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PUMICE@SO_3H CATALYZED ULTRASOUND MEDIATED SYNTHESIS OF POLYHYDROQUINOLINE DERIVATIVES.

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Abstract:
A sustainable and convenient protocol is developed for the synthesis of polyhydroquinoline derivatives under ultrasound irradiation at 45°C in the presence of pumice anchored sulfonic acid (Pumice@SO_3H) as a recoverable catalyst. These polyhydroquinolines were synthesized from aldehydes, dimedone, ethylacetoacetate and ammonium acetate by Hantzsch reaction. The attractive features of the present protocol are green approach, good yield, recovery of catalyst, easy work-up procedure and simple purification of product whereas the catalyst offers simple preparation, high catalytic activity, inexpensive, easy to use, recyclability and stability.

Keywords:
Pumice@SO_3H, polyhydroquinolines, ultrasound irradiation, dimedone, etc.

Introduction:

Pumice stone obtained due to volcanic eruptions has many advantages such as abundance, availability, large surface area, low cost, non-homogeneous nature, and excellent stability. Also due to the remarkable properties such as high porosity and high adsorption capacities have gained much interest in the field of catalysis. In recent years, the volcanic pumice converted into variety of supported active catalytic materials such as pumice@SO_3H[i, ii], Pd–Ag catalysts supported on pumice[iii], Pumice-modified cellulose fiber[iv], Volcanic based hybrid nanocomposite[v], Pumice supported Pd catalyst[vi], Immobilization of TiO_2 on pumice stone[vii], iron-coated pumice[viii, ix], pumice-supported Pd–Cu catalysts[x], etc.

Multi-component reactions (MCRs) are a constructive approach to synthesize heterocyclic compounds with diverse structures. In MCRs, more than two components reacts together in single step to produce a targeted heterocyclic system without isolation of any intermediate. Due to this, requires short time, reduce energy requirement, reduce quantity of precursors, and are useful to increase atom economy. The Hantzsch reaction is one of the most important examples of multicomponent reaction which is used for synthesis of polyhydroquinoline derivatives[xi, xii]. The polyhydroquinoline derivatives is of great attention due to their various activities such as anti-cancer, anti-diabetic, anti-hypertensive, anti-inflammatory, anti-microbial, anti-

tubercular, anti-tumor, bronchodilator, calcium channel blockers, cardiovascular agents, geroprotective, hepatoprotective, neurotropic, and vasodilator[xiii-xxii] etc. These versatile activities have encouraged researchers to design sustainable and convenient catalytic materials for the synthesis of heterocyclic compounds containing polyhydroquinoline moiety. Some illustrations of drugs with 1,4-dihydropyridine framework are outlined in **Fig. 1**.

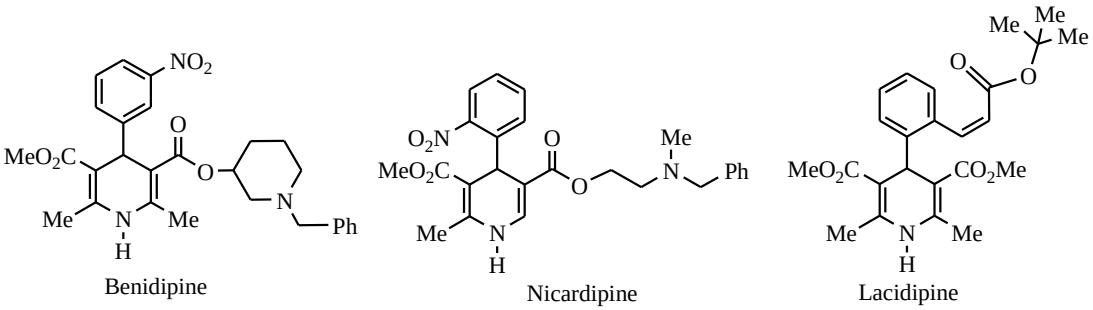


Fig.1. some drugs containing 1,4-dihydropyridine framework

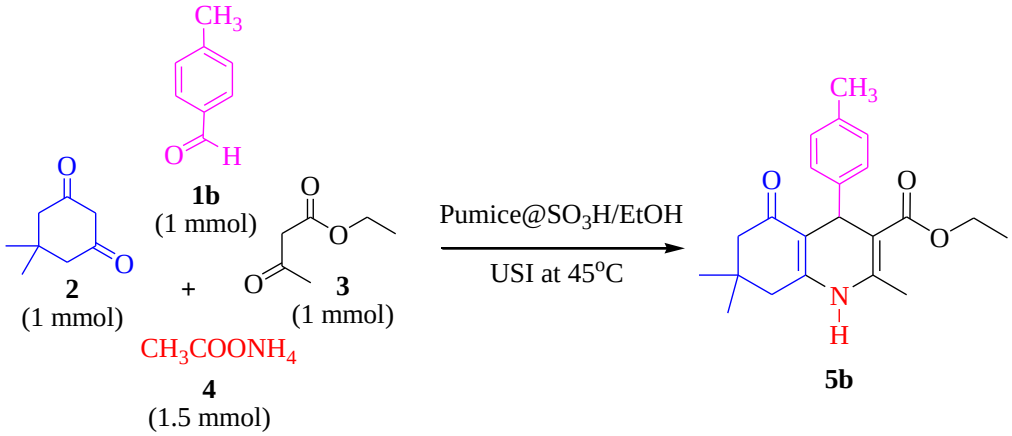
Recently, numerous protocols have been developed for the synthesis of polyhydroquinolines from aromatic aldehyde, dimedone, ethylacetoacetate and ammonium acetate such as nano-materials[xxiii], metal oxide supported materials[xxiv], magnetic materials[xxv], ionic liquids[xxvi], amino acids[xxvii], solar thermal energy[xxviii], Zeolite[xxix], microwave[xxx], and ultrasound[xxxi] etc. Also various bronsted acidic catalyst are used such as Fe_3O_4/SiO_2-OSO_3H[xxxii], silica sulfuric acid[xxxiii], nicotinic acid[xxxiv], Acetic acid[xxxv], Aluminized polyborate[xxxvi], PPA-SiO_2[xxxvii], SBA-15/SO_3H[xxxviii], SBA-15@Glycine[xxxix], PMO-ICS-PrSO_3H[xl], BINOL-phosphoric acid[xli], Carbon-based Solid acid (CBSA)[xlii], COF-SO_3H [xliii], Fe_3O_4@FSM-16-SO_3H [xliv], *p*-TSA[xlv], [MSAIM]HSO_4[xlvi], [Pyridine-SO_3H]Cl[xlvii], Caffeine-H_3PO_4[xlviii], ascorbic acid[xlix], Fe_3O_4@PEO-SO_3H[l], etc.

The ultrasound (US) assisted synthesis is well developed method used for the synthesis of variety of heterocyclic compounds. It proceeds through the development and adiabatic collapse of the transient cavitations bubble. It is used as a green approach that helping to reduce high energy requirements. The US approach provides smooth and cleaner reactions procedure with increasing yields in presence of various catalytic processes [li-lvii].

In continuation of our environmentally benign work [lviii-lxii] and on the application of pumice@SO_3H catalysts[i, ii], here we report a convenient green approach for one-pot synthesis of polyhydroquinolines in the presence pumice anchored sulfonic acid as a bronsted acidic catalyst with good catalytic activity and recyclability.

Results and Discussion:

In order to choose the better reaction condition a model reaction (**Scheme 1**) of *p*-methyl benzaldehyde, dimedone, ethyl acetoacetate and ammonium acetate was carried out in presence of catalyst pumice@SO_3H with and without catalyst and solvent. The reaction did not proceed to any extent in absence of catalyst with and without solvent during stirring at room temperature (**Table 1, Entry 1-3**). Also the negative result was obtained with pumice@SO_3H catalyst at room temperature in presence water and ethanol as well as without solvent under ultrasound irradiation (**Table 1, Entry 4-6**). The reaction proceeds smoothly with catalyst pumice@SO_3H in presence of ethanol as solvent at 45^{o}C under ultrasound irradiation with excellent yield (**Table 1, Entry 7**).



Scheme 1. Model reaction for synthesis of Polyhydroquinoline (**5b**) derivative

Table 1: Optimization of reaction condition for the synthesis of polyhydroquinoline (**5b**)

Entry	Catalyst / Solvent	Reaction Condition	Time in hrs.	Yield [b] in %
1	90 mg pumice@SO_3H / Solvent free	Grinding	0.5	No reaction (NR)
2	90 mg pumice@SO_3H / H_2O	Stirring at RT	3	NR
3	90 mg pumice@SO_3H / EtOH	Stirring at RT	3	NR
4	90 mg pumice@SO_3H / H_2O	USI at RT	3	NR
5	90 mg pumice@SO_3H / H_2O	USI at 45°C	3	NR
6	90 mg pumice@SO_3H / EtOH	USI at RT	3	Trace
7	90 mg pumice@SO_3H / EtOH	USI at 45°C	1.5	80

[a]Reaction condition: **1b** (0.120gm, 1mmol), **2** (0.140gm, 1mmol), **3** (0.130gm, 1mmol), **4** (0.107gm, 1.5mmol), pumice@SO_3H (90 mg), [b]Isolated Yield

Table 2: Optimization of quantity of catalyst for the synthesis of polyhydroquinoline (**4b**)

Entry	Pumice@SO_3H Catalyst (mg)	Time (hrs)	Yield[b] (%)
1	40	2	25
2	60	2	45
3	80	2	70
4	90	1.5	80
5	90	1.5	80

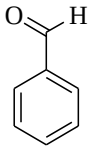
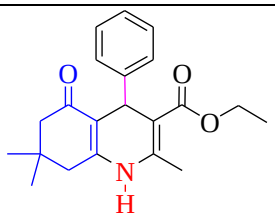
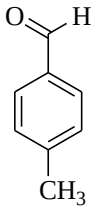
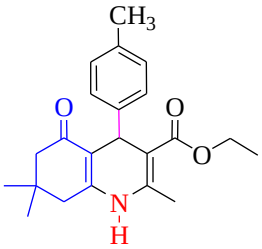
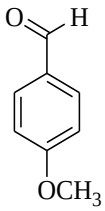
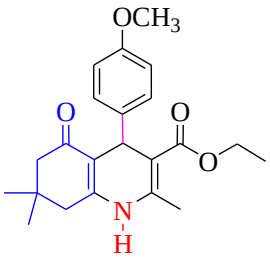
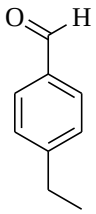
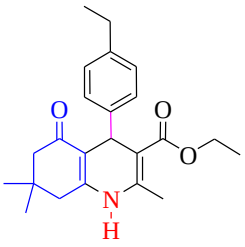
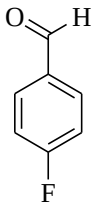
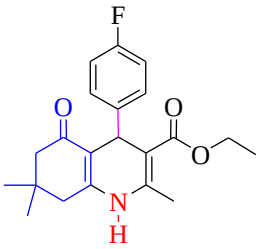
[a]Reaction condition: **1b** (0.120gm, 1mmol), **2** (0.140gm, 1mmol), **3** (0.130gm, 1mmol), **4** (0.107gm, 1.5mmol), USI at 45°C, [b] Isolated Yield

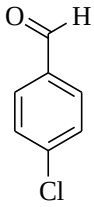
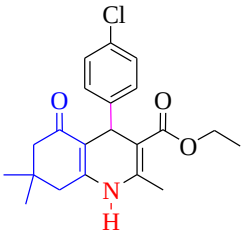
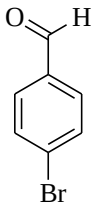
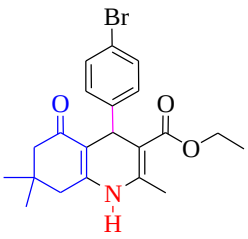
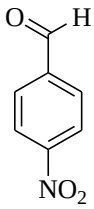
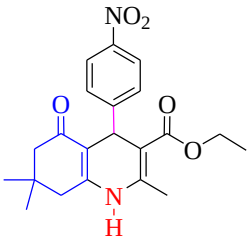
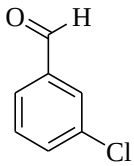
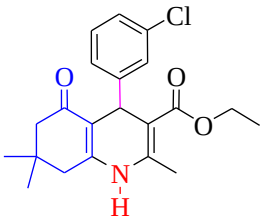
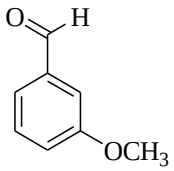
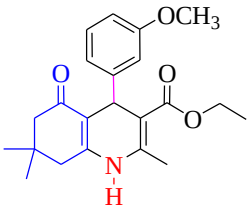
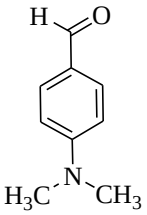
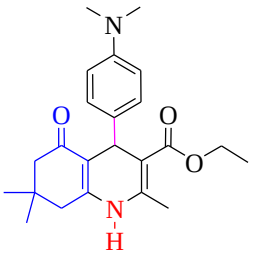
The model reaction was then studied for different amount of catalyst to optimize the amount of catalyst required (**Table 2**). It was found that further increase in the amount of catalyst, there was no significant improvement in the yield of the product.

This outcome enhanced our attention to study the scope, generality and relevance of this protocol for the synthesis of Polyhydroquinoline (**5a-k**) derivatives. The series of Polyhydroquinoline were synthesized using diverse aromatic aldehydes under above optimized

conditions with good yield (74-86%) as mentioned in **Table 3**. The protocol worked very well with aldehydes containing electron deficient and electron rich substituent.

Table 3: Data of synthesized Polyhydroquinoline (**5a-k**) derivatives

Entry	Aldehyde	Product	Time (hrs)	Yield (%)	M.P. (°C)	
					Observed	Reported
1		 **5a**	1.5	85	214-216	217–219[35]
2		 **5b**	1.5	80	252-256	260–262[35]
3		 **5c**	2.0	78	257-260	258–260[35]
4		 **5d**	1.5	80	220-224	--
5		 **5e**	1.5	79	182-184	185–186[35]

6		 **5f**	2.0	82	240-242	245–246[35]
7		 **5g**	2.0	78	250-252	254–256[35]
8		 **5h**	2.0	75	238-240	240–242[35]
9		 **5i**	1.5	80	204-206	205–206[35]
10		 **5j**	1.5	76	192-194	198–200[35]
11		 **5k**	1.5	80	236-238	228–231[29]

[a]Reaction condition: **1a-k** (0.120gm, 1mmol), **2** (0.140gm, 1mmol), **3** (0.130gm, 1mmol), **4** (0.107gm, 1.5mmol), USI at 45°C

After the completion of the reaction, the catalyst used has been recovered by heating the reaction mixture up to the boiling. The resultant hot solution was filtered at hot condition to separate the catalyst. The recovered catalyst was washed with dichloromethane 2-3 times and dried to reuse. The recycled catalyst was reused under the optimal conditions in three cycles of the similar transformation (**Fig. 2**). The formation of Pumice@SO_3H catalyst was proved by spectral studies such as FT-IR, XRD, SEM, TEM and EDS etc. which are reported in our previous work[i]. Here the evidences of recyclability study are provided. The FT-IR, XRD and EDS spectra of the recycled pumice@SO_3H catalyst after third cycle did not show any significant change in catalytic activity.

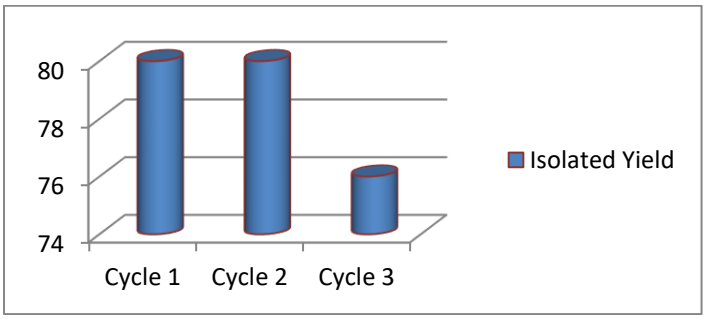


Fig. 2. Reusability of the pumice@SO_3H for the synthesis of Polyhydroquinoline (**4b**)

In the FT-IR spectrum of the recycled pumice@SO_3H (**Fig. 3**), the broad band at 3414.35 cm^{-1} is appeared due to O-H group in sulfonic acid. Also the important bands at 1637.32 cm^{-1} and 1111.05 cm^{-1} are appeared due to the S=O and Si–O–Si respectively. These significant bands indicate that, the recovery of -SO_3H group in the recycled catalyst.

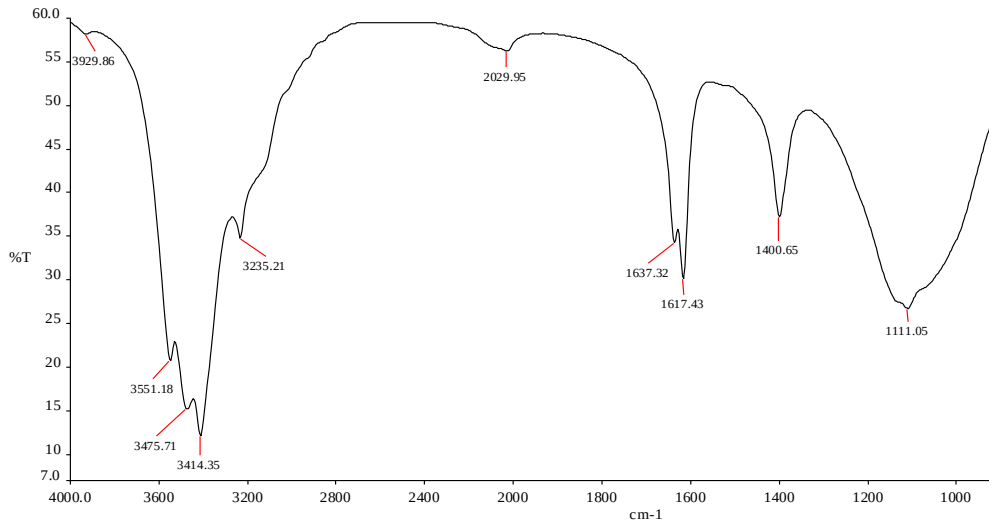


Fig. 3. IR Spectrum of Pumice@SO_3H catalyst

The nature of XRD (**Fig. 4**) and EDS (**Fig. 5**) of recycled catalyst was precisely matched with the reported catalyst. It showed that, the recycled catalyst did not show any variation in composition.

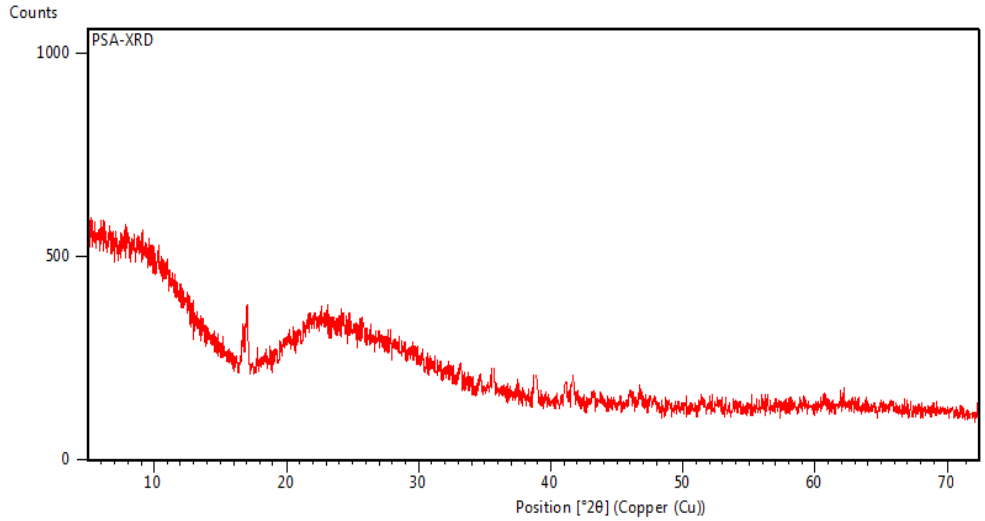


Fig.4. XRD of Pumice@SO_3H catalyst

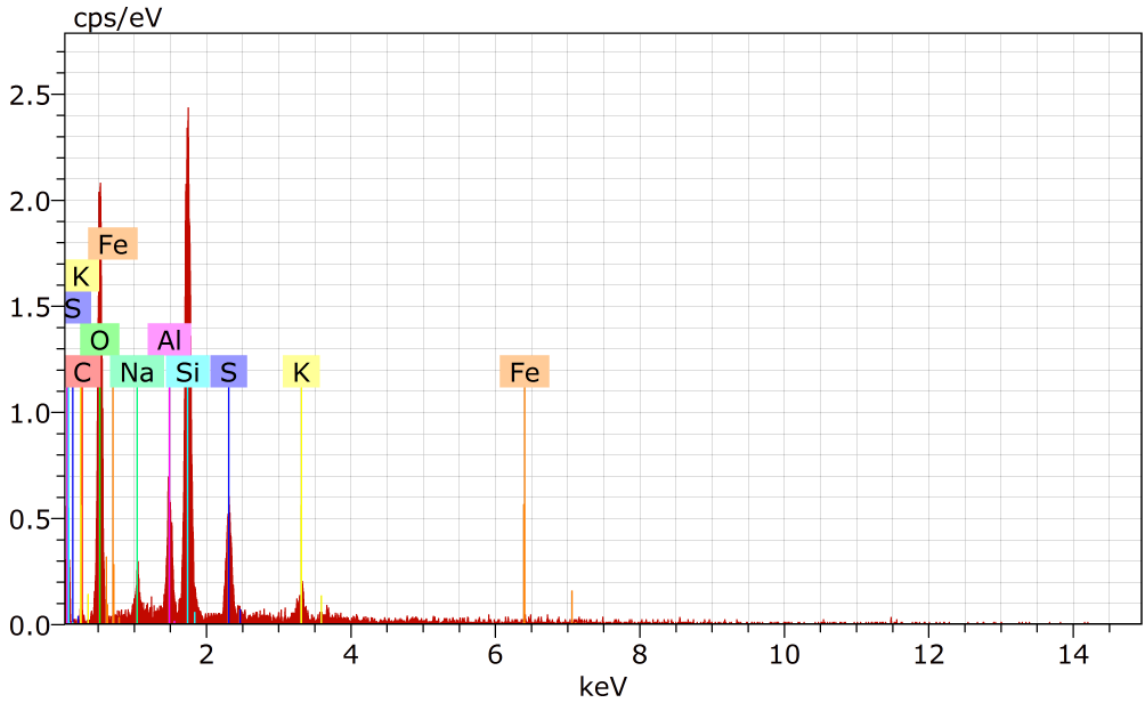


Fig.5. EDS of Pumice@SO_3H catalyst

The comparative study of different protocols for synthesis of polyhydroquinolene derivatives is illustrated in **Table 4**. While the plausible mechanism involved in Pumice@SO_3H promoted synthesis of polyhydroquinolines is shown in **Scheme 4.**

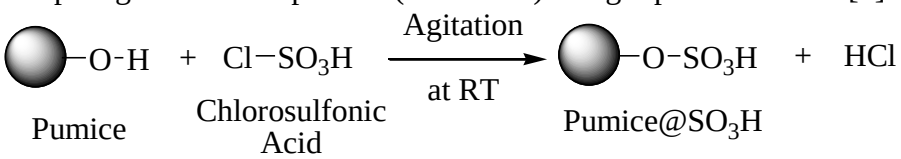
Table 4: Comparative study of different protocols for synthesis of polyhydroquinolene (**5b**)

Entry	Catalyst	Reaction Condition	Quantity of Catalyst in gm	Time in min	Yield (%)	Reference
1	Silica Sulfuric acid	Solvent free/60^oC	0.080	50	92	33

2	Nicotinic acid	Solvent free/80°C	0.1	07	92	34
3	PPA-SiO_2	Solvent free/80°C	0.030	60	90	37
4	PMO-ICS-PrSO_3H	Reflux/EtOH	0.020	20	95	40
5	CBSA	Solvent free/90°C	0.020	35	88	42
6	COF-SO_3H	Solvent free/90°C	0.020	10	95	43
7	Pumice@SO_3H	EtOH/USI, 45°C	0.090	90	80	Present work

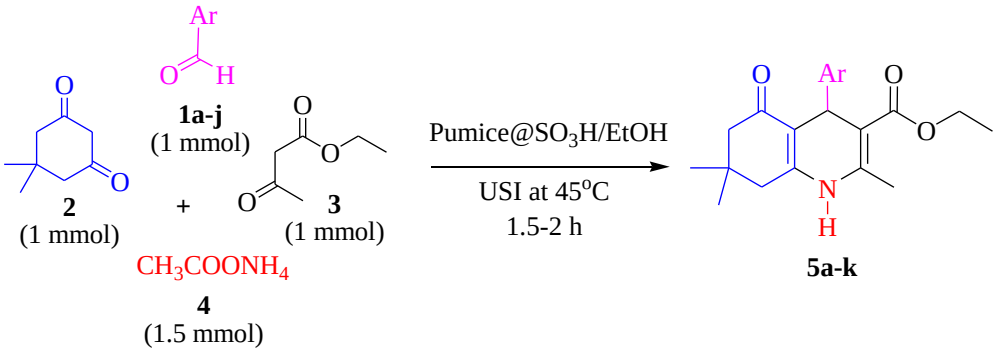
Experimental:
Melting points were recorded in an open capillary and are uncorrected. Infra Red spectra were recorded on a Perkin-Elmer FTIR spectrophotometer. The ^{1}H-NMR and ^{13}C-NMR spectra were recorded on a BRUCKER AVANCE NEO 500MHz NMR Spectrometer in $CDCl_3$ using Tetramethyl silane as a reference compound. Mass spectra were recorded on a Finnigan Mass spectrometer. TLC was carried out by Al-plates pre-coated with silica gel to check the purity of the compounds.

Preparation of pumice anchored sulfonic acid (pumice@SO_3H) catalyst
In the present work, the catalyst pumice anchored sulfonic acid (pumice@SO_3H) has been prepared by simple agitation from pumice (**Scheme 2**) using reported method [1].



Scheme 2: Preparation of pumice anchored sulfonic acid (pumice@SO_3H) catalyst

General procedure for the synthesis of polyhydroquinoline derivatives (5a-k)
A mixture of aldehyde **1** (1 mmol), 5,5-dimethylcyclohexane-1,3-dione **2** (1mmol), ethyl acetoacetate **3** (1 mmol), ammonium acetate **4** (1.5 mmol) and 90 mg of pumice based sulfonic acid was taken in a 100 mL round bottom flask containing 15 mL of ethyl alcohol. The resulting reaction mixture was subjected for ultrasound irradiation at 45°C temperature for appropriate time (**Scheme 3**). The progress of the reaction was studied using TLC. After the completion, the reaction mixture was heated up to the boiling. The resultant hot solution was filtered at hot condition to separate the catalyst. The recovered catalyst was washed with dichloromethane 2-3 times and dried to reuse. After the separation of catalyst, cool the mother liquor, the solid polyhydroquinoline thus obtained. It was dried and in some cases it was purified by recrystallization using hot ethanol.



Scheme 3: Synthesis of Polyhydroquinoline (**5a-k**) derivatives

Discussion of Spectra:

5b: ethyl 1,4,5,6,7,8-hexahydro-2,7,7-trimethyl-5-oxo-4-*p*-tolylquinoline-3-carboxylate

^{1}H NMR (500 MHz, $CDCl_3$): 0.93 (s, 3H, -CH_3), 1.05 (s, 3H, -CH_3), 1.21 (t, 3H, -CH_3), 2.20 (s, 3H, -CH_3), 2.12-2.24 (m, 4H, -CH_2-x2), 2.31 (s, 3H, -CH_3), 4.06 (q, 2H, -OCH_2-), 5.01 (s, 1H, -CH-), 6.66 (s, 1H, NH), 6.99 (d, 2H, *J*=8Hz, Ar-H), 7.18 (d, 2H, *J*=8Hz, Ar-H); ^{13}C NMR (125 MHz, $CDCl_3$): 195.75, 167.58, 148.79, 144.27, 143.56, 135.38, 128.60, 127.87, 112.05, 106.14, 59.78, 50.81, 40.91, 36.14, 32.67, 29.45, 27.19, 21.04, 19.26, 14.24; MS (ESI) : m/z = 354.2110 [M+H].

5c: ethyl 1,4,5,6,7,8-hexahydro-4-(4-methoxyphenyl)-2,7,7-trimethyl-5-oxoquinoline-3-carboxylate

^{1}H NMR (500 MHz, $CDCl_3$): 0.93 (s, 3H, -CH_3), 1.06 (s, 3H, -CH_3), 1.20 (t, 3H, -CH_3), 2.13-2.30 (m, 4H, -CH_2-x2), 2.35 (s, 3H, -CH_3), 3.73 (s, 3H, -OCH_3), 4.07 (q, 2H, -OCH_2-), 4.99 (s, 1H, -CH-), 6.26 (s, 1H, NH), 6.73 (m, 2H, Ar-H), 7.20 (m, 2H, Ar-H); MS (ESI) : m/z = 370.2005 [M+H].

5d: ethyl 4-(4-ethylphenyl)-1,4,5,6,7,8-hexahydro-2,7,7-trimethyl-5-oxoquinoline-3-carboxylate

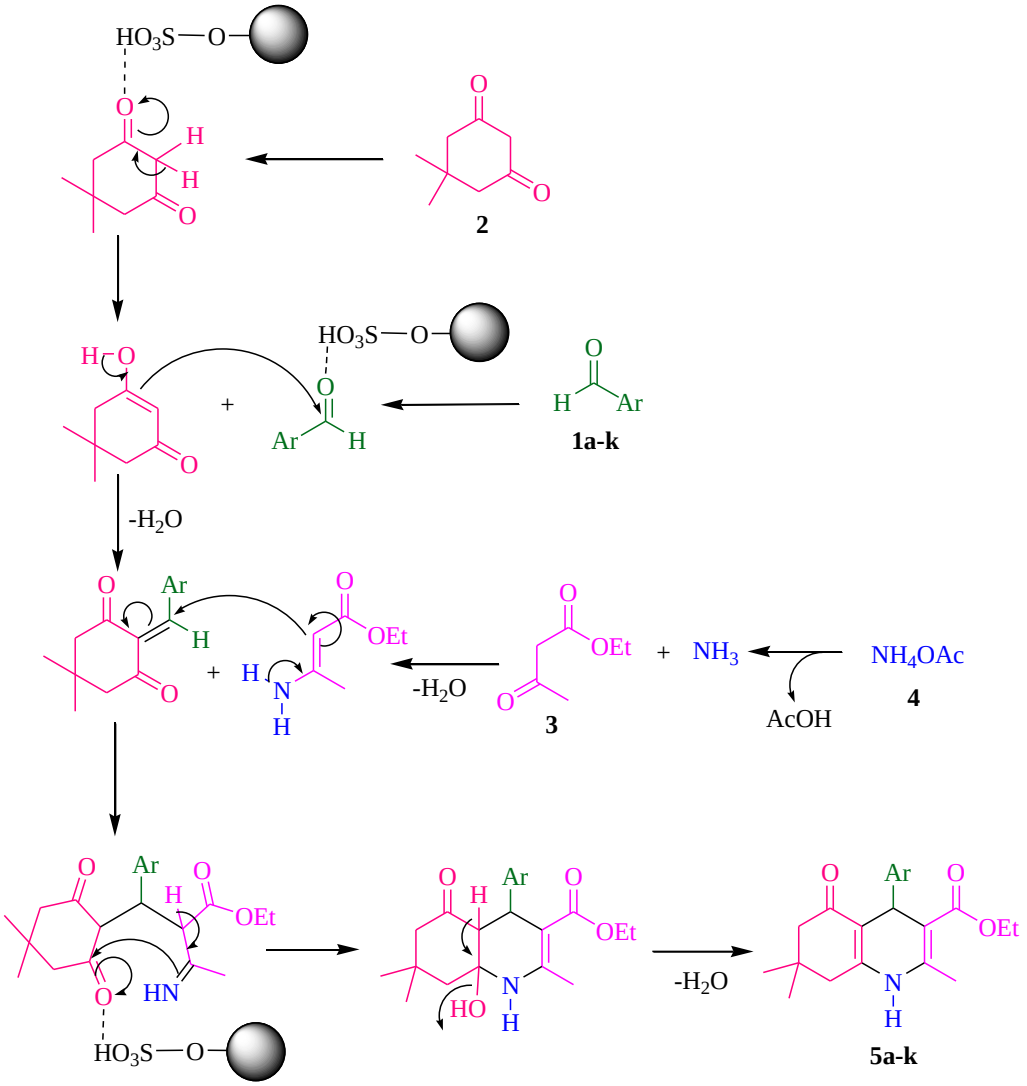
^{1}H NMR (500 MHz, $CDCl_3$): 0.95 (s, 3H, -CH_3), 1.06 (s, 3H, -CH_3), 1.17 (t, 3H, -CH_3), 1.21 (t, 3H, -CH_3), 2.13-2.29 (m, 4H, -CH_2-x2), 2.32 (s, 3H, -CH_3), 2.55 (q, 2H, -CH_2-), 4.06 (q, 2H, -OCH_2-), 5.02 (s, 1H, -CH-), 6.41 (s, 1H, NH), 7.01 (d, 2H, *J*=8Hz, Ar-H), 7.19 (d, 2H, *J*=8Hz, Ar-H); ^{13}C NMR (125 MHz, $CDCl_3$): 195.69, 167.58, 148.49, 144.39, 143.37, 141.68, 127.87, 127.35, 112.16, 106.25, 59.79, 50.79, 41.03, 36.10, 32.71, 29.41, 28.40, 27.28, 19.32, 15.35, 14.23.

5f: ethyl 4-(4-chlorophenyl)-1,4,5,6,7,8-hexahydro-2,7,7-trimethyl-5-oxoquinoline-3-carboxylate

^{1}H NMR (500 MHz, $CDCl_3$): 0.92 (s, 3H, -CH_3), 1.07 (s, 3H, -CH_3), 1.18 (t, 3H, -CH_3), 2.13-2.32 (m, 4H, -CH_2-x2), 2.36 (s, 3H, -CH_3), 4.05 (q, 2H, -OCH_2-), 5.02 (s, 1H, -CH-), 6.29 (s, 1H, NH), 7.16 (m, 2H, Ar-H), 7.23 (m, 2H, Ar-H); MS (ESI) : m/z = 374.1595 [M+H].

5k: ethyl 4-(4-(dimethylamino)phenyl)-1,4,5,6,7,8-hexahydro-2,7,7-trimethyl-5-oxoquinoline-3-carboxylate

^{1}H NMR (500 MHz, $CDCl_3$): 0.95 (s, 3H, -CH_3), 1.04 (s, 3H, -CH_3), 1.24 (t, 3H, -CH_3), 2.12-2.25 (m, 4H, -CH_2-x2), 2.33 (s, 3H, -CH_3), 2.85 (s, 6H, -$N(CH_3)_2$), 4.06 (q, 2H, -OCH_2-), 4.96 (s, 1H, -CH-), 6.58 (d, 2H, *J*=8.5Hz, Ar-H), 6.64 (s, 1H, NH), 7.15 (d, 2H, *J*=8.5Hz, Ar-H); ^{13}C NMR (125 MHz, $CDCl_3$): 195.84, 167.77, 148.94, 148.56, 143.18, 136.02, 128.61, 112.38, 112.24, 106.43, 59.71, 50.84, 40.85, 40.75, 35.38, 32.65, 29.49, 27.28, 19.28, 14.30; MS (ESI) : m/z = 383.2254 [M+H].



Scheme 4: Pluasible mechanism for the synthesis of Polyhydroquinolines

Conclusion:

In summary, we have discovered a sustainable and convenient protocol for the synthesis of polyhydroquinoline derivatives using pumice anchored sulfonic acid (Pumice@SO_3H) as an efficient catalyst under ultrasound irradiation. The attractive features of present protocol are green approach, good yield, recovery of catalyst and easy work-up procedure whereas the catalyst offers simple preparation, high catalytic activity, inexpensive, easy to use, recyclability and good stability.

Abbreviations:

MCRs	= Multicomponent Reactions,
Pumice@SO_3H	= Pumice supported sulfuric acid,
NR	= No Reaction,
RT	= Room Temperature,
SF	= Solvent Free,
USI	= Ultrasound Irradiation.

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