
Research Article

Synthesis, characterization and first application of perovskite LaFeO₃ nano particles for the synthesis of tetrahydropyridine derivatives

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ABSTRACT

In this study, the perovskite-type LaFeO₃ nanoparticles have been fabricated by sol-gel method and characterized by X-ray powder diffraction (XRD), Fourier infrared spectroscopy (FT-IR), scanning electron microscopy (SEM) and transmission electron microscopy (TEM). The average particle size of LaFeO₃ is about 50 nm and the synthesis of tetrahydropyridine derivatives was investigated by the reaction of aryl aldehydes, aryl amines and ethyl acetoacetate in the presence of heterogeneous catalyst. The high efficiency, short time, the very simple separation of the catalyst from the reaction environment is the advantages of the above method. In addition, the catalyst can be reused and can be used for catalytic activity five times without significant reduction.

Keywords: Tetrahydropyridine; LaFeO₃ nano perovskite; Ethanol; Heterogeneous catalyst.

Introduction

Heterocyclic compounds have a wide application in the production of pharmaceutical products, agricultural and veterinary industries. They are also used as antioxidants, anti-corrosion agents and as organic intermediates [1]. In this context, tetrahydropyridines and their derivatives are one of the useful compounds to form the nucleus of many natural

products and a wide range of bioactive and pharmaceutical compounds. It is especially Specified that their medicinal application also increase by the increasing number of their substitutions [2]. As for pharmacological and biological wide properties of tetrahydropyridines, the introduction of new methods for synthesis of these compounds is of particular importance and it has attracted much attention from organic and pharmaceutical chemists.

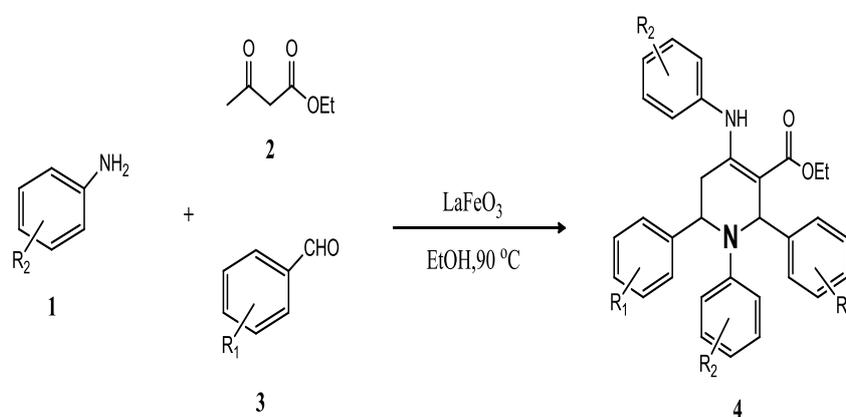
Recently, the synthesis of tetrahydropyridines was reported using Catalysts such as diethanol ammonium hydrogen sulfate (DHS), lemon juice, sulfonic acid pyridinium hydrogen sulfate, $[\text{PySO}_3\text{H}][\text{HSO}_4]$, $(\text{Al}_2\text{O}_3/\text{BF}_3/\text{Fe}_3\text{O}_4, \text{TiCl}_4/\text{SiO}_2, \text{FeCl}_3/\text{SiO}_2, \text{MgFe}_2\text{O}_4/\text{Cellulose}/\text{SO}_3\text{H}, \text{PbCr}_x\text{Fe}_{12-x}\text{O}_{19}, \text{cerium ammonium nitrate}, \text{Fe@Si-Gu-Prs}$ and graphene oxide [3-13]. Perovskites are remarkable for their good catalytic properties. In Many studies, have attempted to determine the relationship between the catalytic activity and the physical and chemical properties of the surface and mass of perovskite oxides. Some of the Advantages of perovskite catalysts are summarized below:

- 1) A wide variety of compounds have been identified with these structures.
- 2) The mass structure is well defined and the surface can be estimated using this defined mass structure.
- 3) The capacity, stoichiometric, and empty spaces can be broadly different.
- 4) There is much information about their physical and chemical properties.

LaFeO_3 nano-perovskite and its associated compounds with the general formula $\text{LaB}_Y\text{Fe}_{(1-Y)}\text{O}_3$ or $\text{La}_{(1-X)}\text{A}_X\text{FeO}_3$ are very important. This importance is due to interesting magnetic properties and high potential of their applications in the memory of computers, electro-ceramic sensors, catalysts of various reactions such as hydrocarbon oxidation, solid electrolytes, etc [14-19]. In the context of catalytic application these materials can be used as

active catalysts for the oxidation or reduction of pollutant gases, for the reaction of hydrogenation of alkenes and hydrogenolysis of alkanes [20].

It has also been used in the production of chemical sensors to detect alcohols, gases and oxygen permeation membranes [21-22]. Various materials are used in the preparation of moisture sensors, which is one of the most important of them Rare-Earth orthoferrite. Another application is the catalytic combustion of hydrocarbons such as methane and natural gas at low temperature, which is associated with NO_x production limitations [23]. Due to the importance of tetrahydropyridines, is considered providing a simple, effective and safe method in terms of environmental conditions. In this study, a simple and effective method is provided for the synthesis of tetrahydropyridine derivatives from the three-component reaction of beta-keto ester, aryl amines, and aryl aldehydes using LaFeO_3 perovskite as a catalyst under reflux conditions in the presence of ethanol solvent (Scheme 1).



Scheme 1. Synthesis of tetrahydropyridine derivatives

2. Experimental

All compounds were obtained from Sigma-Aldrich, Fluka and Merck in analytical grade and used without further purification. NMR spectra were recorded on Advance Bruker-400MHz spectrometers. FT-IR spectra were recorded on an AVATAR-370-FTIR Thermo

Nicolet instrument. Melting points were measured using Electro thermal 9100 apparatus. Scanning electron microscopy (SEM) images were taken using a Zeiss LEO 1450 VP/35Kv instrument (Germany). Transmission-electron microscopy (TEM) images were obtained by Philips CM-10 (Eindhoven, The Netherlands). Powder X-ray diffraction (XRD) patterns were obtained on a Philips diffractometer of the X'pert Company.

2.1. Preparation of LaFeO₃ nano perovskite

Citric acid (10 mL) was added to a mixture of lanthanum (III) nitrate hexahydrate (10 mL) and iron (III) nitrate (10 mL). The reaction mixture stirred in room temperature at 1 h. After that, the reaction mixture was placed in oil bath at 90 °C for 3-4 h to form a brown gel-like complex, and then resulting complex was place in an oven at 120 °C for 24 h, which finally was obtained porous solid with a light brown color. In the last step, the porous solid is ground to become a complete powder. Then, it was ground in agate mortar and turned into powder and fired at 900 °C in air for 4 h.

2.2. Characterization of LaFeO₃ nano perovskite

The strong absorptions at 500-600 cm⁻¹ are corresponding to oxide of perovskite. The absorption at 538 cm⁻¹ is corresponding to Fe-O stretching vibration. O-Fe-O bending vibration appeared at 669 cm⁻¹. The observed absorptions at 1366-1738 cm⁻¹ are corresponding to other phases of LaFeO₃ (Figure 1).

The X-ray diffraction pattern of catalyst, after calcination at 900 °C for 4 h, is shown in Figure 2. The results of the X-ray diffraction pattern show that the calcination temperature up to 900 °C causes the perovskite structure to be completed. The peak of 22.7, 25.4, 32.3, 39.8, 46.3, 57.6, 67.5, 72.3, and 76.6 indicates the crystal structure of the perovskite. Among these, peak of 32.3 is the most important peak of perovskite.

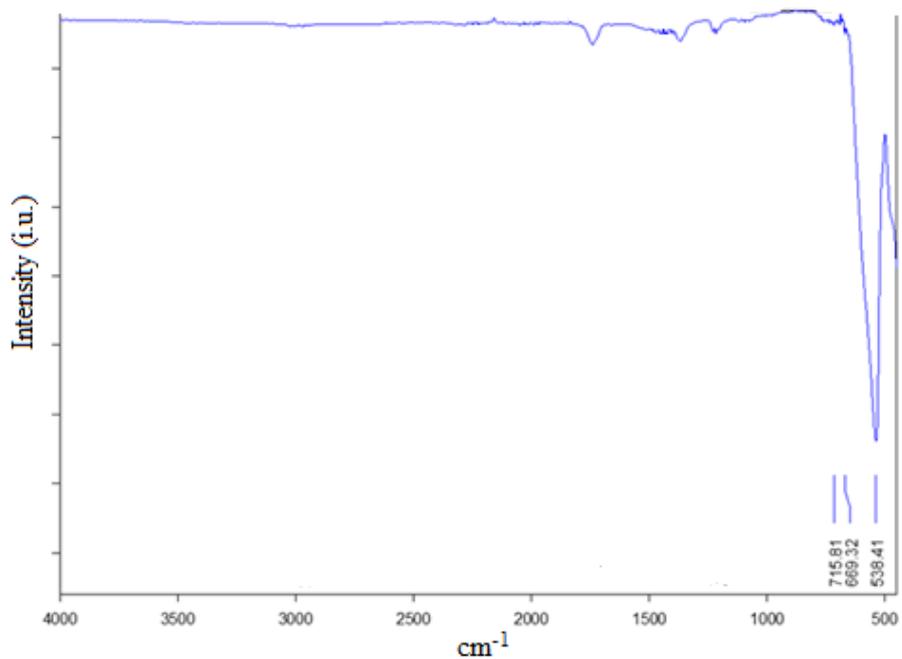


Fig. 1. FTIR spectra of the LaFeO_3 polymeric gel precursors calcined at $900\text{ }^\circ\text{C}$.

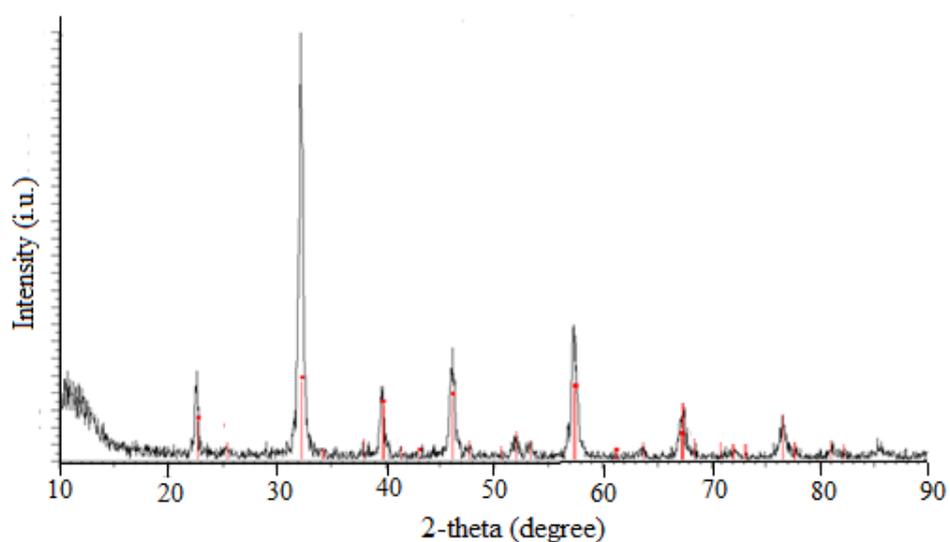


Fig. 2. XRD patterns of the LaFeO_3 powders.

Figure 3 shows the SEM images of LaFeO_3 nanoparticles. These images also exhibit typical morphologies for prepared powder. The particles show uniform particle distribution.

As the SEM images show the nanoparticles have regular shapes with well-defined crystal faces.

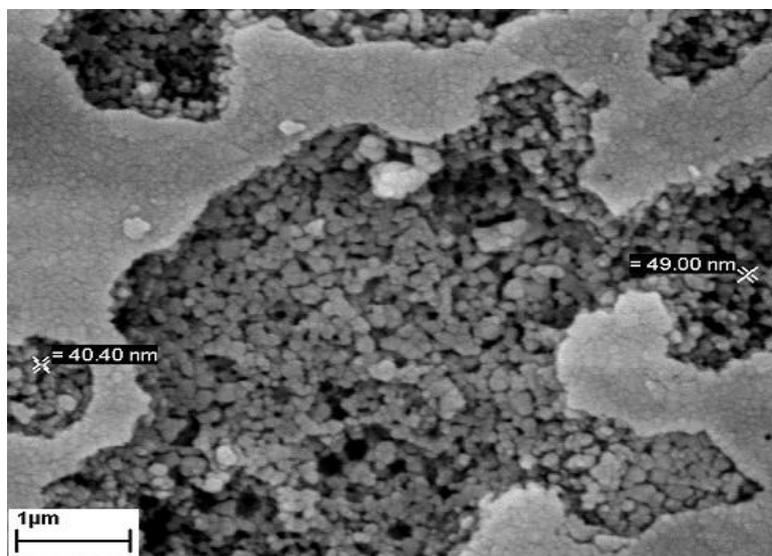


Fig. 3. SEM images of LaFeO₃ nanoparticles

2.3. General procedure for the preparation of tetrahydropyridine derivatives

A mixture of aryl aldehyde (2 mmol), aryl amine (2 mmol), ethyl acetoacetate (1mmol) and LaFeO₃ (15 mol%) was stirred in oil bath at 90 °C in the presence of ethanol solvent. The progress of the reaction was monitored by TLC: ethyl acetate/n-hexane (1:4). After completion of reaction, the reaction mixture was resolved in hot ethanol and catalyst was separated by simple filtration. The crude product was obtained after evaporation and for further purification, recrystallized from ethanol.

2.4. Spectral data for selected compounds

Ethyl 1-(3-iodophenyl)-4-((3-iodophenyl)amino)-2,6-diphenyl-1,2,5,6-tetrahydropyridine-3-carboxylate (4b):

White solid; IR (KBr) = 3251, 3056, 2966, 2855, 1651, 1595, 1448, 1372, 1252,1071,cm⁻¹;
¹HNMR (400 MHz, CDCl₃) δ ppm: 1.53 (t, J = 7.2 Hz, 3H, CH₃CH₂), 2.72(dd, J = 14.4, 2.2 Hz, 1H, H'-5), 2.84 (dd, J = 14.4, 5.4Hz, 1H, H''-5), 4.30-4.33 (m,1H, OCH_aH_b), 4.40-4.48 (m, 1H, OCH_aH_b), 5.08-5.18 (m, 1H, H-6), 6.26-6.33 (m,1H, ArH), 6.37 (s, 1H, H-2), 6.49

(m, 1H, ArH), 6.63 (t, $J = 7.0$ Hz, 2H, ArH), 6.76 (t, $J = 7.5$ Hz, 1H), 6.85 (d, $J = 7.2$ Hz, 2H, ArH), 6.94 (d, $J = 6.0$ Hz, 1H, ArH), 7.15-7.29 (m, 9H, ArH), 7.43 (d, $J = 6.8$ Hz, 1H, ArH), 10.29 (s, 1H, NH); ^{13}C NMR (100 MHz, CDCl_3) δ ppm: 16.02, 34.55, 56.29, 59.29, 61.16, 95.14, 96.58, 99.88, 113.48, 122.74, 126.27, 126.53, 127.3, 127.3, 127.64, 127.8, 128.76, 129.6, 130.2, 131.46, 135.51, 136.07, 140.21, 142.91, 144.18, 149.28, 156.37, 169.28.

Ethyl 1-(4-bromophenyl)-4-((4-bromophenyl)amino)-2,6-bis(4-methoxyphenyl)-1,2,5,6-tetrahydropyridine-3-carboxylate (4d):

White solid; IR (KBr) = 3239, 3064, 2979, 2834, 1647, 1603, 1462, 1370, 1248, 1068, cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ ppm: 1.47 (t, $J = 8.0$ Hz, 3H, CH_3CH_2), 2.70 (dd, $J = 15.4, 2.7$ Hz, 1H, $\text{H}'-5$), 2.83 (dd, $J = 15.4, 5.6$ Hz, 1H, $\text{H}''-5$), 3.79 (s, 6H, OCH₃), 4.27-4.35 (m, 1H, OCH_aH_b), 4.42-4.49 (m, 1H, OCH_aH_b), 5.04 (s, 1H, H-6), 6.20 (d, $J = 6.0$, 2H, ArH), 6.29 (s, 1H, H-2), 6.39 (d, $J = 6.80$ Hz, 2H, ArH), 6.76-6.88 (m, 5H, ArH), 7.04-7.24 (m, 7H, ArH), 10.26 (s, 1H, NH); ^{13}C NMR (100 MHz, CDCl_3) δ ppm: 15.93, 34.73, 55.83, 56.4, 56.48, 58.75, 61.06, 100.17, 109.47, 114.84, 115.29, 115.78, 120.3, 128.3, 128.52, 128.69, 132.7, 133.14, 135.11, 136.21, 138.2, 147.12, 156.45, 159.39, 160.06, 169.29.

Ethyl 2,6-bis(4-chlorophenyl)-1-(p-tolyl)-4-(p-tolylamino)-1,2,5,6-tetrahydropyridine-3-carboxylate (4f):

White solid; IR (KBr): 3244, 3035, 2978, 2917, 2855, 1654, 1594, 1516, 1485, 1254, 1175, 1074 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ ppm: 1.48 (t, $J = 6.8$ Hz, 3H), 2.19 (s, 3H), 2.31 (s, 3H), 2.71 (dd, $J = 2.4, 15.2$ Hz, 1H), 2.78 (dd, $J = 5.2, 15.2$ Hz, 1H), 4.30-4.38 (m, 1H), 4.41-4.49 (m, 1H), 5.08 (br s, 1H), 6.30 (t, $J = 8.112$ Hz, 3H), 6.40 (d, $J = 8.8$ Hz, 2H), 6.90 (d, $J = 8.4$ Hz, 2H), 6.98 (d, $J = 8$ Hz, 2H), 7.07 (d, $J = 8.4$ Hz, 2H), 7.24-7.29 (m, 6H), 10.23 (br s, 1H); ^{13}C -NMR (100 MHz, CDCl_3): 14.80, 20.11, 20.89, 33.61, 54.86, 57.34, 59.73, 97.25, 113.01, 125.77, 125.81, 127.84, 128.07, 128.33, 128.72, 129.57, 132.0, 132.75, 135.01, 135.87, 141.22, 142.80, 144.37, 156.16, 168.02.

Ethyl 1-(4-bromophenyl)-4-((4-bromophenyl)amino)-2,6-bis(4-cyanophenyl)-1,2,5,6-tetrahydropyridine-3-carboxylate (4i):

White solid; IR (KBr): 3244, 3035, 2978, 2917, 2855, 1654, 1594, 1516, 1485, 1254, 1175, 1074 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ ppm: 1.48 (t, $J = 6.8$ Hz, 3H), 2.19 (s, 3H), 2.31 (s, 3H), 2.71 (dd, $J = 2.4, 15.2$ Hz, 1H), 2.78 (dd, $J = 5.2, 15.2$ Hz, 1H), 4.30–4.38 (m, 1H), 4.41–4.49 (m, 1H), 5.08 (br s, 1H), 6.30 (t, $J = 8.11.2$ Hz, 3H), 6.40 (d, $J = 8.8$ Hz, 2H), 6.90 (d, $J = 8.4$ Hz, 2H), 6.98 (d, $J = 8$ Hz, 2H), 7.07 (d, $J = 8.4$ Hz, 2H), 7.24–7.29 (m, 6H), 10.23 (br s, 1H); ^{13}C -NMR (100 MHz, CDCl_3): 14.80, 20.11, 20.89, 33.61, 54.86, 57.34, 59.73, 97.25, 113.01, 125.77, 125.81, 127.84, 128.07, 128.33, 128.72, 129.57, 132.0, 132.75, 135.01, 135.87, 141.22, 142.80, 144.37, 156.16, 168.02.

3. Results and Discussion

In order to achieve standard and optimal conditions, The reaction of benzaldehyde, 3-iodo aniline, ethyl acetoacetate in the presence of LaFeO_3 nano perovskite was selected as a model reaction and were investigated in different conditions. The results are presented in Table 1.

Table 1. Optimization of the catalyst amount and temperature using the model reaction^a

Entry	Temperature ($^{\circ}\text{C}$)	Catalyst (mol%)	Time (min)	Yield ^b (%)
1	room temperature	---	35	---
2	room temperature	10	31	34
3	room temperature	15	28	42
4	room temperature	20	28	40
5	60	10	31	43
6	60	15	27	46
7	70	10	25	51
8	70	15	24	57
9	80	10	21	63
10	80	15	18	75
11	90	10	20	82
12	90	15	15	97
13	90	20	20	87
14	100	15	15	85

^aReaction conditions: benzaldehyde (2 mmol), 3-iodo aniline (2 mmol), ethyl acetoacetate (1 mmol) ^bIsolated yield.

The effect of different amounts of catalyst on the yield of the reaction was investigated and the highest yield was obtained in the presence of 15 mol% of the catalyst. The lower amounts

of catalyst increased the reaction time and reduced yield of product, and increasing the catalyst amounts to 20 mol% did not effect on the yield of reaction.

To optimize the reaction temperature, the desired reaction was performed at higher temperatures, and finally the temperature of 90 °C was selected as the best condition in terms of product efficiency and reaction time. After obtaining the optimal amount of catalyst and temperature, the solvent effect was also investigated. The reaction of benzaldehyde, 3-ido aniline, ethyl acetoacetate and 15 mol% of the catalyst was performed in 5 mL of different solvents and ethanol solvent was selected as the optimal solvent. The reaction in other solvents was slower and less efficient. (Table 2)

Table 2. Optimization of the Solvent using the model reaction^a

Entry	Solvent	Temperature (°C)	Time (min)	Yield ^b (%)
1	Acetonitrile	90	15	41
2	Dichloromethane	90	15	30
3	Methanol	90	15	62
4	Ethanol	90	15	95
5	Acetone	90	15	27
6	Water	90	15	15
7	Solvent-free	90	15	low

^aReaction conditions: benzaldehyde (2 mmol), 3-iodo aniline (2 mmol), ethyl acetoacetate (1 mmol) and LaFeO₃ nano perovskite (15 mol%) ^bIsolated yield.

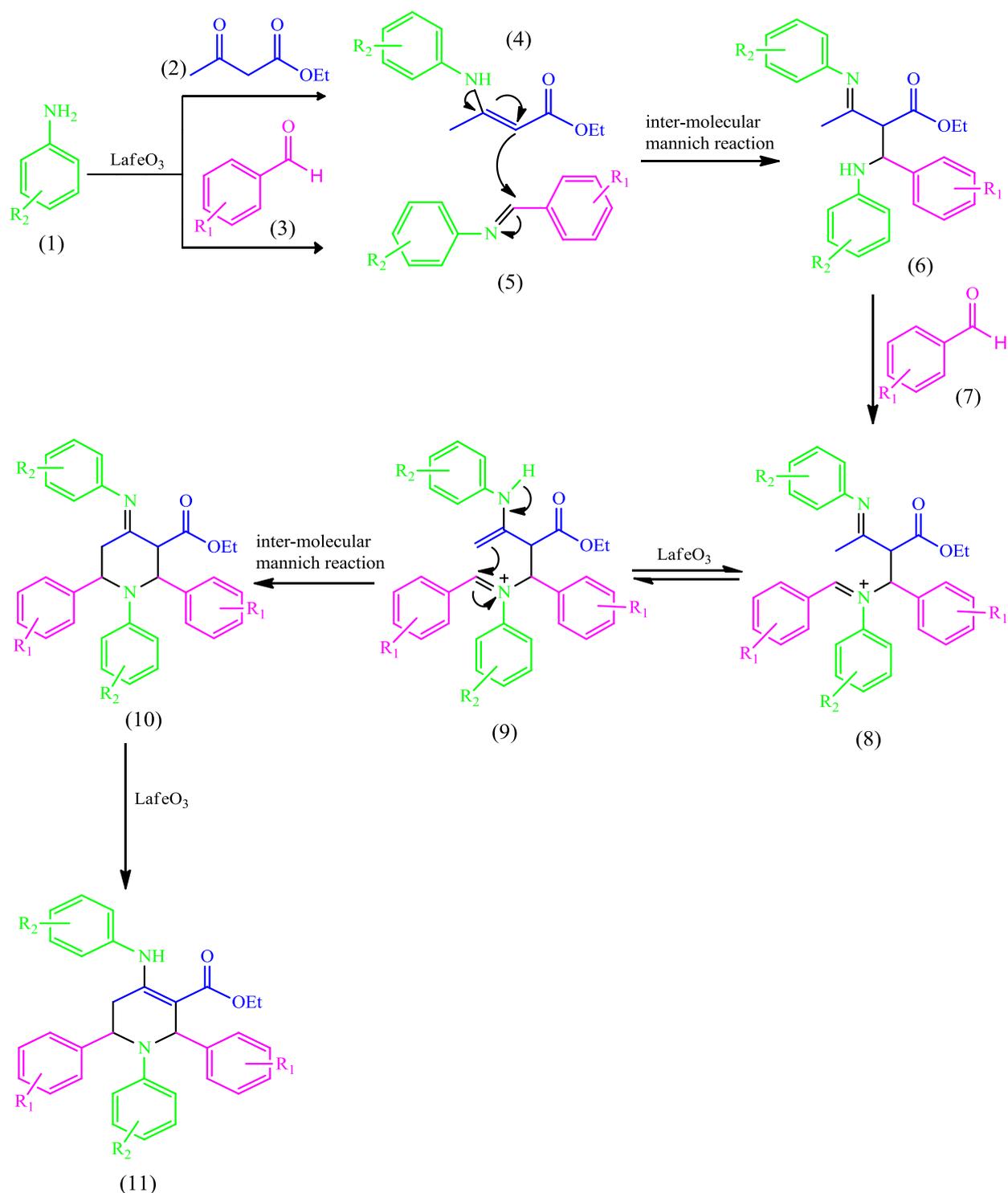
As a result, according to the efficiencies and reaction times, LaFeO₃ nano perovskite was reported to be an effective and efficient catalyst in the synthesis of pyridine derivatives through one-pot and three-component reaction of aryl aldehydes, aryl amines and ethyl acetoacetate in the presence of ethanol solvent and temperature 90 °C. In order to expand the proposed method, various derivatives of tetrahydropyridines were synthesized using optimal conditions. As can be seen in Table 3, the various derivatives of aryl aldehyde and aryl amine containing different substituent groups reacted with ethyl acetoacetate, and the corresponding products were obtained with appropriate yields. The electron-donation and electron-withdrawing substituent groups including methyl, methoxy, cyano, chloro, bromo, iodo, and nitro, were tested in these reactions, acceptable results were obtained in all cases.

Table3. Synthesis of tetrahydropyridine derivatives using LaFeO₃ nano perovskite as catalyst^a

Entry	R ₁	R ₂	Product	Time (min)	Yield ^b (%)	MP (°C) Found	MP (°C) Reported	Reference
1	C ₆ H ₅	C ₆ H ₅	4a	10	91	171-172	175-176	25
2	C ₆ H ₅	3-IC ₆ H ₄	4b	15	95	170-172	170-172	26
3	4-CH ₃ OC ₆ H ₄	C ₆ H ₅	4c	15	92	164-166	165-167	27
4	4-CH ₃ OC ₆ H ₄	4-BrC ₆ H ₄	4d	20	94	184-186	184-186	26
5	4-ClC ₆ H ₄	C ₆ H ₅	4e	10	90	226-228	228-230	28
6	4-ClC ₆ H ₄	4-CH ₃ C ₆ H ₄	4f	10	93	224-227	227-229	29
7	4-CH ₃ C ₆ H ₄	C ₆ H ₅	4g	10	93	227-229	227-229	25
8	4-CH ₃ C ₆ H ₄	4-CH ₃ OC ₆ H ₄	4h	10	91	220-222	219-222	30
9	4-CNC ₆ H ₄	4-BrC ₆ H ₄	4i	15	92	218-220	220-223	27
10	4-NO ₂ C ₆ H ₄	C ₆ H ₅	4j	15	94	245-248	247-250	25

^aReaction conditions: benzaldehyde (2 mmol), 3-iodo aniline (2 mmol), ethyl acetoacetate (1 mmol), LaFeO₃ nano perovskite (15 mol%) and ethanol solvent ^bIsolated yield.

The suggested mechanism for the synthesis of tetrahydropyridine derivatives is presented in scheme 2.



Scheme 2. Suggested mechanism for the synthesis of tetrahydropyridine derivatives using LaFeO_3 nano perovskite as catalyst

Catalytic amount of 15 mol% LaFeO_3 initiate the reaction to form enamine (4) and imine (5) from condensation of aniline (1), β -ketoester (2) and aryl aldehyde (3). Then, intermolecular Mannich-type reaction occurs in between enamine (4) and imine (5) to yield

intermediate (6). The intermediate (6) undergoes condensation reaction with second molecule of aryl aldehyde gives intermediate (8). The intermediate (8) after tautomerization generates intermediate (9) followed by intramolecular Mannich-type reaction to produce desired functionalized tetrahydropyridine derivatives (11).

The reusability of the LaFeO_3 was evaluated using the model reaction under the optimized conditions. For this purpose, after completion of the reaction, the catalyst was retrieved from the reaction medium by simple filtration. After being washed with ethanol for several times and dried at $40\text{ }^\circ\text{C}$, the catalyst can be reused without any deactivation even after five successive cycles of synthesis of Tetrahydropyridine. (Fig. 4)

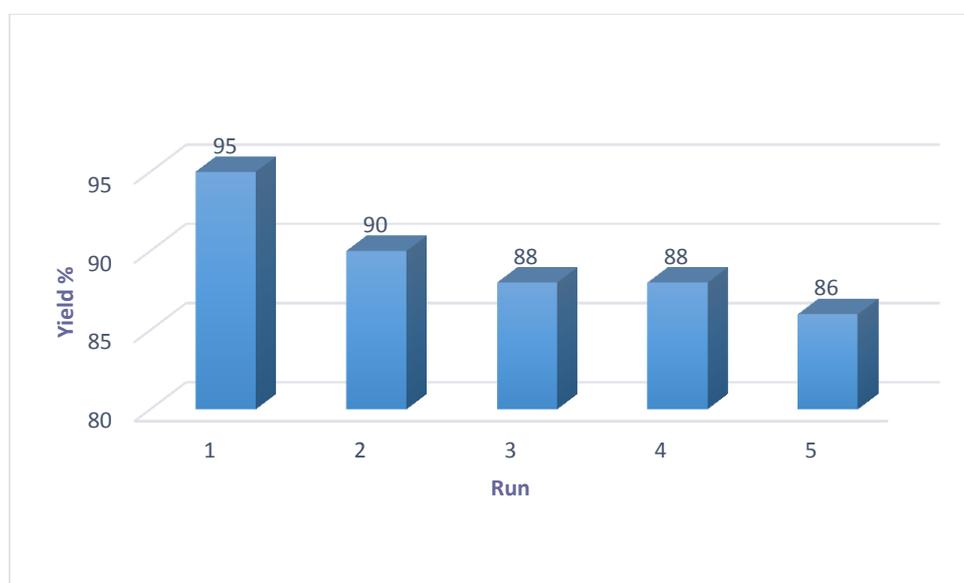


Fig. 4. Reusability of LaFeO_3 using the model reaction.

4. Conclusion

In summary, this paper describes a convenient and efficient process for one-pot three-component synthesis of tetrahydropyridine derivatives in the presence of LaFeO_3 nano perovskite as novel catalyst and ethanol solvent at $90\text{ }^\circ\text{C}$. The protocol used in this research has many advantages such as short reaction times, easy work up, high products yields, low

cost, reusability of catalyst, the simplicity of using the catalyst, separating the catalyst only by filtering, recycling the catalyst, simple processing.

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