

An Affordable and Eco-friendly Method for *Pseudo*-five-component Synthesis of Tetrahydropyridines Using Gum Arabic-OPO₃H₂ as a Natural-based Catalyst

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This study describes a facile and environmentally friendly protocol for the synthesis of highly functionalized tetrahydropyridines *via pseudo*-five-component reaction of aromatic aldehydes, amines and ethyl acetoacetate using GA-OPO₃H₂ as catalyst. In addition to the easy preparation of GA-OPO₃H₂ from bio-renewable materials, this catalyst can also be recycled and reused without obvious loss of catalytic activity. The synthesis of tetrahydropyridines *via* this new green methodology offers significant advantages in terms of no use of a solvent, no hazardous waste, uncomplicated work-up and high yields of products.

Keywords: Gum Arabic-OPO₃H₂, Natural-based catalyst, Tetrahydropyridine, *Pseudo*-five-component reaction, Solvent-free condition

INTRODUCTION

The five-substituted tetrahydropyridines (THPs) have been the subject of considerable synthetic efforts because of their potent pharmacological properties including anti-hypertensive [1] anti-malarial [2], anti-inflammatory [3] and anticonvulsant [4]. Droperidol [5], tazomeline [6] and GTS-21 [7] are known as THPs which have been used as drugs in the treatment of Alzheimer disease (Fig. 1).

In recent years, multicomponent reactions (MCRs) as superior synthetic strategies have been utilized for the rapid preparation of a variety of biologically active compounds [8]. The synthesis of THPs *via* MCRs offers significant advantages in terms of shorter reaction times, higher product yields, atom economy, energy consumption reduction and low waste production [9].

The most common method for synthesis of various THPs *via* MCRs is *pseudo*-five-component reaction of *p*-substituted anilines, *p*-substituted aldehydes and alkyl acetoacetate. Recently, several catalysts such as nano-

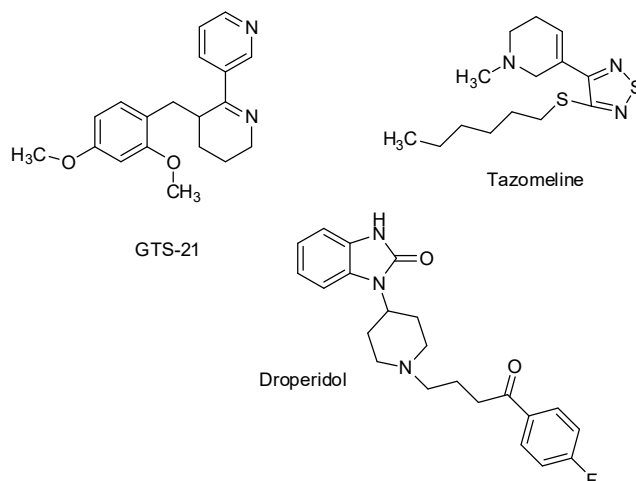


Fig. 1. Effective chemical structures in the treatment of Alzheimer disease.

BF₃/cellulose [10], nano-Al₂O₃/BF₃/Fe₃O₄ [11], InCl₃ [12], bromodimethylsulfonium bromide [13], *L*-proline/TFA [1], tetrabutylammonium tribromid [14], I₂ [15], CAN [16], ZrOCl₂·8H₂O [17], *p*-TsOH·H₂O [18], Fe(NO₃)₃·9H₂O [19], FeCl₃/SiO₂ [20], amberlite IRA400-Cl resin/I₂/KI [21],

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HOAc [22], picric acid [23], nano-silica sulfuric acid [24] and NiFe₂O₄@SiO₂ [25] have been used for this reaction. Despite the remarkable achievements, biocompatible synthesis of these potent pyridines using safer catalysts is still in demand.

Biopolymers have unique properties, which make them attractive alternatives for supports in catalytic applications. Gum Arabic (GA) is a renewable and naturally biopolymer that is emerged from Acacia trees [26]. GA is also known to be a branched and complex acidic heteropolysaccharide whose main chain consists of (1→3)-*β*-*D*-galactopyranosyl units and side chains were 1-arabinofuranosyl, 1-rhamnopyranosyl, *D*-galactopyranosyl, and *D*-glucopyranosyluronic acid units [27]. GA have some advantageous such as low cost, safety, and low environmental risks and in recent years has been used as support [28-30], hydrogel [31,32] and gold nanoparticle stabilizer [33].

Inspired by these results and in pursuit of our previous research in developing green methods to synthesize heterocyclic compounds, this work describes the biocompatible synthesis of THPs *via pseudo*-five-component reaction of aromatic aldehydes, various amines and ethyl acetoacetate using GA-OPO₃H₂ as metal free and natural-based catalyst.

EXPERIMENTAL

General

All compounds were purchased from Merck, Aldrich, and Fluka chemical companies and used without any additional purification FT-IR spectra were run on a Bruker, Equinox 55 spectrometer. Thin-layer chromatography (TLC) was done on silica gel 60 F254 plates (Merck). A Bruker (DRX-400 Avance) NMR was used to record the ¹H NMR and ¹³C NMR spectra. Melting points were determined by a Buchi melting point B-540 B. V. CHI apparatus and were uncorrected. Elemental analysis (C, H, N) was conducted using a Vario EL analyzer. Electrical mortar-heater which was

used for grinding of reaction mixture, was purchased from Borna-Kherad Co., Iran, Yazd.

X-ray diffraction (XRD) patterns were obtained by a Philips Xpert MPD diffractometer equipped with a Cu K α anode ($k = 1.54 \text{ \AA}$) in the 2θ range from 10° to 80°. Quantitative elemental information and maps of GA-OPO₃H₂ was studied *via* energy-dispersive X-Ray spectroscopy (EDS) by a Phenom pro X instrument. Thermal gravimetric analysis (TGA) was conducted using “Universal V4.5A TA” instrument. The morphology was studied using a Philips XL30 scanning electron microscope (SEM).

Preparation of GA-OPO₃H₂

GA is exuded from Acacia trees. Then, the catalyst (GA-OPO₃H₂) is simply prepared by adding P₂O₅ (3 g) to *n*-hexane solution (5 ml) containing gum Arabic (1 g). The mixture was stirred for 10 min at 68 °C. The mixture is sticky so, we added dichloromethane (5 ml) to make it into a powder. The resulted suspension was filtered and washed 2 times with acetonitrile, to remove unreacted P₂O₅ and dried at room temperature.

General Procedure for Synthesis of THP Derivatives

Firstly, a mixture of para-substituted anilines (2 mmol) and ethyl acetoacetate (1 mmol) was heated with stirring at 80 °C for 30 min in the presence of GA-OPO₃H₂ (0.03 g). Then, the para-substituted benzaldehydes (2 mmol) were added, and the final mixture was heated at 80 °C. The progress of the reaction was monitored by TLC (*n*-hexane:EtOAc (7:3)), After completion of the reaction, the mixture was dissolved in hot ethanol and filtered off for separation of catalyst. By adding water and sodium carbonate to filtrate, the product was appeared as a solid. For more purification of product, solid was recrystallized by ethanol.

Spectral Data for Selected Compounds

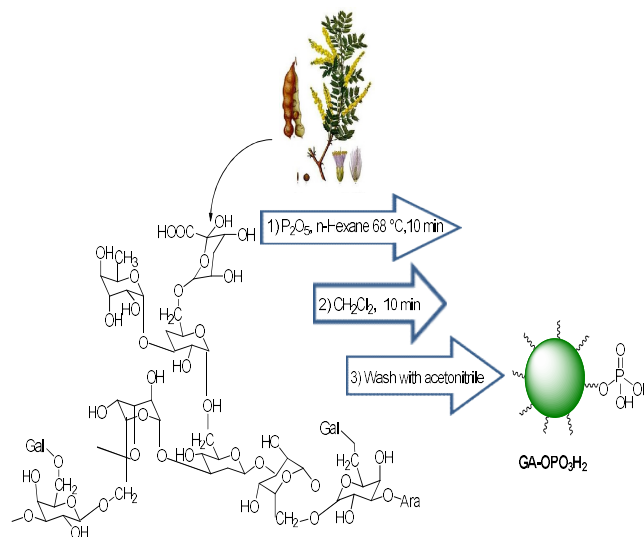
Ethyl-1-(4-tolyl)-4-(4-tolylamino)-2,6-bis(4-bromophenyl)-1,2,5,6-tetrahydropyridine-3-carboxylate. Pale

yellow solid. ^1H NMR (CDCl_3 , 400 MHz) δ 10.21 (s, 1H, NH), 7.39 (d, 5H, $^3J = 7.6$ Hz, Ar-H), 7.19 (d, 2H, $^3J = 7.6$ Hz, Ar-H), 7.00 (d, 2H, $^3J = 7.6$ Hz, Ar-H), 6.96 (d, 2H, $^3J = 7.6$ Hz, Ar-H), 6.89 (d, 2H, $^3J = 7.6$ Hz, Ar-H), 6.36 (br, d, 2H, $^3J = 7.6$ Hz, Ar-H, H-2), 6.29 (br, s, 2H, Ar-H), 5.04 (s, 1H, H-6), 4.44 (m, 1H, OCH_2), 4.33 (m, 1H, OCH_2), 2.75 (m, 2H, H-5,5'), 2.29 (s, 3H, Ar- CH_3), 2.18 (s, 3H, Ar- CH_3), 1.44 (t, 3H, $^3J = 6.4$ Hz OCH_2CH_3). ^{13}C NMR (CDCl_3 , 100 MHz) δ 168.0, 156.1, 144.3, 143.3, 141.8, 141.9, 135.9, 135.0, 131.7, 131.3, 129.6, 128.5, 128.2, 125.88, 125.82, 120.8, 120.1, 113.0, 97.2, 59.7, 57.4, 54.9, 33.6, 20.9, 20.2, 14.8. IR: 3372, 2977, 2917, 2874, 1651, 1599, 1514, 1485, 1363, 1244, 1067, 1009, 826. mp 215-217 °C. Anal. Calcd for $\text{C}_{34}\text{H}_{32}\text{Br}_2\text{N}_2\text{O}_2$: C, 62.87; H, 4.80; N, 4.44. Found: C, 59.68; H, 4.74; N, 4.13.

Ethyl-1-(4-ethylphenyl)-4-((4-ethylphenyl)amino)-2,6-bis(4-chlorophenyl)-1,2,5,6-tetrahydropyridine-3-carboxylate. White solid. ^1H NMR (CDCl_3 , 400 MHz) δ 10.23 (s, 1H, NH), 7.26 (m, 6H, Ar-H), 7.08 (d, 2H, $^3J = 7.6$ Hz, Ar-H), 6.98 (d, 2H, $^3J = 7.6$ Hz, Ar-H), 6.92 (d, 2H, $^3J = 7.6$ Hz, Ar-H), 6.38 (d, 2H, $^3J = 7.6$ Hz, Ar-H), 6.31 (m, 3H, Ar-H), 5.07 (s, 1H, H-6), 4.43 (m, 1H, OCH_2), 4.35 (m, 1H, OCH_2), 2.82-2.75 (m, 2H, H-5,5'), 2.59 (q, 2H, $^3J = 7.5$ Hz, Ar- CH_2CH_3), 2.49 (q, 2H, $^3J = 7.6$ Hz, Ar- CH_2CH_3), 1.45 (t, 3H, $^3J = 6.8$ Hz, OCH_2CH_3), 1.20 (t, 3H, $^3J = 7.6$ Hz, Ar- CH_2CH_3), 1.14 (t, 3H, $^3J = 7.6$ Hz, Ar- CH_2CH_3). ^{13}C NMR (CDCl_3 , 100 MHz) δ 168.1, 156.2, 144.6, 142.9, 142.2, 141.3, 135.2, 132.8, 132.2, 132.1, 128.4, 128.1, 127.9, 125.9, 113.1, 97.3, 59.7, 57.5, 54.9, 33.7, 28.3, 27.6, 15.7, 15.5, 14.8. IR: 3226, 2972, 1648, 1600, 1514, 1486, 1409, 1368, 1316, 1253, 1069, 811. mp 209-211 °C. Anal. Calcd for $\text{C}_{36}\text{H}_{36}\text{Cl}_2\text{N}_2\text{O}_2$: C, 72.11; H, 6.05; N, 4.67. Found: C, 70.95; H, 6.38; N, 4.58.

RESULTS AND DISCUSSION

In this research, an efficient and environmentally benign protocol was developed for the synthesis of THPs derivatives using *pseudo*-five-component reaction of aromatic aldehydes,



Scheme 1. Preparation of GA-OPO₃H₂

various amines and ethyl acetoacetate in the presence of GA-OPO₃H₂. The steps for the synthesis of GA-OPO₃H₂ catalyst are shown in Scheme 1. The synthesized catalyst was characterized by FT-IR, XRD, SEM, MAPPING, EDS and TGA [34].

The FT-IR spectra of GA and GA-OPO₃H₂ are shown in Fig. 2. The characteristic absorption bands of GA at 1069 and 1148 cm^{-1} (C-O, stretch), 1616 (O-H, bending), 1728 cm^{-1} (C=O, aliphatic acid), 2928 cm^{-1} (C-H, stretch), and 3000-3600 cm^{-1} (O-H, stretch) were observed (Fig. 2a). The strong peaks at 900-1200 cm^{-1} are the finger print of carbohydrates. These peaks are observed in the case of GA and GA-OPO₃H₂. The bands at 1071 cm^{-1} (P-O-C) and 1228 cm^{-1} (P=O) are attributed to the stretching vibrations of phosphoric acid section that overlapping with the absorption bands of GA in this region.

The particles size and morphology of GA-OPO₃H₂ was investigated using FESEM. The exterior surface of the GA-OPO₃H₂ appears as irregular rocky surface in which the dimensions of them were achieved below 10 μm (Fig. 3).

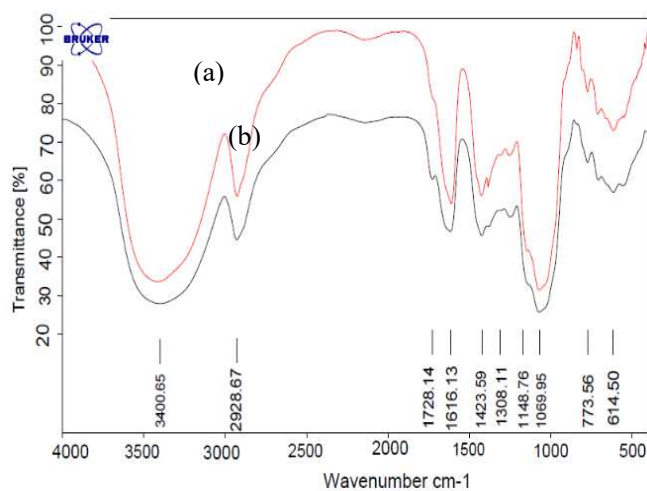


Fig. 2. FT-IR spectra of (a) GA (b) GA-OPO₃H₂.

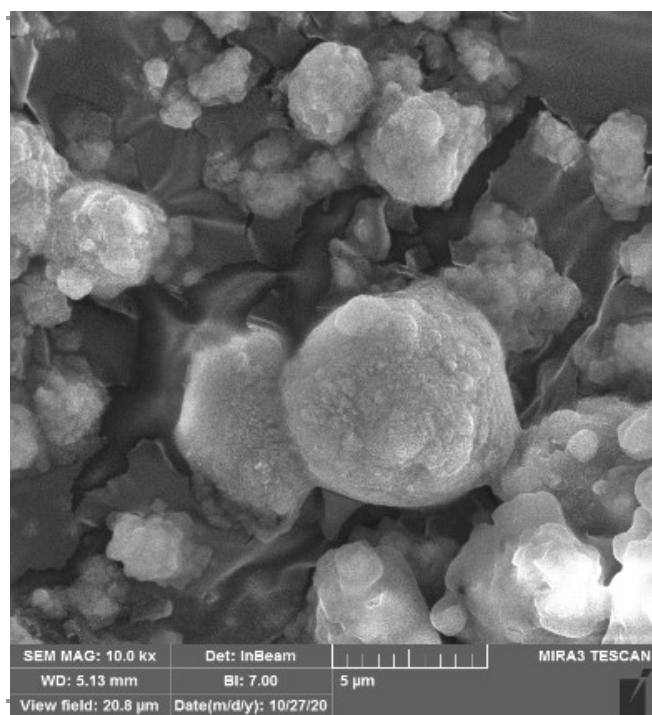


Fig. 3 FESEM image of GA-OPO₃H₂.

Also, the presence of C, O, N, and P in catalyst was investigated by using EDX analysis data (Fig. 5). The percentages of O, C, N and P in catalyst are 57.53, 27.65,

8.33 and 6.49, respectively. Figure 6 shows the elemental mapping of GA-OPO₃H₂. The images and patterns taken, confirm the presence of carbon, nitrogen, oxygen and phosphorus elements in the catalyst. In addition, it shows that catalyst functional groups are well scattered throughout the catalyst.

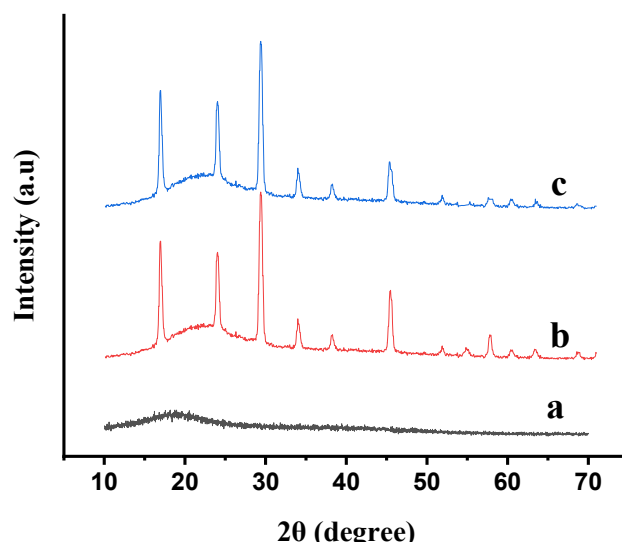


Fig. 4. XRD pattern of a) GA, b) GA-OPO₃H₂, c) reused GA-OPO₃H₂.

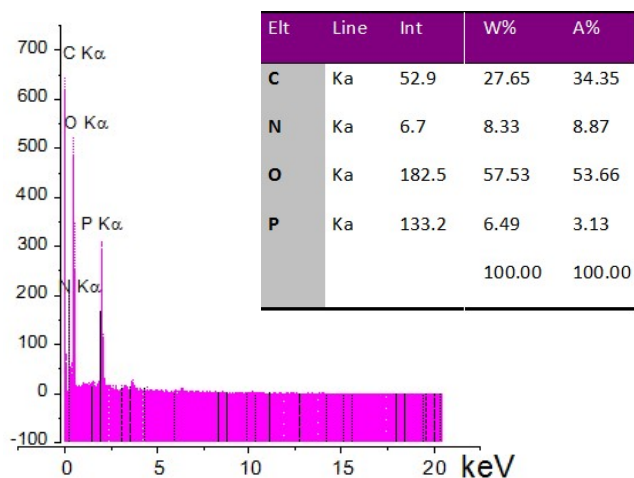


Fig. 5. EDS spectra of GA-OPO₃H₂.

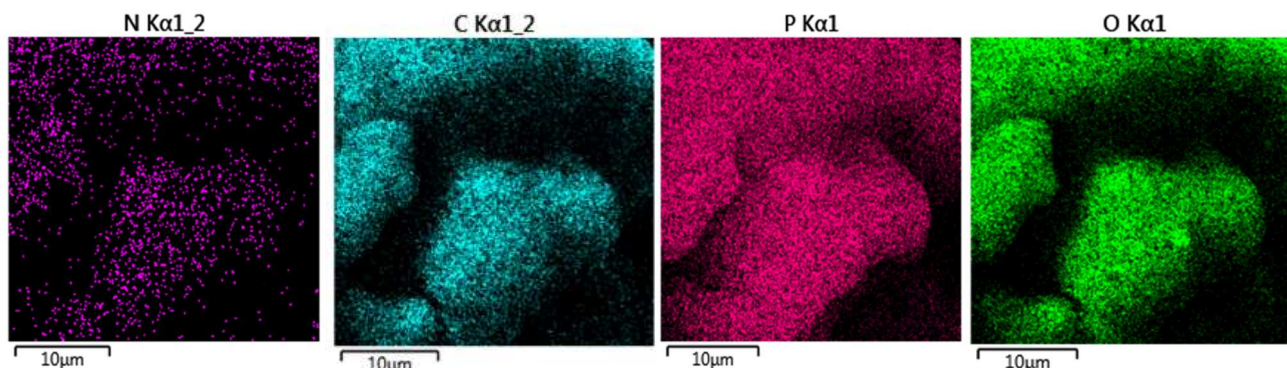


Fig. 6. Elemental mapping images of GA-OPO₃H₂.

The thermal stability of Gam Arabic and GA-OPO₃H₂ were evaluated by thermo-gravimetric analysis (TGA) in the temperature range of 33-404 °C (Fig. 7). The TGA curve of Gam Arabic shows three steps weight loss which contain a) 10 % at 70-150 °C, b) 50% at 250-320 °C and c) 10% at 320-400 °C. The char yield of Gam Arabic at 400 °C is 30%. And so, the TGA curve of GA-OPO₃H₂ shows three steps weight loss which contain a) 5% at 30-120 °C, b) 10% at 150-220 °C and c) 15% at 250-400 °C. The first weight loss is attributed to the evaporation of free water and the second and third weight loss steps were corresponded to decomposition and burning of GA section of catalyst. The char yield of GA-OPO₃H₂ at 400 °C is 63.96%. These evidences show that thermal stability of GA-OPO₃H₂ is higher than of Gam Arabic. Acidic capacity of catalyst was measured *via* titration of it with 0.03 N of NaOH. The number of OPO₃H₂ groups on the gum GA-OPO₃H₂ is 6.6 mmol g⁻¹.

The catalytic efficiency of GA-OPO₃H₂ was checked in the one-pot reaction of aniline derivatives (2 mmol), ethyl acetoacetate (1 mmol) and aldehydes (2 mmol) to the synthesis of THPs derivatives. In order to optimize the reaction conditions, the model reaction of 4-chloroaniline, ethyl acetoacetate and 4-chlorobenzaldehyde in the presence of GA-OPO₃H₂ was initially carried out under the various conditions (Table 1). The highest yield was achieved at 80 °C

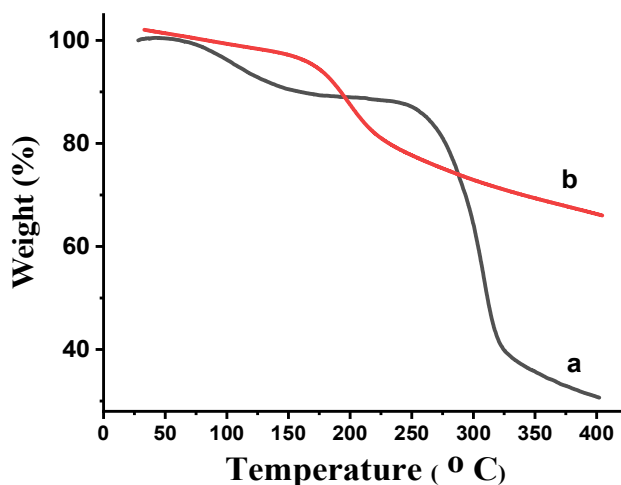
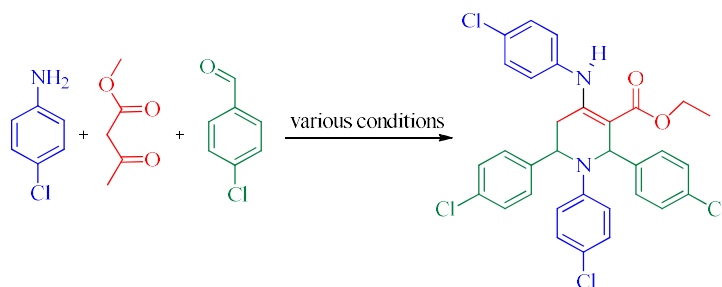


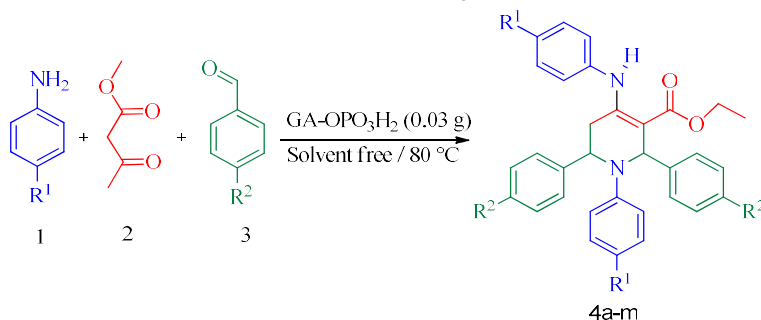
Fig. 7. Thermal gravimetric analysis pattern of (a) Gam Arabic, (b) GA-OPO₃H₂.

under solvent free condition using 0.03 g of catalyst (Table 1, Entry 11).

Due to remarkable results from the above experiments and for scrutiny of this catalyst's efficiency, we have synthesized THP derivatives *via* condensation of para-substituted anilines (2 mmol), ethyl acetoacetate (1 mmol) and para-substituted benzaldehydes (2 mmol) in the presence of GA-OPO₃H₂ (0.03 g) under solvent-free conditions at 80 °C (Table 2). All compounds were identified by physical and spectroscopic data.

Table 1. The Reaction of 4-Chloroaniline (2 mmol), Ethyl Acetoacetate (1 mmol) and 4-Chlorobenzaldehyde (2 mmol) under Various Conditions

Entry	Solvent	Catalyst (g) ^a	Condition	Time (h)	Yield (%) ^b
1	-	-	80 °C	7	51
2	C ₂ H ₅ OH	0.05	r. t. ^c	6	30
3	THF	0.05	Reflux	6	43
4	CHCl ₃	0.05	Reflux	6	40
5	CH ₃ CN	0.05	Reflux	6	37
6	C ₂ H ₅ OH	0.05	Reflux	6	63
7	CH ₃ OH	0.05	Reflux	6	61
8	-	0.05	80 °C	5	70
10	-	0.04	80 °C	5	78
11	-	0.03	80 °C	4	87
12	-	0.02	80 °C	5	64
13	-	0.03	70 °C	5	80
14	-	0.03	60 °C	5	73

^aGA-OPO₃H₂. ^bIsolated yield. ^cRoom temperature.**Table 2.** Synthesis of THPs Derivatives in the Presence of GA-OPO₃H₂ under Solvent-free Condition at 80 °C^a

Entry	R ¹ /R ²	Product ^b	Time (h)	Yield (%) ^c	M.P. °C Found/Lit. [Ref.]
1	Et/H	4a	4	83	188-189/187-189 [10]
2	Et/Me	4b	5	89	175-177/ 176-178 [10]
3	Br/OMe	4c	4	86	217-219/217-219 [11]
4	Br/H	4d	4	88	201-202/200-202 [20]
5	Me/H	4e	4	85	191-193/193-194 [10]
6	Me/Br	4f	5	89	216/215-217 [10]
7	Et/Cl	4g	5	85	210-212/209-211 [11]
8	Br/Cl	4h	5	84	193-195/193-195 [24]
9	Cl/Cl	4i	4	87	214-215/214-215 [11]
10	Cl/H	4j	4	88	200-203/202-204 [19]
11	H/Br	4k	4	80	218-220/217-220 [11]
12	H/Me	4l	5	78	228-230/227-229 [11]
13	H/Cl	4m	4	80	202-203/202-204 [22]

^a1 (mmol):2 (mmol):3 (mmol): GA-OPO₃H₂ (g) is equal to 2:1:2:0.03. ^bAll compounds were identified by physical and spectroscopic data (M.P., FT-IR, ¹H NMR and ¹³C NMR). ^cIsolated yield.

The stereochemistry of products was previously studied by X-ray crystallographic analysis. [15]. The optical purity of products was confirmed by ^1H NMR and only one diastereomer of product was formed ($dr = 100$).

The reusability of the catalyst was also examined on the model reaction, it was separated by filtration, washed 3 times with ethanol, dried in oven at $50\text{ }^\circ\text{C}$ and reused five times in the model reaction. The obtained results showed the good reusability (Fig. 8). Partial loss of activity may be due to blockage of active sites of the catalyst.

The comparison between efficiency of GA-OPO₃H₂ and other reported catalysts are shown in Table 3. According to this comparison, GA-OPO₃H₂ can be a good catalyst for promotion of five-substituted THPs synthesis.

A mechanism for this multi component reaction is proposed in Scheme 2. GA-OPO₃H₂ activates the C=O group in β -ketoester and aldehyde to promote the β -enaminone (4) or imine (5) formation. The intermolecular Mannich addition of the β -enaminone (4) to the imine (5) affords the intermediate (6). Subsequently, the reaction of activated aldehyde with the intermediate (6) proceeds to afford the

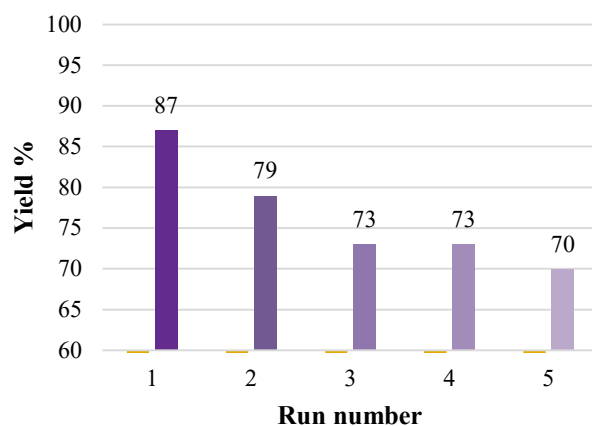


Fig. 8. Catalyst recycling experiments.

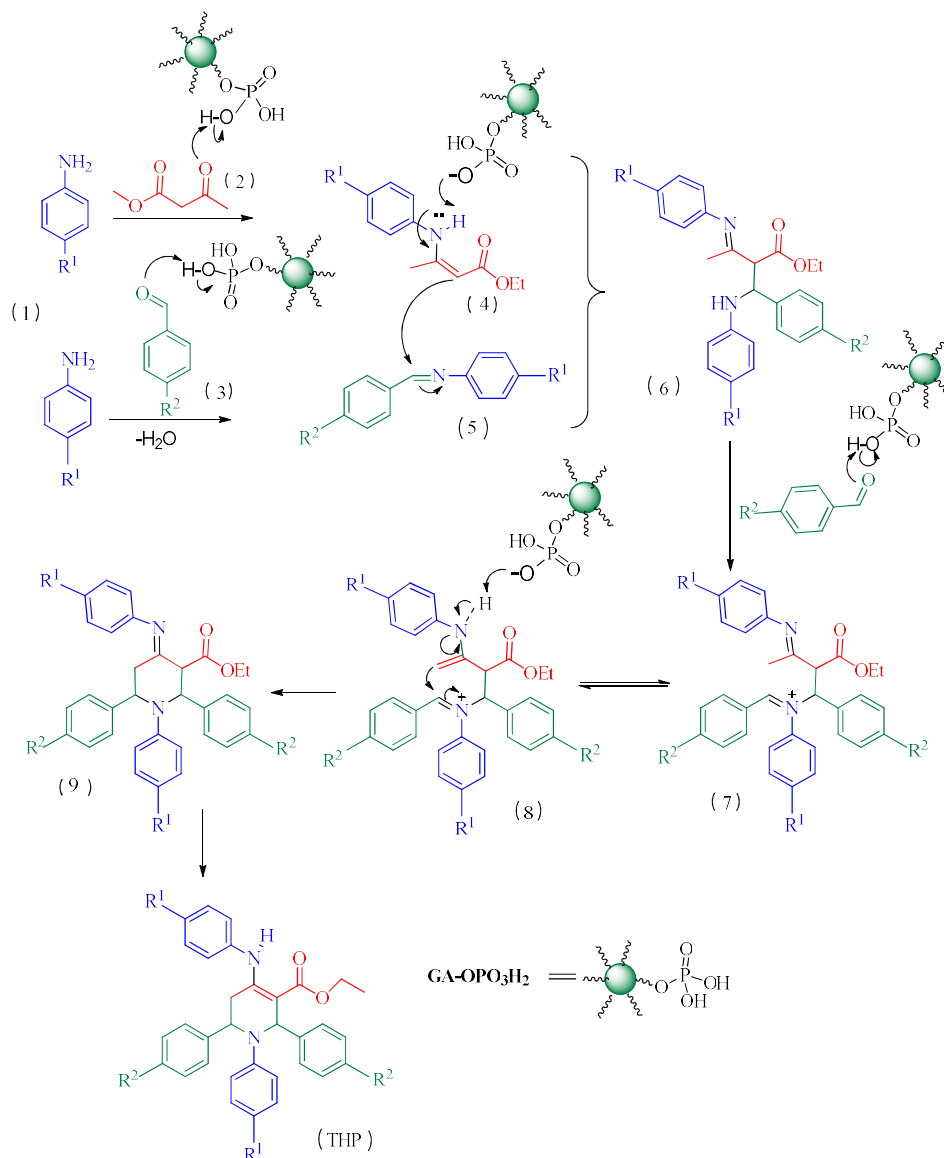
intermediate (7) by the elimination of H₂O. Then, tautomerization of (7) generates intermediate (8), which immediately undergoes intramolecular Mannich-type reaction to give intermediate (9). Finally, the intermediate (9) tautomerizes to generate the desired THP derivative containing conjugated ester group which bonded to NH with hydrogen bonding.

Table 3. Comparison Catalytic Performance of GA-OPO₃H₂ versus some other Catalysts for the Synthesis of Five-Substituted THP Derivatives

Entry	Solvent	Catalyst	Tem. (°C)	Time (h)	Yield (%) ^a	[Ref.]
1 ^c	Ethanol	ZrCl ₄ (0.15 mmol)	R.T. ^b	16	86	[35]
2 ^d	Ethanol	ZrOCl ₂ ·8H ₂ O (20 mol%)	Reflux	3	86	[17]
3 ^c	Ethanol	Fe(NO ₃) ₃ ·9H ₂ O (20 mol%)	R.T. ^b	7	88	[19]
4 ^c	MeOH	FeCl ₃ /SiO ₂ NPs (0.8 mol%)	Reflux	7	86	[20]
5 ^c	HOAc	HOAc (5 ml)	R.T. ^b	7	85	[22]
6 ^c	Ethanol	<i>p</i> -TsOH·H ₂ O (0.11 g)	R.T. ^b	12	88	[18]
7 ^c	CH ₃ CN	CAN (15 mol%)	R.T. ^b	35	68	[16]
8 ^c	CH ₃ CN	L-Proline/THF (20 mol%)	30	22	75	[1]
9 ^f	-	GA-OPO ₃ H ₂ (0.03 g)	80	4	87	[This work]

The amount ratio of aldehyde (mmol), aniline (mmol), ethyl acetoacetate or methyl acetoacetate (mmol) are equal to 2:2:1.

^aIsolated yield. ^bRoom temperature. ^cBenzaldehyde, 4-methylaniline and ethyl acetoacetate were used. ^dBenzaldehyde, 4-methylaniline and methyl acetoacetate were used. ^eBenzaldehyde, 4-chloroaniline and ethyl acetoacetate were used. ^f4-Chlorobenzaldehyde, 4-chloroaniline and ethyl acetoacetate were used.



Scheme 2. Proposed mechanism for the synthesis THP derivatives

CONCLUSIONS

In summery, we have reported a facile and green method for the synthesis of THPs *via* a *pseudo*-five-component reaction between para-substituted anilines, aromatic aldehydes and ethyl acetoacetate using GA-OPO₃H₂ as a natural-based heterogeneous catalyst under solvent-free conditions. GA-OPO₃H₂ does not need special cautions for preparation, handling or storage and act as Bronsted acid to

promote the reaction. This catalyst has shown good utility in accordance with the product yields, reusability, simple separation and benign reaction condition.

ACKNOWLEDGEMENTS

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