -methylene ketone (1.1 mmol) and catalyst (20%, 5 ml), was stirred at room temperature under for appropriate time (Table 3). After completion of the reaction as indicated by TLC, the reaction mixture was diluted with water (30 mL) and the reaction mixture was extracted in  $\text{Et}_2\text{O}$  (2×5 mL), dried over  $\text{MgSO}_4$  and concentrated *in vacuo* to give the crude product which was further purified either by a short column chromatography (silica gel, EtOAc-hexane, 1:9) to afford the corresponding pure quinoline derivatives.

All the compounds were known and characterized by IR, NMR, mass spectrometry and also by comparing their

physical characteristics with those in the literature.<sup>4-7</sup>

Please see supporting information for spectral data for selected compounds.

#### Spectral data for representative compounds:

**Entry 3, Table 3:** Oil; IR (NaCl):  $\nu$ 3068, 2959, 1703, 1614, 1585, 1208, 758  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ):  $\delta$  2.57 (s, 3H), 2.58 (s, 3H), 2.62 (s, 3H), 7.53-8.01 (m, 4H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ ):  $\delta$  15, 23.3, 32.4, 123.5, 126.2, 129, 129.6, 135.6, 138.4, 146.7, 152.4, 206.3; MS (EI, 70 eV):  $m/z$  = 199 ( $\text{M}^+$ ), 158, 125; Anal. Calcd for  $\text{C}_{13}\text{H}_{13}\text{NO}$ : C, 78.36; H, 6.58; N, 7.03. Found: C, 78.32; H, 6.51; N, 7.07.

**Entry 5, Table 3:** Solid; mp 60°C; IR (KBr):  $\nu$ 3065, 2957, 1613, 908, 751  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ):  $\delta$  2.2 (m, 2H), 2.49 (s, 3H), 2.99 (t,  $J$  = 7.55 Hz, 2H), 3.3 (t,  $J$  = 6.9 Hz, 2H), 7.46-8.01 (m, 4H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ ):  $\delta$  14.6, 22.7, 29.4, 34.8, 123.1, 125.0, 126.9, 127.8, 128.9, 133.7, 137.8, 147.2, 166.7; MS (EI, 70 eV):  $m/z$  = 183 ( $\text{M}^+$ ), 168, 154, 140, 127, 115, 102, 90, 77, 63, 57; Anal. Calcd for  $\text{C}_{13}\text{H}_{13}\text{N}$ : C, 85.21; H, 7.15; N, 7.64. Found: C, 85.17; H, 7.07; N, 7.72. **Entry 9, Table 3:** Yellow solid; mp 210 °C; IR (KBr):  $\nu$  3074, 2952, 2866, 1696, 1554, 1477, 1384, 1297, 1198, 1079, 837, 699  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ):  $\delta$  1.15 (s, 6H), 2.56 (s, 2H), 3.25 (s, 2H), 7.14-7.17 (m, 2H), 7.43-7.97 (m, 6H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ ):  $\delta$  28.3, 32.1, 48.3, 54.2, 123.3, 126.7, 127.8, 128.1, 128.3, 130.2, 132.4, 136.8, 147.4, 150.0, 161.4, 197.5; MS (EI, 70 eV):  $m/z$  = 335 ( $\text{M}^+$ ); Anal. Calcd for  $\text{C}_{21}\text{H}_{18}\text{ClNO}$ : C, 75.11; H, 5.40; N, 4.17. Found: C, 75.07; H, 5.43; N, 4.21. **Entry 16, Table 3:** Yellow solid; mp 108 °C; IR (KBr):  $\nu$  3064, 2983, 1725, 1605, 1224, 907, 732  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ): 0.92-0.95 (t,  $J$  = Hz, 3H), 2.73 (s, 3H), 4.03-4.07 (q,  $J$  = 7 Hz 2H), 7.32-8 (m, 8H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ ):  $\delta$  13.5, 23.6, 61.4, 125.1, 125.9, 128.4, 128.7, 129.2, 130.5, 131, 132.3, 135, 145.3, 146, 154, 168; MS (EI, 70 eV):  $m/z$  = 325 ( $\text{M}^+$ ), 296, 280, 252, 217, 189, 176, 149, 123, 109, 88, 71, 57; Anal. Calcd for  $\text{C}_{20}\text{H}_{19}\text{ClNO}_2$ : C, 70.05; H, 4.95; N, 4.30. Found: C, 70.09; H, 4.90; N, 4.36. **Entry 21, Table 3:** Yellow solid; mp 165°C; IR (KBr):  $\nu$ 3059, 2945, 2858, 1599, 1570, 1477, 1440, 1349, 1266, 1165, 1075, 937, 831, 818, 757, 708, 617  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ):  $\delta$  1.55 (m, 2H), 1.59 (m, 2H), 2.56 (t,  $J$  = 6.5 Hz, 2H), 3.3 (t,  $J$  = 7 Hz, 2H), 7.2-8.02 (m, 8H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ ):  $\delta$  23, 28.2, 34.3, 124.6, 127.5, 128.2, 128.9, 129.1, 129.3, 129.5, 130.2, 131.3, 136.5, 144.8, 145.8, 159.6; MS (EI, 70 eV):  $m/z$  = 293 ( $\text{M}^+$ ), 278, 258, 242, 230, 201, 189, 176, 150, 89, 77. Anal. Calcd for  $\text{C}_{19}\text{H}_{16}\text{ClN}$ : C, 77.68; H, 5.49; N, 4.77. Found: C, 77.59; H, 5.34; N, 4.68. **Entry 29, Table 3:** Solid; mp 101-103 °C; IR (KBr):  $\nu$  3055, 1684, 1483, 907, 729  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.4 - 8.02 (m, 14H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ ):  $\delta$  120, 124.4, 126.4, 127.5, 128.7, 128.8, 129.4, 130.4, 131.6, 132.7, 137.7, 139.1, 147.2, 148.4, 157; MS (EI, 70 eV):  $m/z$  = 316 ( $\text{M}^+$ ), 288, 125; Anal. Calcd for  $\text{C}_{21}\text{H}_{14}\text{ClN}$ : C, 79.87; H, 4.47; N, 4.44. Found: C, 79.81; H, 4.52; N, 4.41.

#### IV. CONCLUSION

In conclusion, we have found that aqueous extract of *Acacia Concinna* pods as an efficient, economical and environmental friendly catalyst for the synthesis of 3-acetyl/carboxylate quinolines. The high yield of products in short reaction time with high purity, mild reaction condition and simple work up procedure makes this procedure more attractive. The use of water as a solvent and biodegradable catalyst gives environmental benefits such as no environmental pollution by harmful organic solvents and easy waste treatment.

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