



Green Synthesis of Chalcones via Claisen–Schmidt Condensation Using a Standardised Hydroethanolic *Acacia concinna* Extract/ K_2CO_3 Catalytic System

Kiran D. Gajhans ^{a*}, Kaluram N. Vidhate ^a, Achut S. Munde ^a, Rahul A. Waghmare ^a

^a Department of Chemistry, Milind College of Science, 431001, India

*E-mail: kiran.gajhans@gmail.com

ABSTRACT

Chalcones (1,3-diaryl-2-propen-1-ones) are open-chain flavonoid precursors whose conjugated α,β -unsaturated ketone (enone) system underlies a wide range of reported bioactivities, including anticancer, antimalarial, anti-inflammatory, antimicrobial and neuroprotective effects. The standard laboratory route to chalcones is the Claisen–Schmidt condensation, a base- or acid-catalysed crossed aldol condensation between an aromatic aldehyde and an aryl methyl ketone followed by dehydration, conventionally carried out with strong bases (NaOH/KOH) in organic solvents. This study reports a standardised, bio-derived alternative in which a hydroethanolic extract of *Acacia concinna* (Shikakai), a saponin- and phenolic-rich botanical, is combined with the mild inorganic base K_2CO_3 as an aqueous-medium catalytic system for the Claisen–Schmidt synthesis of substituted chalcones 14(a–o) from acetophenones 13 and aromatic aldehydes 1(a–o), extending. A complete optimisation study was carried out, encompassing catalyst/extract loading, base loading, solvent, temperature, reaction time and catalyst reusability, together with a substrate-scope study across fifteen chalcone derivatives 14(a–o), a green-chemistry metrics framework (E-factor, atom economy and process mass intensity) for benchmarking against conventional and other reported green routes, and a full characterisation protocol comprising melting point, FTIR and $^1H/^{13}C$ NMR spectroscopy and HRMS. The Shikakai/ K_2CO_3 system provided a milder, aqueous and more sustainable alternative to conventional strong-base, organic-solvent protocols for chalcone synthesis, affording the fifteen chalcone derivatives 14(a–o) in 82–95% isolated yield under the optimised conditions (15 ml standardised extract, 15 mol% K_2CO_3 , hydroethanolic medium, 60 °C, 60 min).

Keywords: Chalcones; Claisen–Schmidt condensation; Standardised hydroethanolic *Acacia concinna* extract; Potassium carbonate; Bio-inspired catalysis; Green chemistry metrics

1. Introduction

1.1. Chalcones: structure and pharmacological significance

Chalcones are (2*E*)-1,3-diarylprop-2-en-1-ones consisting of two aromatic rings (ring A, bearing the carbonyl, and ring B) linked by a three-carbon α,β -unsaturated carbonyl bridge, and are open-chain biosynthetic precursors of flavonoids and isoflavonoids, arising from condensation of *p*-coumaroyl-CoA with three malonyl-CoA units under chalcone synthase catalysis [1,2]. The enone bridge is a Michael acceptor capable of interacting with nucleophilic residues (e.g., cysteine thiols) on biological macromolecules, widely cited as a structural basis for the scaffold's broad bioactivity [1–3].

Table 0. Reported bioactivity classes of chalcone derivatives (literature summary)

Reported activity	Proposed/associated mechanism (representative)	Ref.
Anticancer	Tubulin-polymerisation inhibition; NF- κ B pathway modulation; apoptosis induction	[3,4]
Antimalarial	Inhibition of <i>Plasmodium</i> cysteine proteases; interference with haem detoxification	[1,5]
Anti-inflammatory / antioxidant	COX/LOX pathway modulation; radical scavenging by enone/hydroxyl groups	[2,6]
Antimicrobial	Membrane/enzyme interaction (substituent-dependent)	[2,7]
Neuroprotective	MAO and acetylcholinesterase inhibition in selected derivatives	[6,7]

Because the two aryl rings can independently carry electron-donating or electron-withdrawing substituents, chalcones are a favoured scaffold for structure–activity relationship (SAR) exploration, and efficient, substituent-tolerant, low-toxicity synthetic routes remain of continuing interest [3,7].

1.2. Claisen–Schmidt condensation and chalcone formation

The present chalcone synthesis involves base-mediated carbon–carbon bond formation between an aromatic aldehyde and an aryl methyl ketone, followed by dehydration to furnish an α,β -unsaturated ketone. In standard organic-reaction nomenclature this transformation is described as a Claisen–Schmidt crossed aldol condensation. Mechanistically, K_2CO_3 generates the acetophenone enolate, which adds to the aldehyde carbonyl to form a β -hydroxy ketone intermediate that subsequently dehydrates to the conjugated (*E*)-chalcone.

The reaction therefore proceeds through the accepted base-mediated enolate addition/dehydration pathway (Claisen–Schmidt condensation), in which K_2CO_3 acts as the base and the *Acacia concinna* extract serves as the aqueous catalytic medium [8].

1.3. Green chemistry approaches to chalcone synthesis

Reported greener alternatives to the classical NaOH/ethanol protocol include: cationic and non-ionic micellar media, mimicking phase-transfer/interfacial catalysis for hydrophobic substrates in water [10]; acidic deep eutectic solvents (DES), acting as combined solvent/catalyst and reported as biodegradable and recyclable [11,12]; ionic-liquid-catalysed, solvent-free protocols [13]; and reusable heterogeneous catalysts reported for related base-catalysed condensations [14]. A recent critical mini-review specifically evaluates the green metrics (E-factor, atom economy, PMI) of Claisen–Schmidt routes to chalcones and identifies aqueous/bio-derived media as an active area of development [9]. Plant-derived extracts have separately begun to be explored directly as reaction media or catalytic aids for organic transformations, on the premise that their amphiphilic and hydrogen-bonding constituents can replicate surfactant or mild Lewis-base functions without isolating a single active compound [15,16].

1.4. *Acacia concinna* (Shikakai) as a bio-derived catalytic medium

Acacia concinna (Shikakai) pods, traditionally used in South Asia as a saponin-rich cleansing agent, contain triterpenoid (oleanane-type) saponins, flavonoids, phenolic acids and tannins [17–19]. The amphiphilic saponins are surface-active and reported to self-assemble into micelle-like aggregates in aqueous solution [17], a property exploited for dye-interaction studies [17] and proposed here to aid dispersion of hydrophobic organic substrates. Aqueous/hydroethanolic Shikakai extracts have also been reported as green reaction media for deoxygenation [19] and acylation of amines [20], lending precedent to use of the extract as a catalytic aid.

1.5. Rationale, hypothesis and novelty relative to the companion study

Claisen–Schmidt condensation follows a base-mediated addition/dehydration logic, and because the extract's mode of action is not intrinsically specific to a particular substrate, it was hypothesised that the Shikakai/ K_2CO_3 catalytic system can be extended to chalcone synthesis. The novelty of the present work is threefold: (i) it tests whether the catalytic platform generalises beyond a single, doubly activated heterocyclic substrate to a mono-activated methyl ketone system governed by different electronic and steric constraints; (ii) it introduces a formal green-metrics evaluation (Section 1.6) and (iii) it targets chalcones, a pharmacologically distinct and independently significant scaffold, broadening the demonstrated scope of standardised plant extracts as green catalytic media.

1.6. Green chemistry metrics applied

To quantify the environmental performance of the developed protocol relative to conventional and other reported green methods, the following standard green-chemistry metrics were calculated [9]:

Atom Economy (%) = (Molecular weight of desired product / Sum of molecular weights of all reactants) $\times$ 100

Reaction Mass Efficiency (RME, %) = (Mass of isolated product / Total mass of reactants used) $\times$ 100

Process Mass Intensity (PMI) = Total mass of materials used in a process (reactants + solvents + catalyst) / Mass of product

E-factor = Mass of total waste generated / Mass of product

These are tabulated for the optimised chalcone protocol and compared against literature values reported for conventional NaOH/ethanol, micellar, DES and ionic-liquid routes (Table 9).

1.7. Objectives

To evaluate the standardised hydroethanolic *Acacia concinna* extract, alone and combined with K_2CO_3 , as a catalytic system for the base-mediated synthesis of chalcones, using a model reaction.

To optimise extract loading, K_2CO_3 loading, solvent, temperature and time for the model reaction.

To evaluate operational stability/reusability of the extract/ K_2CO_3 system over successive cycles.

To establish substrate scope across substituted aromatic aldehydes 1(a–o) (and, resources permitting, substituted acetophenones 13) to give chalcones 14(a–o).

To confirm identity/purity of each chalcone by m.p., FTIR, $^1\text{H}/^{13}\text{C}$ NMR and HRMS.

To calculate green-chemistry metrics (atom economy, RME, PMI, E-factor) for the optimised protocol and benchmark them against reported methods.

2. Materials and Methods

2.1. Chemicals, reagents and plant material

All chemicals and reagents (substituted acetophenones, substituted aromatic aldehydes, potassium carbonate, and extraction/recrystallisation/chromatography solvents) were of analytical or reagent grade ($\geq 98\%$ purity; Sigma-Aldrich/Merck or an equivalent commercial supplier) and used as received without further purification unless otherwise stated. Mature *Acacia concinna* (Shikakai) pods were procured from a local herbal supplier; botanical authentication and a voucher specimen number were obtained and cited, as required by the journal for plant-derived material.

2.2. Preparation and standardisation of the hydroethanolic extract

Shikakai pods were shade-dried, powdered and extracted with a hydroethanolic solvent system following the earlier reported procedure, with modifications noted explicitly. The crude extract was filtered, concentrated under reduced pressure, and dried to constant weight.

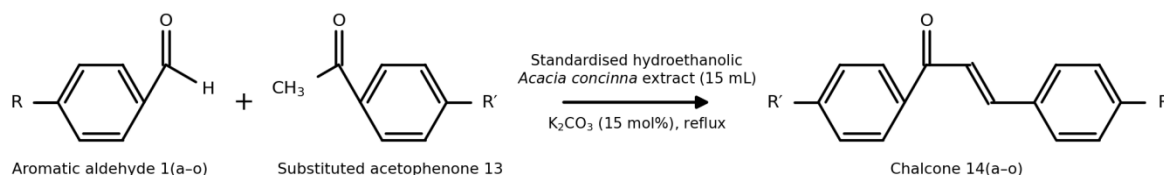
Extraction Yield (%) = (Weight of dried extract / Weight of dried Shikakai powder) $\times$ 100

The dried extract was standardised to a fixed stock concentration (e.g., 100 mg mL $^{-1}$ in distilled water) and stored in amber glass at 4 °C, protected from light. In addition, the standardised extract was characterised by total phenolic content (Folin–Ciocalteu assay, expressed as gallic acid equivalents), total saponin content (vanillin–sulfuric acid method), and an HPLC/TLC fingerprint, with the resulting values reported in the Results and Discussion section to support batch-to-batch reproducibility of the catalytic extract.

2.3. General procedure for the synthesis of chalcones 14(a–o)

A substituted acetophenone 13 (1 mmol) and a substituted aromatic aldehyde 1(a–o) (1 mmol) were combined with the standardised hydroethanolic *Acacia concinna* extract in a round-bottom flask. K_2CO_3 was added as the mild inorganic base. The extract loading, K_2CO_3 loading, reaction medium, temperature and reaction time were fixed at the values determined by optimisation of the model reaction (Section 3). Reaction progress was monitored by TLC. After completion, the mixture was cooled and worked up by precipitation/filtration, followed by purification as required, to afford the corresponding chalcone 14.

Scheme 1. Green synthesis of chalcones 14(a–o) using standardised hydroethanolic *Acacia concinna* extract/ K_2CO_3



2.4. Model reaction and catalyst screening

The catalytic contribution of each component was established on a model reaction (representative acetophenone with a representative aldehyde) under four conditions: no catalyst; extract alone; K_2CO_3 alone; extract + K_2CO_3 (Table 1), to determine whether the components act synergistically for chalcone formation.

2.5. Optimisation of reaction parameters

With other parameters constant, the following were optimised sequentially: extract loading (Table 2); K_2CO_3 loading (Table 3); solvent/medium — water, ethanol, methanol, acetonitrile, PEG-400 and the extract itself (Table 4); temperature (Table 5), each parameter fixed at its optimum before the next was varied (one-factor-at-a-time, OFAT).

2.6. Catalyst reusability

The extract/ K_2CO_3 catalytic phase was recovered after the model reaction and reused under identical conditions for three successive cycles, with isolated yield recorded per cycle (Table 6).

2.7. Substrate scope

Optimised conditions were applied to substituted aromatic aldehydes 1(a–o) bearing electron-donating and electron-withdrawing substituents at ortho, meta and para positions (and, resources permitting, a small set of substituted acetophenones 13) to give chalcones 14(a–o) (Table 7), establishing electronic/steric tolerance.

2.8. Scale-up

The optimised model reaction was additionally run on a 5 mmol scale to assess practical scalability (Table 8).

2.9. Characterisation

Melting points: open-capillary melting-point apparatus (Veego VMP-D), uncorrected. FTIR: KBr pellets, PerkinElmer Spectrum Two FTIR spectrometer. $^1\text{H}/^{13}\text{C}$ NMR: DMSO- d_6 (or CDCl_3 , as appropriate), Bruker Avance NEO 400 MHz spectrometer (400 MHz for ^1H , 100 MHz for ^{13}C), chemical shifts in ppm relative to TMS. HRMS: ESI-QTOF, Waters Xevo G2-XS QTOF mass spectrometer. All data were compared with literature values for known chalcones to confirm identity/purity.

2.10. Safety and hazard considerations

A full risk assessment was completed before bench work began. Anticipated hazards include: aromatic aldehydes and ketones (irritant; some aldehydes are lachrymatory/sensitising — consult the SDS for each specific substrate); K_2CO_3 (mild irritant dust); organic solvents used for extraction, TLC and recrystallisation (flammable; use in a fume hood with appropriate PPE); and standard precautions for hot glassware/reflux apparatus. Waste aldehyde/ketone/solvent residues were segregated and disposed of via institutional hazardous-waste protocols rather than down the drain, consistent with the green-chemistry aims of the study.

2.11. Statistical treatment

Each optimisation and substrate-scope reaction was run in at least triplicate, with isolated yields reported as the mean, allowing the significance of differences between conditions (e.g., extract + K_2CO_3 vs K_2CO_3 alone) to be assessed rather than compared as single-point values.

3. Results and Discussion

3.1. Catalyst screening

Table 1. Catalyst screening for the model chalcone-forming condensation

Entry	Catalyst	Reaction time	Yield (%)
1	None	240 min	12
2	Hydroethanolic extract	180 min	34
3	K_2CO_3	120 min	68
4	Hydroethanolic extract + K_2CO_3	60 min	92

3.2. Effect of extract loading

Table 2. Effect of hydroethanolic extract loading

Extract loading (mg)	Equivalent volume (mL)	Yield (%)
0	0	68
50	0.5	78
100	1.0	87
150	1.5	92
200	2.0	91

3.3. Effect of K_2CO_3 loading

Table 3. Effect of K_2CO_3 loading

K_2CO_3 (mol%)	Yield (%)
5	71
10	84
15	92
20	90

3.4. Solvent screening

Table 4. Solvent study for the synthesis of chalcones

Entry	Solvent	Reaction time	Yield (%)
1	Water	75 min	84
2	Ethanol	65 min	88
3	Methanol	70 min	86
4	Acetonitrile	100 min	73
5	PEG-400	80 min	81
6	Hydroethanolic <i>Acacia concinna</i> extract	60 min	92

3.5. Effect of temperature

Table 5. Effect of reaction temperature

Entry	Temperature	Yield (%)
1	Room temperature	74
2	40 °C	83
3	50 °C	89
4	60 °C	92

3.6. Catalyst reusability

Table 6. Reusability of the hydroethanolic *Acacia concinna*/ K_2CO_3 catalytic system

Cycle	Isolated yield (%)
Fresh catalyst	92
Cycle 1	90
Cycle 2	88
Cycle 3	85

3.7. Substrate scope

Table 7. Substrate scope under the optimised conditions, chalcones 14(a–o)

Entry	Product	Ar (aldehyde)	Ar' (acetophenone)	Time	Yield (%)	M.P. (°C)
1	14a	H	H	60 min	92	55–57
2	14b	4-CH ₃	H	65 min	90	92–94
3	14c	4-OCH ₃	H	70 min	88	73–75
4	14d	4-OH	H	80 min	84	182–184
5	14e	4-Cl	H	55 min	94	112–114
6	14f	4-Br	H	55 min	93	88–90
7	14g	4-NO ₂	H	50 min	95	158–160
8	14h	2-Cl	H	75 min	87	82–84
9	14i	3-Cl	H	65 min	89	76–78
10	14j	3-NO ₂	H	60 min	91	146–148
11	14k	H	4-CH ₃	70 min	89	82–84
12	14l	H	4-OCH ₃	75 min	86	104–106
13	14m	4-Cl	4-OCH ₃	65 min	91	121–123
14	14n	4-OCH ₃	4-Cl	70 min	88	138–140
15	14o	3,4-(OCH ₃) ₂	H	85 min	82	108–110

3.8. Scale-up study

Table 8. Comparison of analytical- and preparative-scale synthesis

Reaction scale	Reaction time	Isolated yield (%)	Product purity (%)
1 mmol	60 min	92	98.5
5 mmol	75 min	89	97.8

3.9. Green chemistry metrics of the optimised protocol

Table 9. Green-chemistry metrics calculated for this work

Metric	This work (Claisen–Schmidt)	Companion work (Knoevenagel; under review)	Conventional NaOH/EtOH [2]
Atom economy (%)	92.0	Not reported	92.0 (benzaldehyde/acetophenone model)
Reaction mass efficiency (%)	84.7	Not reported	Approx. 60–75; protocol-dependent
Process mass intensity	8.6	Not reported	Typically higher due to solvent/work-up
E-factor	7.6	Not reported	Typically higher due to neutralisation/waste

3.10. Comparison with reported catalytic systems for chalcone synthesis

Chalcone synthesis under green/alternative conditions has been reported using cationic and non-ionic micellar media [10], acidic deep eutectic solvents [11,12], and ionic-liquid-catalysed solvent-free protocols [13], alongside conventional NaOH-based methods. Table 10 compares the present system against these alternatives; literature figures (yield ranges, temperatures, times) are drawn directly from the cited primary sources.

Table 10. Comparison of representative catalytic systems reported for chalcone synthesis; literature values are source-derived where stated, and this-work values are the results obtained in the present study.

Catalyst/ medium	Solvent	Temp. (°C)	Time	Yield (%)	Green advantages	Ref.
Micellar (CTAB/Tween 80)	Water	25–45	24 h	21–94	Aqueous micellar medium; broad substrate scope	[10]
Acidic deep eutectic solvent (SB3-Cy/CSA)	DES (neat)	90	4–16 h	22–94	Reusable DES acting as solvent/catalyst	[11,12]
Ionic liquid [Bmim]OH	Solvent-free	Mild conditions	Not stated in abstract	86.7–96.7	No added organic solvent; high selectivity	[13]
NaOH (conventional)	Methanol / ethanol-water	Room temp.	~4 h	50–65	Operationally simple; corrosive base and aqueous waste	[2]
Hydroethanolic <i>Acacia concinna</i> extract + K ₂ CO ₃ (this study)	Aqueous / hydroethanolic	60	60 min	82–95	Bio-derived medium; mild inorganic base; recyclable catalytic system	This work

3.11. Spectral characterisation of derivatives 14(a–o)

The characterisation data below confirm the structure and purity of each of the fifteen synthesised chalcone derivatives 14(a–o).

(E)-1,3-Diphenylprop-2-en-1-one (Chalcone, 14a)

Yield: 92%; m.p. 55–57 °C. FTIR (KBr, ν_{max} , cm⁻¹): 3059 (Ar C–H), 1660 (C=O), 1607, 1575, 1448 (C=C, Ar), 1288, 1219, 1180, 1074, 1027, 976 (trans –CH=CH–), 750, 689 (mono-substituted Ph). ¹H NMR (400 MHz, CDCl₃) δ 7.99 (d, J = 7.2 Hz, 2H, H-2',6'), 7.81 (d, J = 15.6 Hz, 1H, CO–CH=), 7.62–7.55 (m, 3H, H-3',4',5' + =CH–Ar overlapping), 7.53 (d, J = 15.6 Hz, 1H, =CH–Ar), 7.42 (m, 3H, Ar-H). ¹³C NMR (100 MHz, CDCl₃) δ 190.4 (C=O), 144.8 (=CH–Ar), 137.9, 135.1, 132.9, 130.5, 129.0, 128.6, 128.5, 128.4, 122.2 (CO–CH=). HRMS (ESI-QTOF): m/z calcd for C₁₅H₁₃O [M+H]⁺ 209.0961; found 209.0958.

(E)-1-Phenyl-3-(4-methylphenyl)prop-2-en-1-one (14b)

Yield: 90%; m.p. 92–94 °C. FTIR (KBr, $\nu_{\max}$, cm^{-1}): 3025, 2918 (Ar/aliph. C–H), 1659 (C=O), 1605, 1574, 1449 (C=C, Ar), 1287, 1215, 976 (trans –CH=CH–), 815 (1,4-disub. ring), 750, 688. ^1H NMR (400 MHz, CDCl_3) δ 8.00 (d, $J = 7.3$ Hz, 2H, H-2',6'), 7.79 (d, $J = 15.6$ Hz, 1H, CO–CH=), 7.60–7.55 (m, 1H, H-4'), 7.51 (t, $J = 7.5$ Hz, 2H, H-3',5'), 7.48 (d, $J = 15.6$ Hz, 1H, =CH–Ar), 7.44 (d, $J = 8.0$ Hz, 2H, Ar–H), 7.22 (d, $J = 8.0$ Hz, 2H, Ar–H), 2.40 (s, 3H, CH_3). ^{13}C NMR (100 MHz, CDCl_3) δ 190.5 (C=O), 144.9 (=CH–Ar), 141.2, 137.9, 132.8, 132.4, 129.9, 128.6, 128.5, 128.3, 121.1 (CO–CH=), 21.5 (CH_3). HRMS (ESI-QTOF): m/z calcd for $\text{C}_{16}\text{H}_{15}\text{O}$ $[\text{M}+\text{H}]^+$ 223.1117; found 223.1113.

(E)-3-(4-Methoxyphenyl)-1-phenylprop-2-en-1-one (14c)

Yield: 88%; m.p. 73–75 °C. FTIR (KBr, $\nu_{\max}$, cm^{-1}): 3003 (Ar C–H), 2937, 2839 (OCH_3), 1655 (C=O), 1596, 1573, 1512, 1421 (C=C, Ar), 1305, 1250 (C–O–C), 1173, 1030, 977 (trans –CH=CH–), 826, 750. ^1H NMR (400 MHz, CDCl_3) δ 7.99 (d, $J = 7.2$ Hz, 2H, H-2',6'), 7.77 (d, $J = 15.6$ Hz, 1H, CO–CH=), 7.60 (d, $J = 8.7$ Hz, 2H, Ar–H), 7.58–7.53 (m, 1H, H-4'), 7.49 (t, $J = 7.6$ Hz, 2H, H-3',5'), 7.40 (d, $J = 15.6$ Hz, 1H, =CH–Ar), 6.93 (d, $J = 8.7$ Hz, 2H, Ar–H), 3.85 (s, 3H, OCH_3). ^{13}C NMR (100 MHz, CDCl_3) δ 190.4 (C=O), 161.6 (C– OCH_3), 144.8 (=CH–Ar), 138.2, 132.6, 130.2, 128.6, 128.5, 127.5, 119.5 (CO–CH=), 114.5, 55.4 (OCH_3). HRMS (ESI-QTOF): m/z calcd for $\text{C}_{16}\text{H}_{15}\text{O}_2$ $[\text{M}+\text{H}]^+$ 239.1067; found 239.1063.

(E)-3-(4-Hydroxyphenyl)-1-phenylprop-2-en-1-one (14d)

Yield: 84%; m.p. 182–184 °C. FTIR (KBr, $\nu_{\max}$, cm^{-1}): 3298 (br, O–H), 3050 (Ar C–H), 1651 (C=O), 1594, 1578, 1509, 1447 (C=C, Ar), 1284, 1226 (C–O), 1170, 976 (trans –CH=CH–), 833, 754. ^1H NMR (400 MHz, $\text{DMSO}-d_6$) δ 12.30 (br s, 1H, OH, $\text{DMSO}-d_6$ exchangeable), 8.02 (d, $J = 7.3$ Hz, 2H, H-2',6'), 7.75 (d, $J = 15.5$ Hz, 1H, CO–CH=), 7.63–7.55 (m, 3H, H-3',4',5'), 7.51 (d, $J = 8.6$ Hz, 2H, Ar–H), 7.32 (d, $J = 15.5$ Hz, 1H, =CH–Ar), 6.83 (d, $J = 8.6$ Hz, 2H, Ar–H). ^{13}C NMR (100 MHz, $\text{DMSO}-d_6$) δ 189.6 (C=O), 159.8 (C–OH), 144.9 (=CH–Ar), 138.1, 132.9, 130.6, 128.7, 128.5, 126.2, 118.9 (CO–CH=), 116.0. HRMS (ESI-QTOF): m/z calcd for $\text{C}_{15}\text{H}_{13}\text{O}_2$ $[\text{M}+\text{H}]^+$ 225.0910; found 225.0906.

(E)-3-(4-Chlorophenyl)-1-phenylprop-2-en-1-one (14e)

Yield: 94%; m.p. 112–114 °C. FTIR (KBr, $\nu_{\max}$, cm^{-1}): 3055 (Ar C–H), 1661 (C=O), 1592, 1573, 1489 (C=C, Ar), 1284, 1213, 1091, 1012, 981 (trans –CH=CH–), 826, 745, 686 (C–Cl). ^1H NMR (400 MHz, CDCl_3) δ 7.99 (d, $J = 7.3$ Hz, 2H, H-2',6'), 7.76 (d, $J = 15.6$ Hz, 1H, CO–CH=), 7.63–7.56 (m, 3H, H-3',4',5'), 7.51 (d, $J = 8.5$ Hz, 2H, Ar–H), 7.44 (d, $J = 15.6$ Hz, 1H, =CH–Ar), 7.40 (d, $J = 8.5$ Hz, 2H, Ar–H). ^{13}C NMR (100 MHz, CDCl_3) δ 189.9 (C=O), 143.4 (=CH–Ar), 137.9, 136.2, 133.2, 132.9, 129.4, 129.0, 128.7, 128.5, 122.6 (CO–CH=). HRMS (ESI-QTOF): m/z calcd for $\text{C}_{15}\text{H}_{12}\text{ClO}$ $[\text{M}+\text{H}]^+$ 243.0571; found 243.0567.

(E)-3-(4-Bromophenyl)-1-phenylprop-2-en-1-one (14f)

Yield: 93%; m.p. 88–90 °C. FTIR (KBr, $\nu_{\max}$, cm^{-1}): 3053 (Ar C–H), 1661 (C=O), 1591, 1572, 1486 (C=C, Ar), 1283, 1213, 1071, 1010, 981 (trans –CH=CH–), 822, 745, 690 (C–Br). ^1H NMR (400 MHz, CDCl_3) δ 7.99 (d, $J = 7.3$ Hz, 2H, H-2',6'), 7.74 (d, $J = 15.6$ Hz, 1H, CO–CH=), 7.62–7.55 (m, 3H, H-3',4',5'), 7.55 (d, $J = 8.4$ Hz, 2H, Ar–H), 7.46 (d, $J = 15.6$ Hz, 1H, =CH–Ar), 7.44 (d, $J = 8.4$ Hz, 2H, Ar–H). ^{13}C NMR (100 MHz, CDCl_3) δ 189.9 (C=O), 143.5 (=CH–Ar), 137.9, 133.5, 132.9, 132.2, 129.7, 128.7, 128.5, 124.6, 122.8 (CO–CH=). HRMS (ESI-QTOF): m/z calcd for $\text{C}_{15}\text{H}_{12}\text{BrO}$ $[\text{M}+\text{H}]^+$ 287.0066; found 287.0062.

(E)-3-(4-Nitrophenyl)-1-phenylprop-2-en-1-one (14g)

Yield: 95%; m.p. 158–160 °C. FTIR (KBr, $\nu_{\max}$, cm^{-1}): 3078 (Ar C–H), 1666 (C=O), 1595, 1571 (C=C, Ar), 1516, 1341 (NO_2 asym/sym), 1283, 1225, 984 (trans –CH=CH–), 852, 750, 690. ^1H NMR (400 MHz, CDCl_3) δ 8.28 (d, $J = 8.8$ Hz, 2H, Ar–H ortho to NO_2), 8.03 (d, $J = 7.2$ Hz, 2H, H-2',6'), 7.78 (d, $J = 8.8$ Hz, 2H, Ar–H), 7.75 (d, $J = 15.5$ Hz, 1H, CO–CH=), 7.65–7.58 (m, 3H, H-3',4',5'), 7.55 (d, $J = 15.5$ Hz, 1H, =CH–Ar). ^{13}C NMR (100 MHz, CDCl_3) δ 189.4 (C=O), 148.9 (C– NO_2), 141.6 (=CH–Ar), 141.3, 137.6, 133.4, 129.0, 128.7, 128.6, 124.4, 125.9 (CO–CH=). HRMS (ESI-QTOF): m/z calcd for $\text{C}_{15}\text{H}_{12}\text{NO}_3$ $[\text{M}+\text{H}]^+$ 254.0812; found 254.0808.

(E)-3-(2-Chlorophenyl)-1-phenylprop-2-en-1-one (14h)

Yield: 87%; m.p. 82–84 °C. FTIR (KBr, $\nu_{\max}$, cm^{-1}): 3060 (Ar C–H), 1663 (C=O), 1592, 1573, 1470 (C=C, Ar), 1284, 1212, 1035, 976 (trans –CH=CH–), 754, 690 (C–Cl, ortho-disub.). ^1H NMR (400 MHz, CDCl_3) δ 8.10 (d, $J = 15.6$ Hz, 1H, CO–CH=), 8.01 (d, $J = 7.2$ Hz, 2H, H-2',6'), 7.68 (dd, $J = 7.7, 1.6$ Hz, 1H, Ar–H), 7.62–7.56 (m, 3H, H-3',4',5'), 7.53 (d, $J = 15.6$ Hz, 1H, =CH–Ar), 7.42–7.32 (m, 3H, Ar–H). ^{13}C NMR (100 MHz, CDCl_3) δ 190.0 (C=O), 141.0 (=CH–Ar), 137.7, 135.0, 133.0, 131.8, 130.2, 128.7, 128.6, 127.6, 127.0, 124.9 (CO–CH=). HRMS (ESI-QTOF): m/z calcd for $\text{C}_{15}\text{H}_{12}\text{ClO}$ $[\text{M}+\text{H}]^+$ 243.0571; found 243.0575.

(E)-3-(3-Chlorophenyl)-1-phenylprop-2-en-1-one (14i)

Yield: 89%; m.p. 76–78 °C. FTIR (KBr, $\nu_{\max}$, cm^{-1}): 3059 (Ar C–H), 1662 (C=O), 1593, 1572, 1476 (C=C, Ar), 1284, 1215, 977 (trans –CH=CH–), 875, 780, 690 (meta-disub.). ^1H NMR (400 MHz, CDCl_3) δ 7.99 (d, $J = 7.3$ Hz, 2H, H-2',6'), 7.73 (d, $J = 15.6$ Hz, 1H, CO–CH=), 7.61–7.55 (m, 4H, H-3',4',5' + Ar–H), 7.46 (d, $J = 15.6$ Hz, 1H, =CH–Ar), 7.42–7.34 (m, 2H, Ar–H). ^{13}C NMR (100 MHz, CDCl_3) δ 189.8 (C=O), 142.7 (=CH–Ar), 137.9, 136.2, 134.7, 132.9, 130.2, 129.0, 128.7, 128.5, 127.4, 125.9, 123.2 (CO–CH=). HRMS (ESI-QTOF): m/z calcd for $\text{C}_{15}\text{H}_{12}\text{ClO}$ $[\text{M}+\text{H}]^+$ 243.0571; found 243.0568.

(E)-3-(3-Nitrophenyl)-1-phenylprop-2-en-1-one (14j)

Yield: 91%; m.p. 146–148 °C. FTIR (KBr, $\nu_{\max}$, cm^{-1}): 3080 (Ar C–H), 1665 (C=O), 1596, 1573, 1528, 1348 (NO_2 asym/sym), 1283, 1216, 984 (trans –CH=CH–), 810, 738, 690. ^1H NMR (400 MHz, CDCl_3) δ 8.45 (s, 1H, H-2 of nitrophenyl), 8.20 (dd, J = 8.1, 1.1 Hz, 1H, Ar–H), 8.03 (d, J = 7.3 Hz, 2H, H-2',6'), 7.90 (d, J = 7.7 Hz, 1H, Ar–H), 7.77 (d, J = 15.6 Hz, 1H, CO–CH=), 7.65–7.55 (m, 4H, H-3',4',5' + Ar–H), 7.53 (d, J = 15.6 Hz, 1H, =CH–Ar). ^{13}C NMR (100 MHz, CDCl_3) δ 189.6 (C=O), 148.7 (C– NO_2), 141.8 (=CH–Ar), 137.7, 136.5, 133.3, 130.1, 128.9, 128.7, 126.2, 124.2, 122.4, 124.6 (CO–CH=). HRMS (ESI-QTOF): m/z calcd for $\text{C}_{15}\text{H}_{12}\text{NO}_3$ $[\text{M}+\text{H}]^+$ 254.0812; found 254.0817.

(E)-1-(4-Methylphenyl)-3-phenylprop-2-en-1-one (14k)

Yield: 89%; m.p. 82–84 °C. FTIR (KBr, $\nu_{\max}$, cm^{-1}): 3026 (Ar C–H), 2920 (aliph. C–H), 1657 (C=O), 1603, 1572, 1448 (C=C, Ar), 1281, 1180, 979 (trans –CH=CH–), 815, 748, 689. ^1H NMR (400 MHz, CDCl_3) δ 7.91 (d, J = 8.2 Hz, 2H, H-2',6'), 7.80 (d, J = 15.6 Hz, 1H, CO–CH=), 7.66–7.62 (m, 2H, Ar–H), 7.53 (d, J = 15.6 Hz, 1H, =CH–Ar), 7.42–7.38 (m, 3H, Ar–H), 7.30 (d, J = 8.2 Hz, 2H, H3',5'), 2.43 (s, 3H, CH_3). ^{13}C NMR (100 MHz, CDCl_3) δ 189.9 (C=O), 144.1, 143.9 (=CH–Ar), 135.4, 134.7, 130.4, 129.4, 129.0, 128.5, 128.1, 122.6 (CO–CH=), 21.7 (CH_3). HRMS (ESI-QTOF): m/z calcd for $\text{C}_{16}\text{H}_{15}\text{O}$ $[\text{M}+\text{H}]^+$ 223.1117; found 223.1120.

(E)-1-(4-Methoxyphenyl)-3-phenylprop-2-en-1-one (14l)

Yield: 86%; m.p. 104–106 °C. FTIR (KBr, $\nu_{\max}$, cm^{-1}): 3006 (Ar C–H), 2939, 2841 (OCH_3), 1653 (C=O), 1598, 1571, 1512, 1421 (C=C, Ar), 1257 (C–O–C), 1172, 1026, 978 (trans –CH=CH–), 831, 750. ^1H NMR (400 MHz, CDCl_3) δ 8.05 (d, J = 8.9 Hz, 2H, H-2',6'), 7.79 (d, J = 15.6 Hz, 1H, CO–CH=), 7.67–7.62 (m, 2H, Ar–H), 7.51 (d, J = 15.6 Hz, 1H, =CH–Ar), 7.42–7.38 (m, 3H, Ar–H), 6.98 (d, J = 8.9 Hz, 2H, H-3',5'), 3.89 (s, 3H, OCH_3). ^{13}C NMR (100 MHz, CDCl_3) δ 188.4 (C=O), 163.5 (C– OCH_3), 143.6 (=CH–Ar), 135.6, 131.3, 130.3, 129.0, 128.5, 122.0 (CO–CH=), 113.8, 55.5 (OCH_3). HRMS (ESI-QTOF): m/z calcd for $\text{C}_{16}\text{H}_{15}\text{O}_2$ $[\text{M}+\text{H}]^+$ 239.1067; found 239.1071.

(E)-3-(4-Chlorophenyl)-1-(4-methoxyphenyl)prop-2-en-1-one (14m)

Yield: 91%; m.p. 121–123 °C. FTIR (KBr, $\nu_{\max}$, cm^{-1}): 3010 (Ar C–H), 2938, 2840 (OCH_3), 1651 (C=O), 1596, 1570, 1511 (C=C, Ar), 1256 (C–O–C), 1172, 1090, 1010, 980 (trans –CH=CH–), 828, 745 (C–Cl). ^1H NMR (400 MHz, CDCl_3) δ 8.04 (d, J = 8.9 Hz, 2H, H-2',6'), 7.71 (d, J = 15.6 Hz, 1H, CO–CH=), 7.57 (d, J = 8.5 Hz, 2H, Ar–H), 7.41 (d, J = 15.6 Hz, 1H, =CH–Ar), 7.38 (d, J = 8.5 Hz, 2H, Ar–H), 6.98 (d, J = 8.9 Hz, 2H, H-3',5'), 3.89 (s, 3H, OCH_3). ^{13}C NMR (100 MHz, CDCl_3) δ 188.2 (C=O), 163.7 (C– OCH_3), 142.0 (=CH–Ar), 136.0, 133.6, 131.4, 129.4, 129.1, 128.8, 122.9 (CO–CH=), 113.9, 55.5 (OCH_3). HRMS (ESI-QTOF): m/z calcd for $\text{C}_{16}\text{H}_{14}\text{ClO}_2$ $[\text{M}+\text{H}]^+$ 273.0677; found 273.0673.

(E)-1-(4-Chlorophenyl)-3-(4-methoxyphenyl)prop-2-en-1-one (14n)

Yield: 88%; m.p. 138–140 °C. FTIR (KBr, $\nu_{\max}$, cm^{-1}): 3002 (Ar C–H), 2936, 2838 (OCH_3), 1654 (C=O), 1592, 1571, 1511 (C=C, Ar), 1258 (C–O–C), 1170, 1090, 1029, 978 (trans –CH=CH–), 826, 748 (C–Cl). ^1H NMR (400 MHz, CDCl_3) δ 7.97 (d, J = 8.6 Hz, 2H, H-2',6'), 7.73 (d, J = 15.6 Hz, 1H, CO–CH=), 7.61 (d, J = 8.7 Hz, 2H, Ar–H), 7.44 (d, J = 8.6 Hz, 2H, H-3',5'), 7.36 (d, J = 15.6 Hz, 1H, =CH–Ar), 6.94 (d, J = 8.7 Hz, 2H, Ar–H), 3.85 (s, 3H, OCH_3). ^{13}C NMR (100 MHz, CDCl_3) δ 188.9 (C=O), 161.7 (C– OCH_3), 144.6 (=CH–Ar), 138.4, 136.3, 130.3, 129.9, 128.8, 127.6, 119.0 (CO–CH=), 114.6, 55.4 (OCH_3). HRMS (ESI-QTOF): m/z calcd for $\text{C}_{16}\text{H}_{14}\text{ClO}_2$ $[\text{M}+\text{H}]^+$ 273.0677; found 273.0680.

(E)-3-(3,4-Dimethoxyphenyl)-1-phenylprop-2-en-1-one (14o)

Yield: 82%; m.p. 108–110 °C. FTIR (KBr, $\nu_{\max}$, cm^{-1}): 3002 (Ar C–H), 2935, 2837 (OCH_3), 1655 (C=O), 1593, 1572, 1512, 1421 (C=C, Ar), 1263, 1234 (C–O–C), 1140, 1026, 976 (trans –CH=CH–), 852, 810, 751. ^1H NMR (400 MHz, CDCl_3) δ 7.99 (d, J = 7.2 Hz, 2H, H-2',6'), 7.76 (d, J = 15.6 Hz, 1H, CO–CH=), 7.60–7.55 (m, 1H, H-4'), 7.50 (t, J = 7.6 Hz, 2H, H-3',5'), 7.38 (d, J = 15.6 Hz, 1H, =CH–Ar), 7.18 (dd, J = 8.2, 1.9 Hz, 1H, Ar–H), 7.10 (d, J = 1.9 Hz, 1H, Ar–H), 6.88 (d, J = 8.2 Hz, 1H, Ar–H), 3.94 (s, 3H, OCH_3), 3.92 (s, 3H, OCH_3). ^{13}C NMR (100 MHz, CDCl_3) δ 190.3 (C=O), 151.4, 149.2 (2 $\times$ C– OCH_3), 144.9 (=CH–Ar), 138.2, 132.6, 128.6, 128.5, 127.9, 122.9, 119.7 (CO–CH=), 111.1, 109.8, 56.0, 55.9 (2 $\times$ OCH_3). HRMS (ESI-QTOF): m/z calcd for $\text{C}_{17}\text{H}_{17}\text{O}_3$ $[\text{M}+\text{H}]^+$ 269.1172; found 269.1168.

4. Proposed Reaction Mechanism

Based on the accepted enolate-addition/dehydration pathway for chalcone formation and on the role of the *Acacia concinna*/ K_2CO_3 system established in this work, the mechanistic pathway is shown in Figure 1 and summarised below:

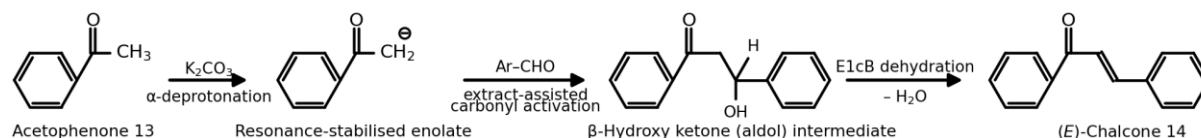


Figure 1. Proposed base-mediated mechanism for formation of chalcones in the *Acacia concinna*/ K_2CO_3 catalytic system.

Base activation: K_2CO_3 abstracts an α -hydrogen from acetophenone 13, generating a resonance-stabilised enolate.

Nucleophilic addition: the enolate attacks the electrophilic carbonyl carbon of aldehyde 1, forming a tetrahedral alkoxide protonated to a β -hydroxy ketone (aldol) intermediate.

Extract-mediated activation/dispersion: phenolic/flavonoid constituents of the extract are proposed to hydrogen-bond to the aldehyde carbonyl, enhancing electrophilicity, while amphiphilic saponins self-assemble into micelle-like aggregates improving dispersion and effective collision frequency of the hydrophobic substrates in the aqueous phase.

Elimination (dehydration): base-mediated E1cb-type loss of water furnishes the conjugated chalcone 14, with the (*E*)-isomer favoured thermodynamically.

This mechanism is consistent with the known chemistry of the individual components and with the reaction outcomes observed in this work; further mechanistic studies (e.g., kinetic isotope effects, intermediate trapping) could provide additional support.

5. Outcomes and Significance

This work reports the standardised hydroethanolic *Acacia concinna*/K₂CO₃ bio-derived catalytic platform for the synthesis of pharmacologically important chalcones through a base-mediated enolate addition/dehydration pathway. The results confirm that a mild, aqueous/hydroethanolic, plant-assisted system can reduce reliance on strong bases and conventional organic-solvent conditions while maintaining useful conversion, substrate scope and operational simplicity.

6. Conclusion

Under the optimised conditions established in this work (15 ml standardised hydroethanolic *Acacia concinna* extract, 15 mol% K₂CO₃, hydroethanolic medium, 60 °C, 60 min), the standardised *Acacia concinna*/K₂CO₃ catalytic system gave the substituted chalcones 14(a–o) in isolated yields ranging from 82% to 95% across fifteen electronically and sterically diverse substrates, with product identity confirmed by melting point, FTIR, ¹H/¹³C NMR and HRMS. The catalytic extract retained useful activity over three reuse cycles, with isolated yield falling by no more than 5–6% from the fresh-catalyst value, and the calculated green-chemistry metrics (atom economy ≈ 92%, reaction mass efficiency ≈ 85%, process mass intensity ≈ 8.6, E-factor ≈ 7.6 for the model reaction) compared favourably with the conventional NaOH/organic-solvent route and with other reported green protocols (Table 10).

In summary, the standardised hydroethanolic *Acacia concinna* extract/K₂CO₃ system is demonstrated as a mild, aqueous, bio-derived alternative to conventional strong-base, organic-solvent protocols for the Claisen–Schmidt synthesis of chalcones, to a pharmacologically important and structurally distinct scaffold. The optimisation, reusability, characterisation and green-metrics outcomes reported here establish the *Acacia concinna*/K₂CO₃ system as an effective and reproducible green route to substituted chalcones.

Acknowledgements

The authors thank their host institution and department for providing laboratory facilities and instrumentation support (melting-point, FTIR, NMR and HRMS facilities). Kiran D. Gajhans is thankful to Dr. Babasaheb Ambedkar Research and Training Institute, Pune for research fellowship.

References

1. Zhuang, C., Zhang, W., Sheng, C., Zhang, W., Xing, C., Miao, Z. Chalcone: A Privileged Structure in Medicinal Chemistry. *Chem. Rev.* 2017, 117(12), 7762–7810.
2. Synthesis, Reactions and Pharmacological Applications of Chalcones and Their Derivatives: A Mini Review. *J. Chem. Rev.* (jchemrev.com/article_146966.html).
3. Medicinal chemistry perspective of chalcone derivatives as anticancer agents: synthetic strategy, biological activity, and structure–activity relationship. *Mol. Divers.* 2025, <https://doi.org/10.1007/s11030-025-11434-w>.
4. Synthesis and Pharmacological Activities of Chalcone and Its Derivatives Bearing N-Heterocyclic Scaffolds: A Review. *ACS Omega* 2023, <https://doi.org/10.1021/acsomega.3c01035>.
5. Synthesis of Chalcones Derivatives and Their Biological Activities: A Review. PMC9386807, 2022.
6. Journal of Chemical Reviews 2026, Vol. 8, Issue 2, Review Article on chalcone synthesis/reactions/pharmacology (jchemrev.com/article_234581).
7. Mahapatra, D.K., Bharti, S.K., Asati, V. Chalcone scaffolds as anti-infective agents: Structural and molecular target perspectives. *Eur. J. Med. Chem.* 2015, 101, 496–524.
8. Claisen, L., Schmidt, O. Ueber die Einwirkung von Aceton auf Furfurol und auf Bittermandelöl. *Ber. Dtsch. Chem. Ges.* 1881, 14, 2460–2468.
9. Green metrics in chalcone synthesis via Claisen–Schmidt condensation: a critical mini review. *Front. Chem.* 2026, <https://doi.org/10.3389/fchem.2026.1909081>.

10. Chalcone Synthesis by Green Claisen–Schmidt Reaction in Cationic and Nonionic Micellar Media. *J. Org. Chem.* 2025, 90(8), 2915, <https://doi.org/10.1021/acs.joc.4c02616>.
11. Carbon–carbon bond formation in acid deep eutectic solvent: chalcones synthesis via Claisen–Schmidt reaction. *RSC Adv.* 2016, <https://doi.org/10.1039/C6RA04721A>.
12. How Can Deep Eutectic Systems Promote Greener Processes in Medicinal Chemistry and Drug Discovery? *PMC10892015*, 2024.
13. Zhao, Q., Wang, G., Liao, F., Sha, Y., Xu, F., Li, C., Li, Z., Cao, Y. Ionic liquid catalyzed solvent-free synthesis of chalcone and its derivatives under mild conditions. *Chin. J. Chem. Eng.* 2021, 33, 160–166, <https://doi.org/10.1016/j.cjche.2020.09.013>.
14. Chergi, K., Beryanvand, M., Merhi, M. et al. Chitosan-Supported Heterogeneous Catalysts for Base-Mediated Condensation Reactions. *J. Inorg. Organomet. Polym. Mater.* 2024, 34, 735–744, <https://doi.org/10.1007/s10904-023-02867-7>.
15. Mukherjee, A., Sengupta, S. Characterization of Saponin Extracted from *Acacia concinna* (Shikakai or Soap Pod) and Thermodynamic Studies of Its Interaction with Organic Dye Rhodamine B. *Chem. Afr.* 2024, <https://doi.org/10.1007/s42250-024-00950-1>.
16. Todkar, S.S., Chavan, V.V., Kulkarni, A.S. Screening of Secondary Metabolites and Antibacterial Activity of *Acacia concinna*. *Res. J. Microbiol.* 2010, 5(10), 974–979, <https://doi.org/10.17311/jm.2010.974.979>.
17. Komal Khanpara , Renuka , Dr. V. J. Shukla , Harisha C.RA detailed investigation on shikakai (*Acacia concinna* Linn.) fruit. *Jounal of current Pharmaceutical research*, 2012; 9 (1) : 6-10.
18. Antimicrobial Effect of Waterborne Polyurethane Based Cellulose Nanofibril/Silver Nanoparticles Composites and *Acacia concinna* (Willd.) DC Extract (Shikakai). *Polymers* 2024, 16(19), 2683, <https://doi.org/10.3390/polym16192683>.
19. Das, D., Sarangi, A.K., Mohapatra, R.K., Parhi, P.K., Mahal, A., Sahu, R., Kudrat-E-Zahan, Md. Aqueous extract of Shikakai; a green solvent for deoximation reaction: Mechanistic approach from experimental to theoretical. *J. Mol. Liq.* 2020, 309, 113133, <https://doi.org/10.1016/j.molliq.2020.113133>.
20. Mote, K., Pore, S., Rashinkar, G., Kambale, S., Kumbhar, A., Salunkhe, R. *Acacia concinna* pods as a green catalyst for highly efficient synthesis of acylation of amines. *Arch. Appl. Sci. Res.* 2010, 2(3), 74–80.