

Alumina nanoparticles: A new and reusable catalyst for synthesis of dihydropyrimidinones derivatives

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ABSTRACT

Present study demonstrates the synthesis of alumina nanoparticles (NPs) by sol-gel method and characterized by various standard microscopic techniques like Powder X-ray Diffraction (PXRD), Scanning Electron Microscopy (SEM), Transmission Electron Microscopy (TEM), Atomic Force Microscopy (AFM), Energy Dispersive Spectroscopy (EDS), FT-IR, UV-visible, and Thermogravimetry-Differential Thermogravimetry (TG-DTA) to evaluate the size, shape and composition of nanoparticles. Furthermore, a solvent free green synthesis of 3, 4-dihydropyrimidinones derivatives were carried by using alumina nanoparticles (γ - Al_2O_3 NPs) as a catalyst through Biginelli reaction of aromatic aldehydes, β -ketoesters, and urea. The structures of the obtained products were confirmed by Fourier transform infrared (FT-IR), and proton nuclear magnetic resonance (^1H NMR) spectra. Effective results were obtained by using nano catalyst. Therefore, present protocol is favored as it offers advantages of higher productivity, short reaction times, simplicity and easy workup. Besides, catalyst is an inexpensive, reusable and recyclable up to four cycles without loss of its activity. Copyright © 2016 VBRI Press.

Keywords: Alumina NPs; microscopic techniques; reusable catalyst; dihydropyrimidinones derivatives; biginelli reaction.

Introduction

Nowadays, metal oxide nanoparticles are attracting noteworthy interest as they can change the viable unconventional to conventional materials in various fields of solid state chemistry. Metal oxide nanoparticles are being fundamentally used as a heterogeneous nanocatalyst in a variety of organic transformations as they contained high surface area than their bulk counterparts [1-2]. Besides, colloidal and monodisperable nanomaterials have also paying tremendous attention because of their small size effects particular in catalytical and biological applications [3-4]. Predominantly, aluminum oxide nanoparticles commonly known as alumina (Al_2O_3 NPs) have trapped the awareness of many researchers due to its great catalytical activities. An alumina nanoparticles can be synthesized by using many techniques including ball milling, spray combustion, hydrothermal, sputtering, sol-gel, microwave and laser ablation [5-10]. Out of all the methods, sol-gel method proved more helpful to obtain well shaped materials with designed texture and composition at low processing temperatures [11-12]. The literature survey reveals that Al_2O_3 was synthesized by sol-gel method using different precursor viz. aluminum triisopropylate in a hydrolysis system consisting of octanol and acetonitrile, aluminum nitrate in aqueous medium, aluminum secondary butoxide in an alcoholic medium [13-15]. However,

chemically synthesized alumina powders are well known for their nanometric sized particles. Therefore, we were synthesized alumina NPs effectively by sol-gel method and further evaluated their catalytic activity in synthesis of dihydropyrimidinones derivatives through Biginelli reaction.

Thus, after tacit the above background we have endeavor to synthesize the first time facile and environmentally benign method for dihydropyrimidinones derivatives through Biginelli reaction of aromatic aldehydes, β -ketoesters, and urea using alumina nanocatalyst. Fortunately, catalyst showed an excellent catalytic activity, less reaction time, recyclability, facile synthesis, ecofriendliness and recoverability of the nano catalyst. The reactions carried out under solvent free conditions are ecofriendly in nature. Thus, this work encourages us to use nano catalyst for Biginelli reaction. Consequently, alumina nanoparticles were used as a catalyst in one-pot, three-component synthesis of 3, 4-dihydropyrimidin-2(1H)-one. Nevertheless, most of the works were studied on pyrimidone derivatives [16-18] and their biological activities such as antiviral, antitumor, antibacterial, and anti-inflammatory. Recently, dihydroxyypyrimidines (DHPMs) have emerged in the form of orally active antihypertensive agent or α -1 adrenoceptor-selective antagonists. Study reveals DHPMs can be used for the development of anticancer drugs. DHPs are

commercially used as calcium channel blockers for the treatment of cardiovascular diseases, including hypertension. The synthesis of DHPs with respect to Multidrug Resistance (MDR) reversal in tumor cell gave a new dimension to their applications [19–22]. In addition, 1, 4-DHP class of compound is excellent starting material for development of anti-tubercular agents [23]. Oxidative aromatization reactions of DHPs are taking place in biological systems in the presence of certain enzymes. Besides, pyrimidone compounds were synthesized by using metal nanoparticles as a catalyst [24–25]. However, these derivatives were not prepared earlier by using nano alumina as a catalyst through Biginelli reaction.

With this regard, we were synthesized biologically active compounds by using nano alumina by one-pot, three-component system. In a present article we highlighted on synthesis and microscopic characterization of alumina nanoparticles and investigated its catalytic role in the Biginelli reaction. To the best of our knowledge, the synthesis of pyrimidone derivatives by using nano alumina is reported for the first time in the Biginelli reaction. Therefore, it is bring to a close that the present study reflects the novel work of alumina nanoparticles by alternative sol-gel method and synthesis of dihydropyrimidinones derivatives through one-pot, multicomponents system using nano catalyst *via* the Biginelli reaction. This work favored the higher productivity, simplicity and short reaction times for synthesis of organic compounds. Besides, nano catalyst is an inexpensive, reusable and recyclable up to four cycles. Schematic route for synthesis of dihydropyrimidinones derivatives using alumina NPs has been displayed in **Scheme 1**. Notwithstanding, in our previous work we were synthesized metal/metal oxides NPs by using protective agents and investigated their catalytic and antibacterial activities [26–28].

Experimental

Materials

All chemicals were purchased from Merck, Aldrich and SD fine chemical companies and were used without further purification. All yields refer to isolated products. Aluminium nitrate (Sigma Aldrich), Ammonia (Sigma Aldrich), Polyvinyl pyrrolidone (E. Merk), different aromatic aldehydes (E. Merk and S. fine), ethylacetoacetate (E. Merk), urea or thiourea (E. Merk) and ethyl acetate (E. Merk).

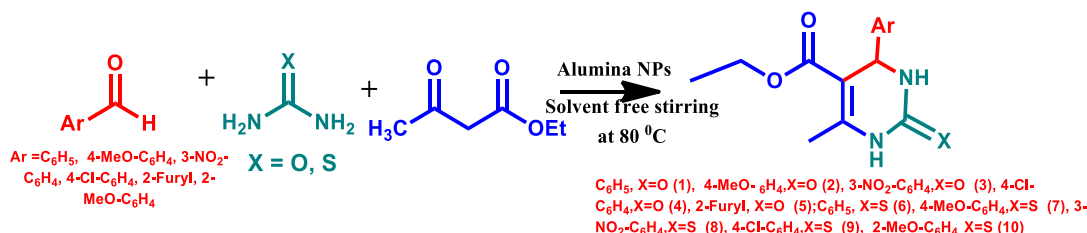
Methods

The purity determination of the substrates and reaction

monitoring were accompanied by thin layer chromatography (TLC) and visualized under ultra-violet (UV) light chamber, while melting points were determined in open capillaries and are uncorrected. All compounds were well known and identified by comparing with melting points, FT-IR spectra, mass analysis and ^1H NMR spectra of some selected compounds with those of the authentic samples. The size and morphology of Alumina NPs were examined by JEOL model JSM-690LV, Scanning Electron Microscopy (SEM) whose maximum magnification is 300,000 X and resolution is 3 nm at the Sophisticated Test and Instrumentation Center, Cochin University, Kerala. The TEM images were formed using CM200 which can produce magnification details up to 1,000,000X with resolution better than 10 Å at Indian Institute of Technology, Pawai (Mumbai). AFM analyses were carried out at DNN Sathyabama University Chennai. The qualitative elemental analyses of the powder sample were studied by JEOL Model JED-200, Energy Dispersive Spectroscopy (EDS) and thermal analyses (TG/DTG/DTA) at heating rate 10 °C/min under nitrogen atmosphere at the Sophisticated Test and Instrumentation Center, Cochin University, Kerala. Further, the crystal structure of the sample was characterized by PXRD, Bruker AXS D8 Advance X-ray diffractometer using $\text{CuK}\alpha$ radiation. Infrared spectroscopy was recorded at a 2 cm^{-1} resolution from 4000 to 400 cm^{-1} on a Bruker IFS 66v Fourier transform spectrometer using KBr pellets. ^1H NMR spectra of organic compounds were carried out on NMR Spectrometer model Avance-II (Bruker) is the acquisition in the SAIF Chandigarh, India. The instrument is equipped with a cryomagnet of field strength 9.4 T. Its ^1H frequency is 400 MHz.

Synthesis of Alumina (Al_2O_3) nanoparticles

The Al_2O_3 nanoparticles were synthesized by precipitation of aluminium hydroxide gels in aqueous solution using aluminium nitrate as a precursor and aqueous ammonia as the precipitating agent. The 50 ml saturated solution of aluminium nitrate was taken in a 250 ml conical flask, then after 150 ml of ammonia was added drop wise, and 0.01 g PVP with continuous stirring and heating. A white color precipitated of aluminum hydroxide was formed. After completion of the precipitation, the mixture was stirred at room temperature for 12 h, filtered, repeatedly washed with distilled water, dried at 120 °C, and calcined at 500 °C for 2 h. The temperature of the muffle furnace was increased linearly from room temperature to 500 °C at 10 °C/min. At 500 °C, the temperature was maintained for 2 h to yield the final material.



Scheme 1. Synthesis of dihydropyrimidinones derivatives catalyzed by Alumina NPs.

General procedure for synthesis of dihydropyrimidinones derivatives catalyzed by alumina NPs

A mixture of the aromatic aldehydes (1mmol), ethylacetoacetate (1mmol), urea or thiourea (1.5 mmol) and alumina NPs (12mol %) was stirred under solvent free condition at room temperature. The progress of reaction was checked on TLC. After completion, the reaction mixture cooled at room temperature. Then, it was extracted with ethyl acetate. The organic layer dried over sodium sulphate and concentrated in a vacuum to afford the crude products. The crude products were purified by crystallization in ethanol. Further, alumina NPs was recovered by centrifugation (5250 rpm) the aqueous layer and reutilized four times for the same reaction.

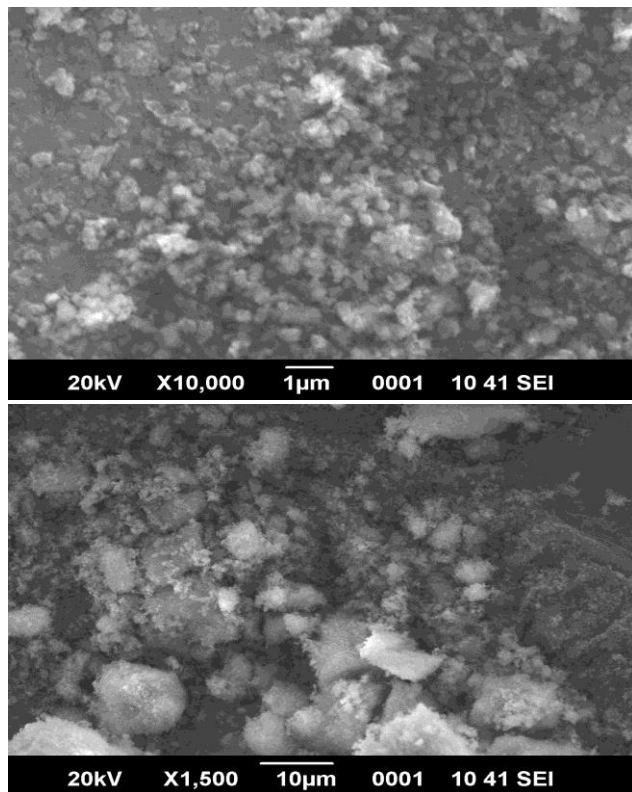


Fig. 1. SEM images of alumina nanoparticles.

Spectral data

Selected data for typical compounds are given bellows:

1. Ethyl 6 - methyl - 2 - oxo - 4 - phenyl - 1, 2, 3, 4 tetrahydropyrimidine-5-carboxylate (**Table 2**, product entry 1): FT-IR (KBr): 3310, 2900-3330, 1738, 1619, 1523, 1439, 1350, 1280, 1245, 1160, 982, 825 cm^{-1} . ^1H NMR (400 MHz, $\text{DMSO}-d_6$): δ 9.10 (s, 1H, NH), 7.79 (s, 1H, NH), 7.30 (s, 5H, Ar), 5.16 (d, 1H, $J = 3.6$ Hz, CH), 3.55 (q, 2H, $J = 7.09$ Hz, OCH_2), 2.35 (s, 3H, CH_3), 1.09 (t, 3H $J = 7.10$ Hz, CH_3).
2. Ethyl 4 - (furan-2-yl) - 6 - methyl - 2 - oxo - 1, 2, 3, 4-tetrahydropyrimidine-5-carboxylate (**Table 2**, product entry 6): FT-IR (KBr): 3410, 2950-3300, 1738, 1628, 1545, 1458, 1450, 1280, 1245, 1169, 982, 835 cm^{-1} . ^1H NMR (400 MHz, $\text{DMSO}-d_6$): δ 9.26 (s, 1H, NH), 7.79 (s, 1H, NH), 7.60 (dd, 1H, $J = 1.8, 3.0$ Hz, Ar), 6.44 (d, 1H, $J = 3.3$ Hz, Ar), 5.23 (d, 1H, $J = 3.3$ Hz,

CH), 4.05 (q, 2H, $J = 7.04$ Hz, OCH_2), 2.43 (s, 3H, CH_3); 1.09 (t, 3H, $J = 7.03$ Hz, CH_3).

3. Ethyl 4-(4-methoxyphenyl)-6-methyl-2-oxo-1, 2, 3, 4-tetrahydropyrimidine-5-carboxylate (**Table 2**, product entry 7): FT-IR (KBr): 3310, 3100-3350, 1630, 1618, 1531, 1439, 1352, 1283, 1245, 1169, 982, 825 cm^{-1} . ^1H NMR (400 MHz, $\text{DMSO}-d_6$): δ 9.12 (s, 1H, NH), 7.87 (s, 1H, NH), 6.95 (s, 2H, Ar-H), 6.65 (s, 2H, Ar-H), 4.95 (d, 1H $J = 3.6$ Hz, CH), 4.19 (m, 2H, CH_2), 3.83 (s, 3H, OCH_3), 3.73 (s, 3H, CH_3), 3.45 (q, 2H, $J = 7.08$ Hz, OCH_2), 1.71 (t, 3H, CH_3), 1.30 (t, 3H $J = 7.06$ Hz, CH_3).

Results and discussion

Microscopic characterization of alumina catalyst

Initially, we examined the synthesis and characterization of alumina nano catalyst. Literature record shows that there various numbers of methods are available for the production of alumina NPs. In the present article nano catalyst was synthesized by sol-gel method. Furthermore, it was characterized by several standard techniques like UV-visible spectroscopy, Fourier-transform infrared spectroscopy, energy dispersive spectroscopy, scanning electron microscopy, transmission electron microscopy, atomic force microscopy, powder X-ray diffraction, and thermal analysis to assess their size, shape and composition. The PXRD pattern of the synthesized nano catalyst has been shown in **Fig. S1** (Supporting information). The obtained diffraction peaks at 2θ of 37.54° , 45.8° and 66.73° were assigned to (3 1 1), (4 0 0) and (4 4 0) miller indices of nano γ -alumina. An average crystalline size was found to be 9 nm by Debye-Scherrer formula with using (4 0 0) plane. The successfully obtained nano γ -alumina was supported by JCPDS file no. 00-010-0425 of γ -aluminium oxide nanoparticles [29]. In addition, the morphology and size were established by various standard microscopic techniques like SEM, TEM, and AFM, while elemental analysis was found out by EDS. The SEM image (**Fig. 1**) unveil a fine particulate matter where the particles are elongated having spherical and rod in shape, it is appeared like aggregation of different cotton shape structures.

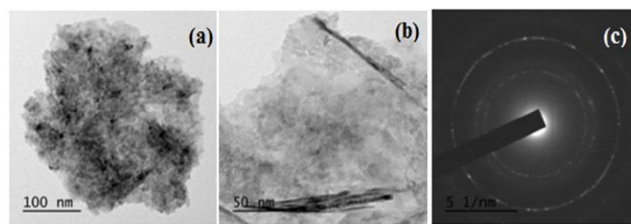


Fig. 2. (a-b) TEM images of alumina NPs at different resolution, (c) selected area electron diffraction pattern (SAED).

The spherical shape crystals were observed unlike uniform arrangement. Furthermore, sample was characterized by TEM technique. TEM micrographs (**Fig. 2(a-b)**) show a fine needle shaped particles with an average particle size ranging from 9 to 25 nm at the bottom level, while at apex side it was approximately 9-15 nm. However, spherical shape particles were found with an average size below 10 nm, while other bigger aggregate form molecules found their sizes in 30-40 nm. The SAED

image (**Fig. 2(c)**) highlighted a high degree of crystallinity, with planes clearly emphasizing the crystallization of γ - Al_2O_3 [30]. Additionally, AFM 3D and 2D-Topological images (**Fig. 3**) of alumina NPs were explored, representing both surface roughness and particle agglomeration. The particle size of alumina obtained from AFM histogram (**Fig. S2**) supported with that calculated from TEM and PXRD results using Scherrer equation. Nevertheless, EDS analysis confirmed the composition of Al_2O_3 nanoparticles by viewing the peaks of C, Al and O.

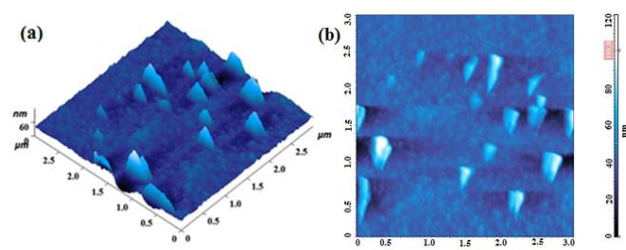


Fig. 3. (a) AFM 3D image (3×3 μm) (b) AFM 2D image of alumina NPs.

Furthermore, infrared spectroscopy was used to study the chemical affinity of the sample towards the surface of the nanoparticles, and its spectrum was carried out in the region 4000–400 cm^{-1} (Figure S3). **Fig. S3** shows strong peaks around 3438, 3429, 3420 and 3400 cm^{-1} which may be due to the stretching vibration of O–H group (air, water or CO_2 background) [31–34]. Yet again the peak obtained at 1634 cm^{-1} could be assigned due to the bending vibration of H–OH (adsorbed water molecules). The peaks in the range 510–749 cm^{-1} could be corresponded to the metal–oxygen bond (Al–O–Al and Al–O) which established the existence of Al_2O_3 . However, one unknown peak was obtained at 2300 cm^{-1} which could not give any assignment, such ambiguity interpretation was reported previously on formation of an unknown carbonaceous organic residue [35]. Furthermore, the metal oxide nature of catalyst was confirmed by UV–visible study (Figure S4) that shows strong absorption band at 405 nm corresponds to band gap energy of 3.06 eV which was supported with good agreement [36–39].

Additionally, the thermal property of nano catalyst was evaluated by TGA–DTA techniques at heating rate 10 $^{\circ}\text{C}/\text{min}$ under N_2 atmosphere (Figure S5) for understanding the thermal stability. These techniques are widely used to evaluate the thermal degradation behaviour and thermal stability nano catalyst [40–41]. The TG/DTG curves were recorded gradually weight loss from 39–739 $^{\circ}\text{C}$ with T_{DTG} peak at 65.67 $^{\circ}\text{C}$ which may be due to existence of moisture (water) on the surface of catalyst and carbonaceous organic moiety, which were supported by FTIR spectroscopy. Further, DTG was supported by DTA with one medium broad endothermic peak at T_{DTA} 109 $^{\circ}\text{C}$ and a broad exothermic peak at T_{DTA} 109 $^{\circ}\text{C}$.

Nano Al_2O_3 -catalyzed dihydropyrimidinones derivatives synthesis

In a continuation of previous work [27–28] now we have made attempts to synthesize new organic derivatives by using nano alumina catalyst. In the present article, we tried to develop alternative method for the synthesis of

biologically active organic dihydropyrimidinones compounds in a solvent free green synthesis by using nano catalyst. Therefore, we chose the Biginelli reaction for the synthesis of dihydropyrimidinones in a facile way. In our initial study for optimization of reaction conditions, the reaction of benzaldehyde, ethylacetoacetate and urea was used as a model reaction (Scheme 1) to optimize the reaction conditions. Firstly, as trial and error way the reaction was conducted in various solvents using nano catalyst under refluxing under solvent free conditions. As it can be seen from scenario (**Table 1, Entry 6**), the best results obtained under the solvent free conditions. Obviously, **Table 1** put in force for synthesis of dihydropyrimidinones derivatives under solvent free condition.

Table 1. Synthesis of dihydropyrimidinones derivatives using nano catalyst in various solvents and solvent free condition.

Entry	Solvent	Time(min)	Yield(%) ^a
1	Ethanol	80	60
2	Methanol	80	59
3	CH_3CN	80	58
4	CHCl_3	80	66
5	Toluene	80	45
6 ^b	Solvent-free Condition	60	94,93,91, 90

^aIsolated Yield, ^bCatalyzed recycled Four times

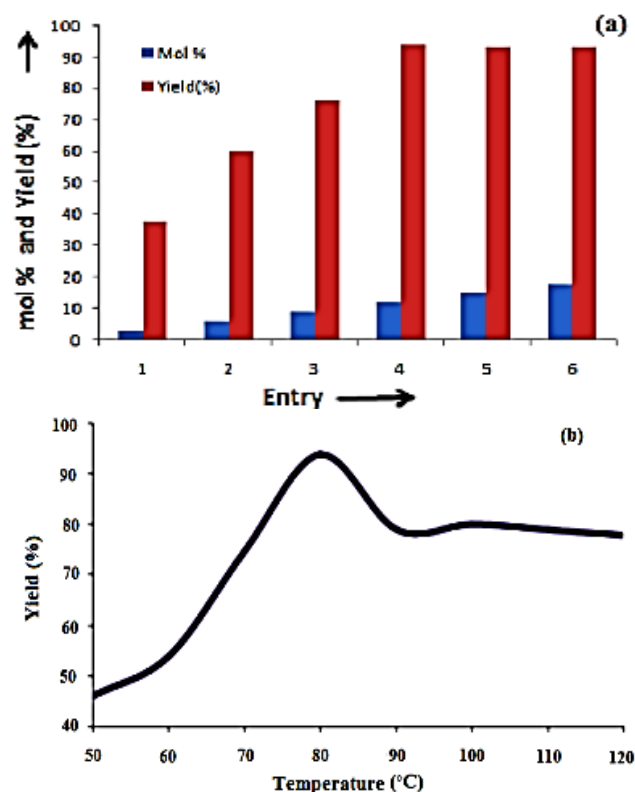


Fig. 4. (a) Effect of amount of nano alumina catalyst for the synthesis of dihydropyrimidinones derivative, (b) Effect of temperature in synthesis of dihydropyrimidinones derivative.

After then, we turned our attention toward screening of appropriate concentration of alumina nanoparticles from (3 mol% to 18 mol%) for the synthesis of dihydropyrimidinones. Interestingly, the yield of products

increases remarkably from 32% to 94 % with increasing the concentration of catalyst amount from 3 mol% to 12 mol%, but when amount of catalyst increased from 12 mol% to 18 mol%, no further increase in the yield of products was observed. As a result, the amount of 12 mol% of alumina NPs was selected for all subsequences reaction (Fig. 4a).

Table 2. Synthesized dihydropyrimidinones derivatives by using alumina NPs as catalyst under solvent free condition.

Entry	Ar	X	Time (hr)	Yield (%)	Mp (°C)	
					Found	Lit ^[42-44]
1	C ₆ H ₅	O	5	91	203	201-203
2	4-MeO-C ₆ H ₄	O	5	87	203	204-205
3	3-NO ₂ -C ₆ H ₄	O	7.5	85	225	226-228
4	4-Cl-C ₆ H ₄	O	4.5	79	212	212-214
5	2-Furyl	O	5	79	202	200-202
6	C ₆ H ₅	S	6.5	90	206	207-209
7	4-MeO-C ₆ H ₄	S	6	88	153	148-150
8	3-NO ₂ -C ₆ H ₄	S	7.5	74	210	210-212
9	4-Cl-C ₆ H ₄	S	6.5	73	183	180-182
10	2-MeO-C ₆ H ₄	S	6.5	70	190	188-189

Furthermore, we check out the effect of temperature in synthesis of products. The effect of temperature (Fig. 4b) for the synthesis of dihydropyrimidinones derivatives was studied by carrying out the reaction at 60 °C, 70 °C, 80 °C, 90 °C, 100 °C, 110 °C and 120 °C. Fig. 4b shows best yield obtained at 80 °C temperature for all reactions. Under the optimized reaction conditions, a series of dihydropyrimidinones derivatives (1-10) were synthesized. The results are summarized in Table 2. In all cases, aromatic aldehydes substituted with either electron-donating or electron-withdrawing groups underwent the reaction smoothly and gave the expected products in good to high yields under the same reaction conditions. Moreover, heteroaromatic aldehydes like furfural were successfully converted to the corresponding heteroaryl moderate yields (Table 2, entry 5). On the other hand, a watchful examination of the results from Table 2 show that the compounds with electron donating moiety reacted efficiently requiring not as much of time (Table 2, entries 1-2, 4,7, 9-10), however compound with electron-withdrawing group required more time to react (Table 2, entries 3 and 8) provided that analogous yields.

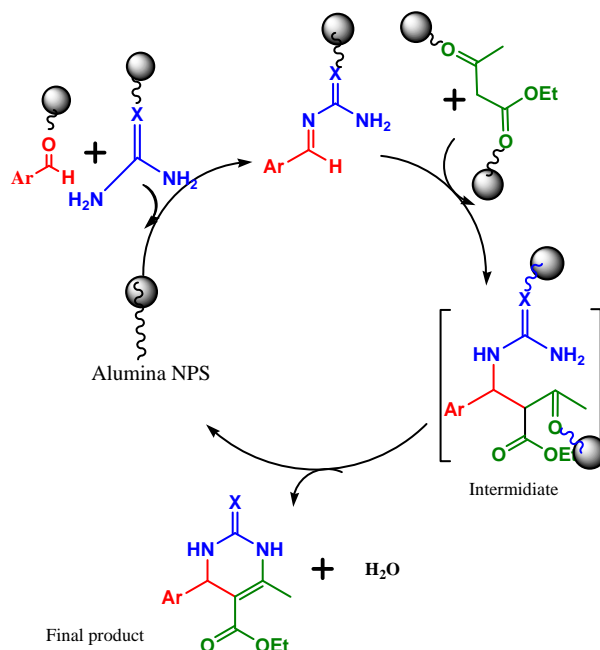
Table 3. Reutilization of alumina NPs catalyst for the synthesis of dihydropyrimidinones derivatives.

No. of Cycles	Fresh	1	2	3	4
Yields (%)	91	90	90	88	85

The suggested mechanism (Scheme 2) illustrates the postulated interaction of the acylimine intermediate with alumina nanoparticles. A superficial inspection of the reaction mechanism suggests that electron withdrawing groups in the aryl aldehydes would accelerate the rate of the reaction. However, it is intriguing to note that the reaction proceeded rapidly to afford such high yields irrespective of whether electron-withdrawing or electron-donating functional group was attached to the reactant aryl aldehydes. This could be attributed due to strong catalytical activity of alumina NPs as compared to conventional methods.

Reusability of nano catalyst

We also investigated the possibility of recycling the alumina NPs catalyst. Recyclability of catalyst is an important task. In a present work the recovery of nano catalyst is an easy and efficient. The catalyst recovered by filtration from the model reaction and it was checked in the subsequent runs without further purification (Table 3). The activities of the catalyst get affected slightly for the next cycle in terms of yields (Table 3, no. of cycles 1, 2, and 3) after four successive runs for the model reaction. It reveals that the catalyst displayed very good reusability.



Scheme 2. A plausible mechanism for synthesis of dihydropyrimidinones derivative using alumina nano catalyst.

Conclusion

In summary, we have successfully synthesized alumina nanoparticles by sol-gel method and characterized by various standard spectroscopic techniques to know its particle sizes and shape. Further we effectively developed a method involving the use of alternate, an efficient, safer nanocatalyst for solvent free green synthesis of dihydropyrimidinones derivative. To the best of our knowledge, this is the first time reported synthesis of dihydropyrimidinones derivative by using alumina catalyst. The mildness of the conversion, experimental simplicity, excellent yields, and shorter reaction time makes this procedure more gorgeous in synthesizing a variety of derivatives. In case of catalyst concentration, a better yield of product was obtained for 12 mol % of alumina. Along with this a facile approach in recovery and reusability of the alumina catalyst is significant toward environmentally benign procedures. Therefore, alumina nanoparticles can contribute the green synthesis of aromatic organic compounds in solvent free condition. Also, it can be used for various organic transformation reactions.

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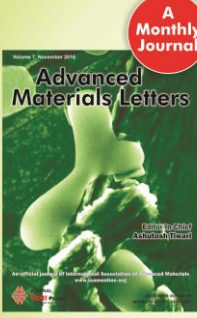
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Author contributions

Conceived the plan: Juneja, Chaudhary, Gandhare ; Performed the experiments: Tanna, Chaudhary; Data analysis: Chaudhary, Juneja, Gandhare, Tanna; Wrote the paper: Chaudhary, Tanna. Authors have no competing financial interests.

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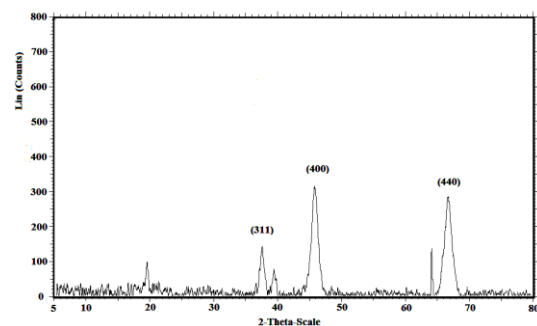


Fig. S1. XRD Spectra of alumina nanoparticles.

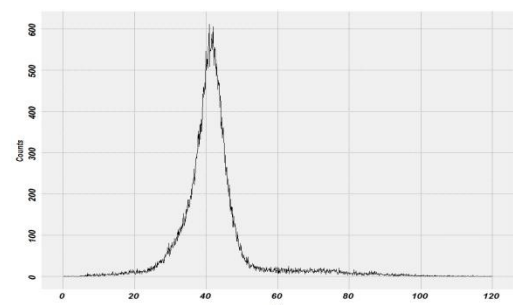


Fig. S2. Histogram of alumina NPs.

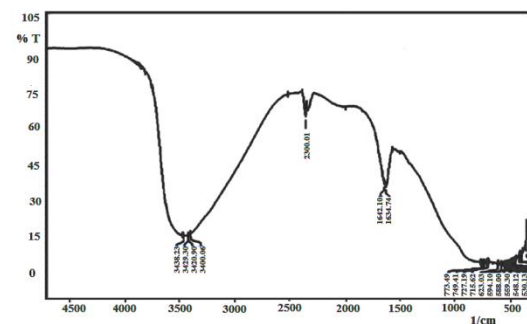


Fig. S3. FTIR spectra of alumina NPs.

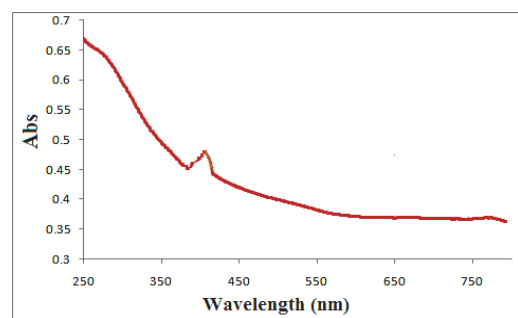


Fig. S4. UV-visible spectra of Alumina NPs.

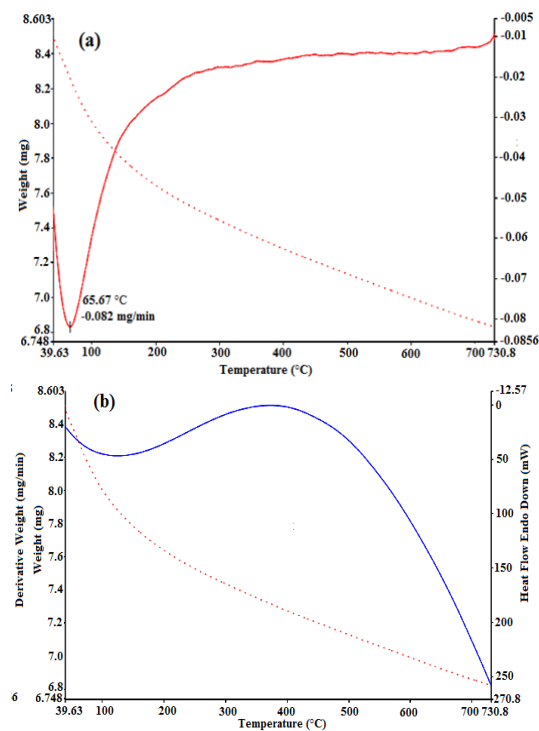


Fig. S5. (a) TG/DTG, and (b) TG/DTA spectra of alumina NPs.