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An glycerol mediated catalyst free green and sustainable protocol for synthesis of polyhydroquinolines under heating condition

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Abstract
A facile, eco-friendly, and sustainable protocol has been developed for the synthesis of polyhydroquinoline derivatives via a one-pot multicomponent condensation reaction involving various substituted aromatic aldehydes, ethyl acetoacetate, dimedone, and ammonium acetate. Glycerol, a biodegradable and inexpensive by-product of the biodiesel industry, serves as both solvent and catalyst in this transformation. The present methodology offers several noteworthy advantages, including mild reaction conditions, excellent product yields, shorter reaction times, and a straightforward work-up process. Furthermore, the use of a non-toxic, metal-free, and recyclable medium underscores the environmental compatibility of this procedure. The operational simplicity, cost-effectiveness, and elimination of hazardous organic solvents make this protocol an attractive and sustainable alternative for the synthesis of polyhydroquinoline scaffolds.

Keywords: Short reaction time, simple work-up procedure, eco-friendly, metal free catalyst

Introduction
Now a days, green chemistry has become a key importance as it is incorporated in pharmaceutical, food and chemical industries to develop eco-friendly protocol that minimize waste production and help to make alternative way by avoiding use of hazardous chemicals [1-2]. The green reaction is most sustainable approach among all other for synthesis of nitrogen containing biological active heterocyclic compounds as it helps to maintain environment balance to large extent [3-6]. The traditional chemical synthesis of biological active heterocyclic compounds contributes to environment pollution in large extent leads to generation of problems for living system, so green chemistry performs major role with the help of twelve principles to minimize the use and generation of unwanted substances [7]. The green tools include physical and chemical methods such as ball milling, microwave irradiation, photocatalysis, hydrothermal synthesis, magnetic field synthesis, ultrasonication, green solvent, nanomaterials, use of ionic liquid and ecofriendly catalyst are frequently implemented by researchers for synthesis of target molecules.
Multi-component reactions (MCRs) is well famous and crucial tool for design a chemical conversions to synthesis of biological potent heterocyclic moieties in which three or more than starting material added simultaneously to give product with high degree of atom economy, efficiency, sustainability, saving time, minimize cost, environmental friendliness and application in the several of convergent synthesis of multifaceted organic molecules from easy and convenient substrates in a solitary synthetic operation [8-11].
The maximum number of reactions used liquid phase for organic transformation which help to communicate starting materials and initiate reaction towards products side [12]. The use of green solvent should possess various features such as non-toxic, non-inflammable, non-volatile, biodegradable, inexpensive, easily available, safe to handle [13]. A glycerol fulfils all the demands of safety environment as it bears distinctive combination of physical and chemical properties mentioned above by green chemistry principles [14]. Moreover, glycerol is promisingly used for high yield and more selective organic synthesis of pharmaceutically significant scaffolds without use of catalytic reagent under harsh reaction condition of temperature and pressure etc [15].
By considering environmental point of view, use of biodegradable solvent like glycerol shows better results than conventional organic solvents [16]. Glycerol is recognized as one of the

most sustainable, biodegradable, and safe solvents that can also act as a catalyst in various organic transformations. It possesses several advantageous physicochemical properties, including non-toxicity, non-flammability, non-corrosiveness, high polarity, recyclability, a high boiling point, and a large dielectric constant, making it an ideal medium for green synthesis[17-20]. In addition, its high solubility for a wide range of organic and inorganic compounds further enhances its utility, providing an additional advantage of glycerol as a versatile reaction medium in sustainable synthesis [21].

Polyhydroquinoline and their derivatives contain fused, bioactive, highly acknowledged and privileged skeleton of heterocyclic moiety with structurally resembles with Biginelli reaction product exhibit interesting importance in medicinal field [22]. The derivatives of polyhydroquinolines are commercially available as analogue of nicotinamide adenine dinucleotide (NADH) coenzymes are used broadly as calcium channel blockers [23]. The polyhydroquinolines is one of the principal heterocycles which have received noticeable response due to its potent biological activities and used for treatment of cardiovascular diseases including hypertension [24]. These scaffold exhibit distinguished biological activities such as anti-bacterial [25], anti-tubercular [26], anti-cancer [27], anti-oxidant [28] anti-malarial [29], anti-tumor [30], anti-plasmodial [31], anti-proliferative [32], anti-diabetic [33], anti-oxidant [34] anti-cancer [35] properties.

As the polyhydroquinolines exhibit broad range of chemical, industrial and pharmaceutical applications, several protocols for their preparations have been reported in the literature. Arthur Hantzsch in 1881 firstly reported the multicomponent reaction of synthesis of substituted 1,4-dihydropyridines using one pot condensation of formaldehyde, ethyl acetoacetate and ammonia or ammonium acetate in water for a lengthen reaction time. Afterwards significant attempt has been improved in above protocol and was reported using catalyst such as PMO-ICS-PrSO$_3$H [36], Nano-Fe$_2$O$_3$-SO$_3$H [37], SPPN [38], H$_2$N-SO$_3$H [39], Morpholine [40], O$_2$VO-SO$_3$H [41], ZnFe$_2$O$_4$@SiO$_2$@L-methionine-Zr [42], Fe$_3$O$_4$/SiO$_2$-OSO$_3$H [43], Sulfated polyborate [44], Cu (II) complex [45], (BaFe$_{12}$O$_{19}$@GO@Fu) [46], [CEMIM][MSA] [47], [Bmim]OH [48], Cu-IRMOF-3 [49], Uio-66-NH-CH$_2$-CH$_2$N(CH$_2$PO$_2$H$_2$)$_2$ [50], AgI NPs [51], Fe$_{3-x}$TiO$_x$O$_4$-SO$_3$H NPs [52], Cell-Pr-NHSO$_3$H [53], Chiral phosphoric acid catalyst [54], NS-[C$_4$(DABCO-SO$_3$H)$_2$].4Cl [55]. However, these protocols suffered from various drawbacks such as use of long reaction time, harsh reaction conditions, excess use of solvents, difficulty in product isolation. So here we report facile and catalyst free green protocol for synthesis of polyhydroquinolines by using aromatic aldehyde, dimedone, ethyl acetoacetate and ammonium acetate in presence of glycerol at 65 ^{0}C temperature.

Experimental

General

All chemicals, reagents, and solvents used in the present study were of analytical grade and procured from Merck, Loba Chemie, and Avra Laboratories. They were employed without any further purification. Melting points were determined using a Labtronics digital melting/boiling point apparatus and are reported in degrees Celsius (°C) as uncorrected values. Thin-layer chromatography (TLC) was performed on precoated silica gel 60 F254 plates, using 20% ethyl acetate in hexane as the mobile phase. Spots were visualized under ultraviolet light at 254 or 365 nm. Infrared (IR) spectra were recorded on a Shimadzu FT-IR spectrophotometer at Jijamata College of Arts and Science, Bhende, Ahmednagar, and the absorption frequencies (νmax) are expressed in cm^{-1}. The ^{1}H NMR spectra were obtained at Savitribai Phule Pune University, Pune, using a Bruker 400 MHz spectrometer in CDCl$_3$ with tetramethylsilane (TMS) as an internal standard. Chemical shifts (δ) are reported in parts per million (ppm), and coupling constants (J) are given in Hertz (Hz).

The spectral characterization of selected representative compounds is provided below

ethyl 2,7,7-trimethyl-5-oxo-4-phenyl-1,4,5,6,7,8-hexahydroquinoline-3- carboxylate

White Solid; Yield 94%; M.P. 214-216^{0}C; ^{1}H NMR (400 MHz, DMSO-d6): 0.86 (s, 3H), 1.02 (s, 3H), 1.14 (t, J= 7.2 Hz, 3H), 1.99 (d, J= 16.0 Hz, 1H), 2.18 (d, J= 16.0 Hz, 1H), 2.30 (d, J= 16.4 Hz, 1H), 2.30 (s, 3H), 2.43 (d, J= 16.8 Hz, 1H), 3.99 (q, J= 7.2 Hz, 2H), 4.87 (s, 1H), 7.06- 7.10 (m, 1H), 7.15-7.21 (m, 4H), 9.08 (s, 1H); ^{13}C NMR (100 MHz, DMSO-d6): 14.6, 18.7, 26.9, 29.6, 32.6, 36.3, 50.7, 59.5, 104.1, 110.4, 126.1, 127.9, 128.2, 145.4, 148.1, 150.0, 167.3, 194.7; MS m/z (ESI); 340.9 (M+H)$^+$

ethyl 2,7,7-trimethyl-4-(3-nitrophenyl)-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3- carboxylate

White Solid; Yield 93%; M.P. 172-175^{0}C; IR : 687, 1153, 1207, 1355, 1478, 1532, 1610, 1704, 2946, 3078, 3202, 3280 cm^{-1} ; ^{1}H NMR (400 MHz, CDCl$_3$): δ 0.92 (s, 3H), 1.07 (s, 3H), 1.19 (t, J=7.1 Hz, 3H), 2.04-2.32 (m, 4H), 2.37 (s, 3H), 4.05 (q, J=7.1 Hz, 2H), 5.15 (s, 1H), 6.67 (s, 1H), 7.37 (t, J=7.9 Hz, 1H), 7.71 (d, J=7.7 Hz, 1H), 7.97 (dd, J= 8.1, 0.7 Hz, 1H), 8.11 (d, J=1.6 Hz, 1H); ^{13}C NMR (100 MHz, CDCl$_3$): 14.30, 19.49, 27.19, 29.48, 32.86, 37.11, 40.98, 50.67, 60.19, 105.19, 111.20, 121.39, 122.98, 128.72, 134.89, 144.72, 148.34, 149.36, 149.45, 167.06, 195.71; MS m/z (ESI); 385 (M+H)$^+$

ethyl 4-(4-methoxyphenyl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate

White Solid; Yield 82%; M.P. 255-256^{0}C; IR: 757, 842, 1037, 1160, 1214, 1378, 1501, 1602, 1696, 2945, 3070, 3202, 3280 cm^{-1} ; ^{1}H NMR (500 MHz, CDCl3): δ 0.86 (s, 3H), 1.00 (s, 3H), 1.13 (t, J=7.1 Hz, 3H), 1.95-2.38 (m, 4H), 2.29 (s, 3H), 3.66 (s, 3H), 3.97 (q, J=7.1 Hz, 2H), 4.78 (s, 1H), 6.74 (d, J=7.9 Hz, 2H), 7.05 (d, J=7.7 Hz, 2H), 9.00 (s, NH); ^{13}C NMR (100 MHz, CDCl$_3$): 14.64, 18.73, 26.97, 29.61, 32.60, 40.49, 40.58, 50.74, 55.31, 59.46, 104.37, 110.65, 113.55, 128.85, 140.47, 145.08, 149.70, 157.74, 167.39, 194.73; MS m/z (ESI); 370.20 (M+H)$^+$

ethyl 2,7,7-trimethyl-4-(4-nitrophenyl)-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3- carboxylate

White Solid; Yield 82%; M.P. 210-212^{0}C; IR : 687, 1153, 1207, 1355, 1478, 1532, 1610, 1704, 2946, 3078, 3202, 3280 cm^{-1} ; ^{1}H NMR (400 MHz, DMSO-d6): δ 0.83 (s, 3H), 1.02 (s, 3H), 1.12 (t, J=6.8 Hz, 3H), 1.99 (d, J= 16.0 Hz, 1H), 2.20 (d, J=16.4 Hz, 1H), 2.29-2.33 (m, 4H), 2.45 (d, J= 16.8 Hz, 1H), 3.97 (q, J= 7.2 Hz, 2H), 4.98 (s, 1H), 7.43 (d, J= 8.8 Hz, 2H), 8.11 (d, J= 8.8 Hz, 2H), 9.26 (s, NH); ^{13}C NMR (100 MHz, DMSO-d6): 14.5, 18.8, 26.8, 29.4, 32.6, 37.1, 50.5, 59.7, 102.8, 109.5, 123.6, 129.2, 146.1, 146.6, 150.5, 155.4, 166.8, 194.7; MS m/z (ESI); 384.9 (M+H)$^+$ ethyl 4-(4-bromophenyl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate

White Solid; Yield: 92%; M.P. 253-256^{0}C; IR : 532, 734, 851, 1013, 1067, 1206, 1377, 1486, 1595, 1704, 2961, 3078, 3202, 3280 cm^{-1} ; ^{1}H NMR (400 MHz, CDCl$_3$): δ 0.83 (s, 3H), 1.00 (s, 3H), 1.11 (t, J=7.1 Hz, 3H), 1.95-2.39 (m, 4H), 2.28 (s, 3H), 3.96 (q, J=7.1 Hz, 2H), 4.81 (s, 1H), 7.10 (d, J=7.7 Hz, 2H), 7.37 (d, J=7.7 Hz, 2H), 9.10 (s, 1H); ^{13}C NMR (100 MHz, CDCl$_3$): δ 14.59, 18.76, 26.92, 29.54, 32.61, 40.49, 40.58, 50.63, 59.59, 103.51, 110.06, 119.17, 130.22, 131.08, 145.88, 147.45, 150.10, 167.11, 194.73; MS m/z

(ESI); 419.1 $(M+H)^+$ ethyl 4-(4-methylphenyl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8- hexahydroquinoline-3-carboxylate

White Solid; Yield: 95%; M.P. 280-283^{0}C; ^{1}H NMR (400 MHz, DMSO-d6): δ 0.86 (s, 3H), 1.02 (s, 3H), 1.15 (t, J= 7.2 Hz, 3H), 1.98 (d, J= 16.0 Hz, 1H), 2.15-2.21 (m, 4H), 2.29-2.31 (m, 4H), 2.43 (d, J= 16.8 Hz, 1H), 3.98 (q, J= 7.2 Hz, 2H), 4.82 (s, 1H), 6.99 (d, J= 7.6 Hz, 2H), 7.04 (d, J= 7.6 Hz, 2H), 9.04 (s, 1H); ^{13}C NMR (100 MHz, DMSO-d6): δ 14.7, 18.8, 21.1, 26.9, 29.7, 32.6, 35.9, 50.8, 59.5, 104.3, 110.6, 127.9, 128.8, 135.0, 145.3, 149.8, 167.4, 194.7 MS m/z (ESI); 354.9.1 $(M+H)^+$

General Procedure for synthesis of Polyhydroquinoline Derivatives

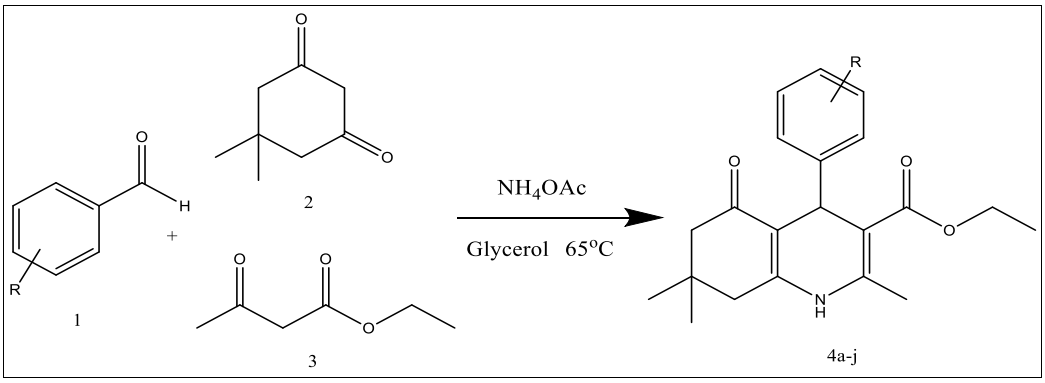


Fig 1: Schematic representation of polyhydroquinoline synthesis via multicomponent reaction.

In the present study, a mixture of aromatic aldehyde (1.0 mmol), ethyl acetoacetate (1.5 mmol), dimedone (1.0 mmol), and ammonium acetate (1.5 mmol) was added to glycerol (10 mL) in a reaction flask. The mixture was stirred at 65 ^{0}C for the required time period. The progress of the reaction was periodically monitored by thin-layer chromatography (TLC). Upon completion, as indicated by TLC, the reaction mixture was allowed to cool to room temperature, followed by the addition of hot water. The resulting crude product was extracted with ethyl acetate (3 × 10 mL) and the organic layer was dried over anhydrous sodium sulfate. The solvent was then evaporated under reduced pressure to afford the crude polyhydroquinoline derivatives, which were purified by recrystallization from ethanol. The separated glycerol from the aqueous layer was recovered and reused for up to four consecutive reaction cycles without significant loss of activity.

Results and Discussions

In order to investigate the feasibility of the strategy, we choose one-pot reaction of 4-nitro benzaldehyde (1.0 mmol), ethyl acetoacetate (1.5 mmol), dimedone (1.0 mmol) and ammonium acetate (1.5 mmol) as a model starting material using different solvents such as methanol, ethanol, acetonitrile, isopropanol, and glycerol was screened in catalyst free reaction and results were summarized in Table 1. In presence of glycerol, desired product was obtained in 82 % yield at 50^{o}C, so further increase in temperature up to 65^{o}C, significant rise in yield was observed up to 94 % in 60 min. indicated in Table 1 Entry 7. So further derivatives of polyhydroquinolines was prepared at 65^{o}C temperature and results are included in 2.

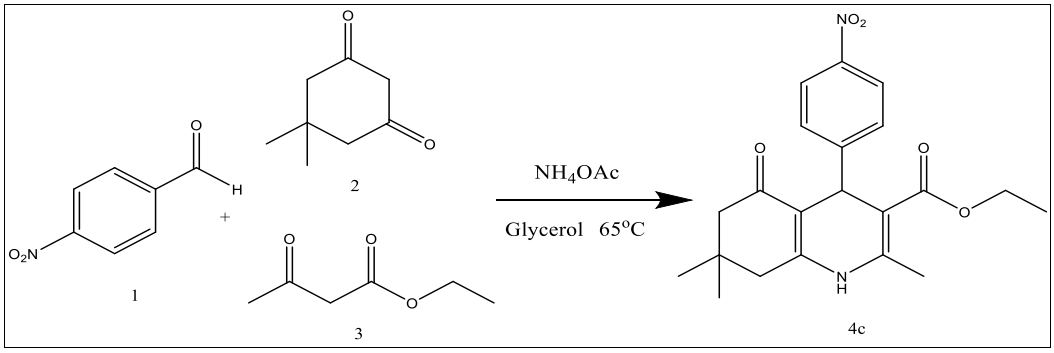


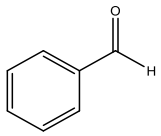
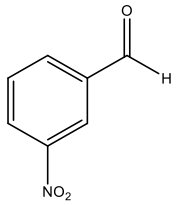
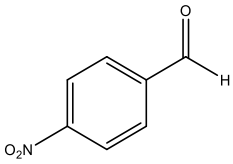
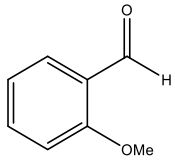
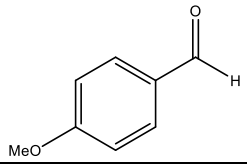
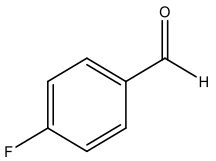
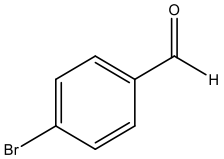
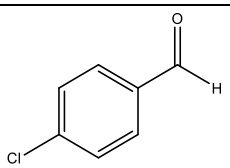
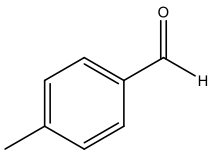
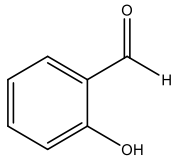
Fig 2: Schematic representation of polyhydroquinoline derivative synthesis using glycerol medium.

Table 1: Comparison with different solvent for the synthesis of 4c[a]

Entry	Solvent	Condition (^{o}C)	[b]Time (min.)	[c]Yield (%)
1	-	50	180	trace
2	Methanol	50	180	32
3	Ethanol	50	180	45
4	Acetonitrile	50	180	52
5	Isopropanol	50	180	56
6	Glycerol	50	90	82
7	**Glycerol**	**65**	**60**	**94**

[a]reaction condition: 4-nitro benzaldehyde (1 mmol), ethyl acetoacetate (1.5 mmol), dimedone (1.0 mmol) and ammonium acetate (1.5 mmol) was taken at different solvent reaction condition. [b]Time in min. [c]Isolated yield

Table 2: Synthesis of Polyhydroquinolines using aromatic aldehyde, ethyl acetoacetate, dimedone and ammonium acetate in presence of glycerol as solvent cum catalyst[a].

Entry	Aldehyde	[b]Time in min.	[c]Yield (%)	Melting point ºC
4a		60	94	214-216
4b		50	93	172-175
4c		60	94	210-212
4d		65	88	238-240
4e		60	90	255-256
4f		65	88	185-187
4g		65	90	253-256
4h		65	92	239-241
4i		60	90	280-283
4j		75	90	196-197

[a]reaction condition: aromatic aldehyde (1.0 mmol), ethyl acetoacetate (1.5 mmol), dimedone (1.0 mmol) and ammonium acetate (1.5 mmol), at 65ºC temperature in glycerol. [b]Time in min. [c]Isolated yield.

The reusability of glycerol as a catalyst-cum-solvent was evaluated using a model reaction comprising 4-nitrobenzaldehyde (1.0 mmol), ethyl acetoacetate (1.5 mmol), dimedone (1.0 mmol), and ammonium acetate (1.5 mmol) in glycerol (10 mL) at 65 ^{0}C. Upon completion, the reaction mixture was extracted with ethyl acetate, and the glycerol layer was separated from the aqueous phase. The recovered glycerol was subsequently reused for four consecutive reaction cycles without any significant loss in product yield, as summarized in Table 3.

Table 3: Reusability of glycerol

Runs	Fresh	1	2	3	4
Time (min.)	60	68	70	72	70
[b]Yield (%)	94	92	88	90	86

[a]reaction condition: 4-nitro benzaldehyde (1.0 mmol), ethyl acetoacetate (1.5 mmol), dimedone (1.0 mmol) and ammonium acetate (1.5 mmol), 65°C in glycerol solvent. [b]Isolated yield.

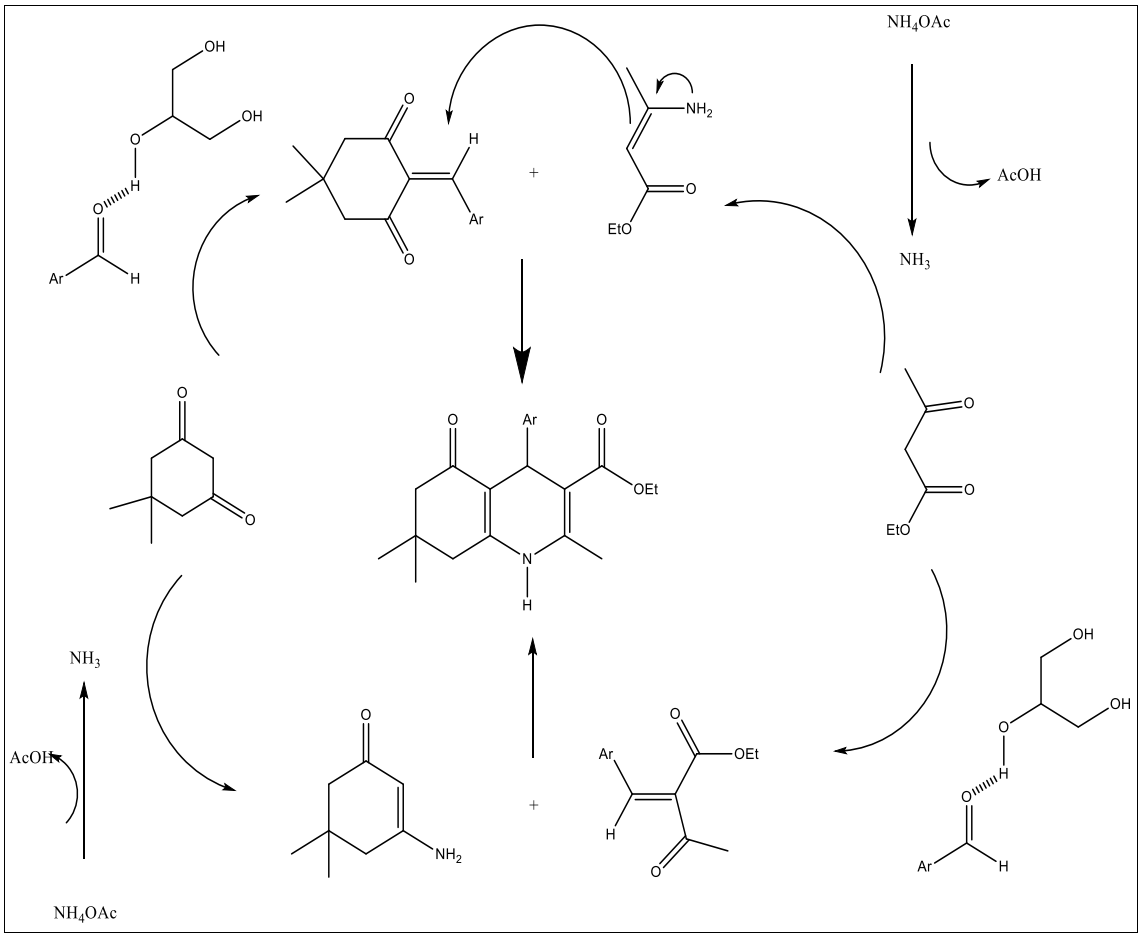


Fig 3: Putative mechanism for formation of polyhydroquinoline derivatives

Conclusions

In conclusion, a simple, green, and sustainable protocol has been successfully developed for the synthesis of polyhydroquinoline derivatives via the condensation of various substituted aromatic aldehydes, ethyl acetoacetate, dimedone, and ammonium acetate using glycerol at 65 ^{0}C. Glycerol plays a dual role in this transformation, functioning both as an efficient solvent and as a catalyst. Its strong hydrogen-bonding capability enhances the activation of electrophilic substrates, thereby promoting the reaction. The present methodology offers several advantages, including mild reaction conditions, excellent product yields, short reaction times, an easy work-up process, and the use of an environmentally benign, metal-free, and recyclable medium. These features make this protocol a promising and convenient approach for the synthesis of biologically active heterocyclic compounds.

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