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ORIGINAL PAPER

Nano-pistachio hull-O_xTiCl_{4-x}: synthesis, characterization and application as an effective and novel nanocatalyst for one-pot synthesis of dihydropyrano[3,2-*b*]chromenedione derivatives

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Abstract

According to the Food and Agriculture Organization (FAO), Iran is the largest pistachio (*Pistacia vera*) producer in the world. Nevertheless, pistachio by-products (PBs) contain a high level of phenolic and cellulose compounds, it can be used as a substrate for the preparation of solid acid catalysts. In this work, the novel pistachio hull-O_xTiCl_{4-x} catalyst was synthesized via preparing pistachio hull, product of Kerman, as a support followed by treatment with titanium tetrachloride ($TiCl_4$) and identified by Fourier transform infrared spectroscopy, field emission scanning electron microscopy (FE-SEM), X-ray diffraction spectroscopy (EDX), thermogravimetric analysis (TG), Brunauer–Emmett–Teller (BET) and X-ray powder diffraction (XRD). The size of the nano-pistachio hull-O_xTiCl_{4-x} nanocatalyst was shown by a scanning electron microscope below 100 nm. The catalytic activity of the solid acid catalyst has been successfully examined in a one-pot, three-component condensation reaction of aromatic aldehydes, dimedone and kojic acid in refluxing ethanol to furnish dihydropyrano[3,2-*b*] chromendione derivatives. Consequently, pyran annulated heterocycles were obtained. The proposed approach has some advantages as excellent yields, mild reaction conditions, short reaction times, use of agricultural waste and eco-friendly.

Keywords Kojic acid · Nanocatalyst · Pistachio hull · Green biosynthesis · Solid acid

Introduction

Nanoscience and nanotechnology have considerable ecological interest as a very influential and efficient strategy in the field of medicinal organic, and combinatorial chemistry. Due to the unique dimensions and size, physical properties, easy recyclability, large specific surface area of nanostructures [1], there is widely current attention in nanoparticle application as catalysts, biomedical use, energy storage, enzyme encapsulation, photonics, and drug delivery [2–5]. During the past years, the use of recyclable heterogeneous catalyst (a solid catalyst that is dispersed on a supporting material to increase surface area and provide stability) has received significant importance in organic chemistry [6].

The pistachio hull is an agricultural by-product and would be an excellent bio-resource for preparing solid acids because of its high adsorption ability, low cost, and easy sustainability [7]. Much like other agricultural lignocellulosic biomass, such as coconut shell [8], sawdust [9], and peanut shell [10], pistachio hull is composed mainly of lignin, cellulose and hemicelluloses. Consequently, the main active sites of pistachio hull are a wide variety of hydroxyl groups that can be used for the preparation of solid acid catalysts [7].

The multi-component (three reactant or more) reactions (MCRs) have been developed in organic chemistry and modern drug discovery owing to the generation of biologically active products [11].

In addition, high atom economy, cost-effectively, energy saving, lower reaction time and raw materials, structural complexity and environmentally benign synthesis of chemically and biologically important organic frameworks are the most advantageous features encountered in MCRs [12–14]. Dihydropyrano[3,2-*b*]chromendione derivatives are some examples of multi-component synthesis that three starting materials react in a single flask [15–20].

Pyrans and its derivatives are an important category of oxygen-containing heterocycles which is present in a number of important pharmaceuticals and natural products such

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as alkaloids, carbohydrates, polyether antibiotics, iridoids and pheromones. Polyfunctionalized pyrans have attracted considerable attention because of their broad spectrum of significant biological and pharmaceutical properties such as antimicrobial, antifungal, anticancer, antioxidant, anti-tumor and anti-HIV [21–25].

Considering the importance of biologically active pyran templates, an efficient and environmentally benign approach was developed for the synthesis of dihydropyrano[3,2-*b*]chromendiones via one-pot three-component condensation of aromatic aldehydes, 5-hydroxy-2-hydroxymethyl-4*H*-pyran-4-one (kojic acid) and 5,5-dimethyl-1,3-cyclohexanedione(dimedone) in the presence of nano-pistachio hull-O_xTiCl_{4-x} as a readily available, highly efficient, recyclable and novel catalyst in EtOH as an inexpensive and non-toxic medium.

Experimental

General

The chemicals used in this work were purchased from Fluka (Buchs, Switzerland) and were used without further purification. Melting points were determined with an Electrothermal 9100 apparatus. IR spectra were recorded on a Shimadzu IR-470 spectrometer. ^1H and ^{13}C NMR spectra were recorded on Bruker DRX-400 Avance spectrometer at solution in $CDCL_3$ using TMS as internal standard. The morphologies of the nanoparticles were observed using FESEM of a MIRA3 TESCAN microscope with an accelerating voltage of 15 kV. The IR spectra of the catalyst were recorded using a model Bruker Tensor 27 Fourier transform infrared spectroscopy (FT-IR). The EDX analysis was done using a SAMx-analyzer. Thermogravimetric analysis was performed by using a TGA 209 F3Tarsus. X-ray diffraction (XRD) pattern was obtained in the 2θ range from 10 to 80 by a Philips Xpert MPD diffractometer equipped with a Cu Kα anode ($k = 1.54$ A°). Surface areas and pore size of nano-pistachio hull-O_xTiCl_{4-x} were undertaken using BELSORP-miniII from N_2 adsorption at relative pressure $P/P_0 = 0.99$ in the Brunauer–Emmet–Teller (BET) model.

Preparation of nano-pistachio hull-O_xTiCl_{4-x}

At first, the pistachio hull was collected from Kerman city of Iran and washed several times with deionized water to remove adhering impurities and then dried at 60 °C for 24 h. The dried pistachio hull was pulverized at the mortar and sieved using a 20-mesh sieve and labeled as pistachio hull. To prepare nano-pistachio hull-O_xTiCl_{4-x}, $TiCl_4$ (0.7 mL) was added dropwise over 10 min via a syringe to pistachio hull powder (1.0 g) in 5 mL of chloroform at 0 °. The reaction mixture was stirred and, after 60 min, the ashy powder was separated and washed with chloroform (15 mL) and *n*-hexane (15 mL), respectively. Subsequently, for obtaining a fine and homogenized pistachio hull-O_xTiCl_{4-x} powder, the obtained solid was dried in an oven at 70 °C for 4 h and then pulverized at the mortar.

General procedure for preparation of compounds 4a–m

Nano-pistachio hull-O_xTiCl_{4-x}(7.0 mg) were added to a stirred mixture of kojic acid **1** (1.0 mmol), dimedone **2** (1.0 mmol) and aromatic aldehyde **3** (1.0 mmol). The materials were mixed and heated under reflux in EtOH (5 mL) for the appropriate time as mentioned in Table 4. The progress of the reaction was followed by TLC (*n*-hexane/ethyl acetate 3:1). After completion of the reaction, the reaction mixture was filtered to remove the catalyst and then the crude product was recrystallized from hot ethanol to obtain the pure compound by evaporation of the solvent.

Results and discussion

In association with our recent achievement in the design of innovative recyclable heterogeneous nanocatalysts to the production of heterocycles [8–10], herein we studied synthesis of nano-pistachio hull-O_xTiCl_{4-x} as highly efficient novel heterogeneous nanocatalyst for the preparation of dihydropyrano[3,2-*b*]chromendiones. The novel pistachio hull-O_xTiCl_{4-x} catalyst was simply prepared as given in Scheme 1 and its characteristics instrumentally were evaluated.

As shown in Fig. 1, the average particle size of pistachio hull-O_xTiCl_{4-x} and its distribution were analyzed by FESEM. As shown in FESEM, the size of pistachio hull-O_xTiCl_{4-x} is below 100 nm, and on the other hand, the spots on the pistachio shell surface can be attributed to titanium tetrachloride groups (Fig. 1c). The spot EDX results indicate these observations.

As shown in Fig. 2, elemental composition of the pistachio hull and nano-pistachio hull-O_xTiCl_{4-x} was investigated by EDX analysis which is shown in Table 1. Pistachio hull is composed of C, O, K and Cl (Fig. 2a), while pistachio hull-O_xTiCl_{4-x} (Fig. 2b) is composed of C, O, K, Cl and Ti. The presence of Ti and increase in the percentage of Cl in the EDX indicate that the chemical interaction of titanium tetrachloride with the surface of pistachio hull.

Thermogravimetric analysis (TGA) and derivative thermogravimetric analysis (DTG) were performed to assess the thermal stability of nano-pistachio hull-O_xTiCl_{4-x}

$$\text{Pistachio hull-OH} + TiCl_4 \rightarrow \text{Pistachio hull-}O_xTiCl_{4-x} + HCl$$

Scheme 1 Preparation of nano-pistachio hull-O_xTiCl_{4-x}

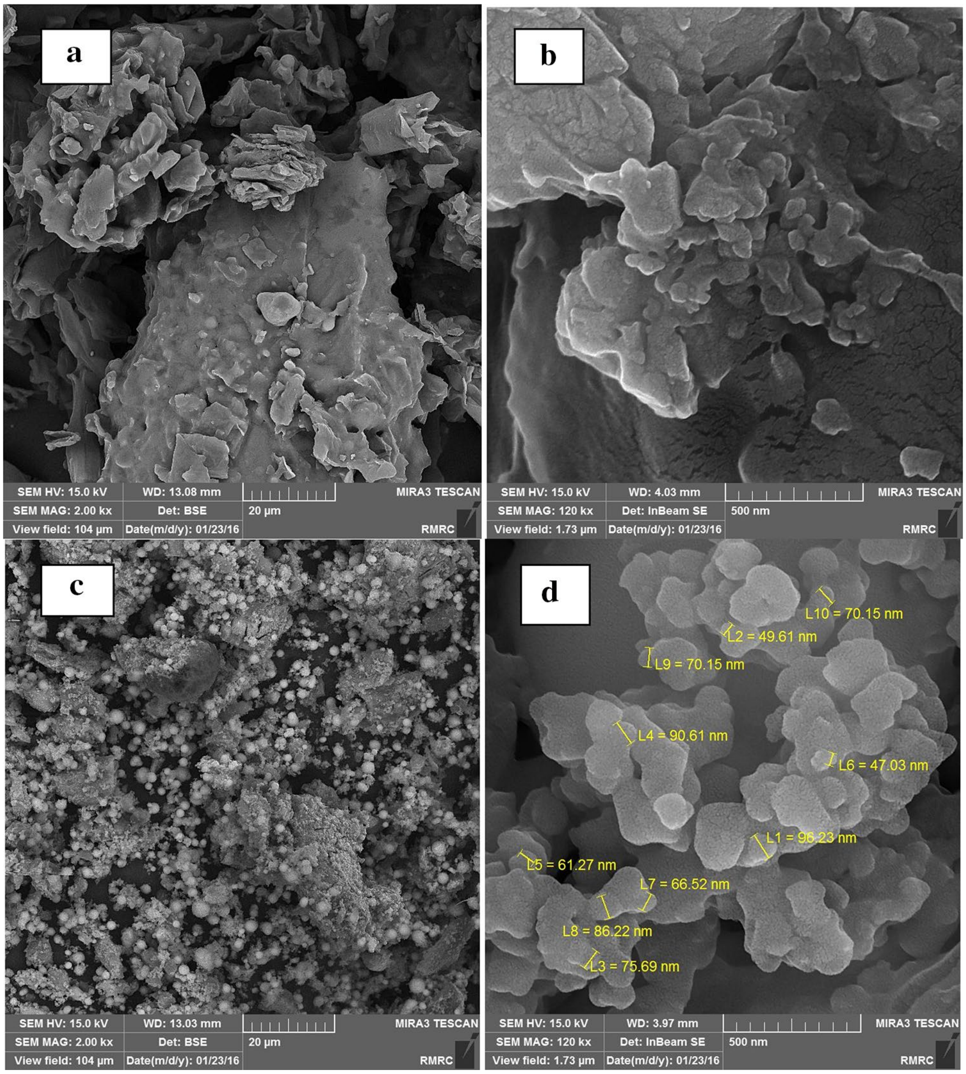


Fig. 1 **a**, **b** SEM micrograph of pistachio hull (**c**, **d**) SEM micrograph of nano-pistachio hull-O$_x$TiCl$_{4-x}$

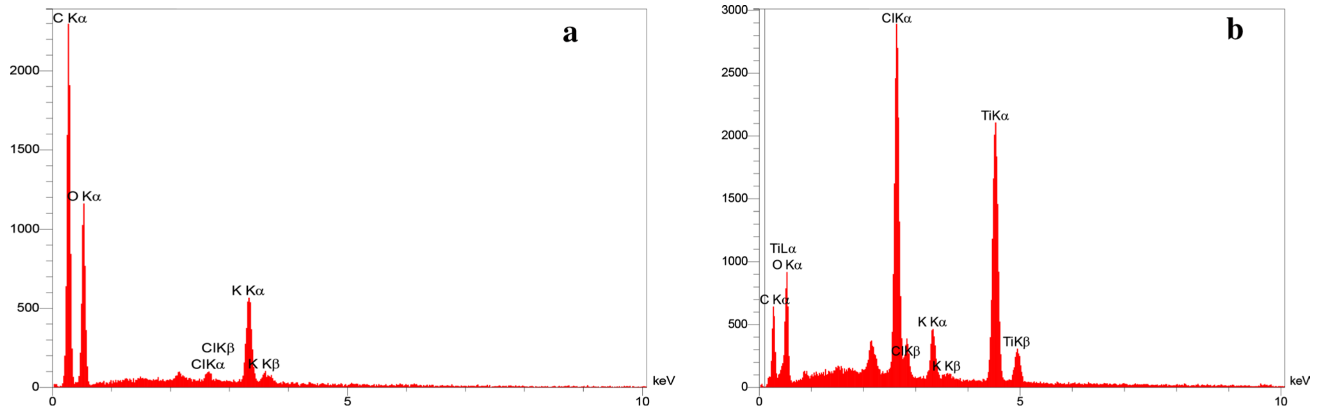


Fig. 2 **a** EDX of pistachio hull and **b** EDX of nano-pistachio hull-O$_x$TiCl$_{4-x}$

(Fig. 3). Three thermal decomposition mass loss steps are observed for the pistachio hull-O$_x$TiCl$_{4\text{-}x}$: (a) There was a 35.73% weight loss from 25 to 190 °C for sample assigned to desorption of H$_2$O/Cox molecules from the surface of nano-pistachio hull-O$_x$TiCl$_{4-x}$ and the further dehydration of pistachio (190 °C); (b) 23.18% weight loss at 200–290 °C is due

Table 1 Chemical analysis of pistachio shell and nano-pistachio hull-O_xTiCl_{4-x}

Element	Pistachio shell (W%)	Nano-pistachio hull-O_xTiCl_{4-x} (W%)
C	50.26	30.73
O	43.81	30.16
Cl	0.41	13.92
K	5.52	2.41
Ti	–	22.77

to the decomposition of cellulose; and (c) 18.80% weight loss in the range of 300–1000 °C is due to the decomposition of lignin units. (The first decomposition shoulder peak at about 300 °C is attributed to thermal depolymerization of hemicelluloses or pectin. The major second decomposition peak at about 390–400 °C is attributed to cellulose decomposition, and the small peak at 420 °C may be attributed to oxidative degradation of the charred residue. Lignin as a component of the fibers was degraded at a higher temperature. The structure of lignin is a highly aromatic polymer. Possibly, it was removed by benzene in larger amount than the other solvents). Based on nano-pistachio hull-O_xTiCl_{4-x} TG-DTG diagram, the substance is stable up to 144.5 °C; hence, the catalyst can be used at temperatures below 150 °C for promotion of organic synthesis. In the case of initial TiO_2-NPs, there was no further mass loss up to 900 °C [26].

In FT-IR spectrum of nano-pistachio hull-O_xTiCl_{4-x} exhibited a broad peak for the hydroxyl bands of cellulose units at 3446 cm^{-1} (Fig. 4). The C–H stretching vibrations of the aliphatic systems for cellulose observed at 2937 cm^{-1}. The absorption bands at nearly 1649 cm^{-1} indicate the C=C bond in phenyl rings of lignin unit. The C–O band was observed at 1152 cm^{-1}, and the Ti–O stretching vibrations observed at 673 cm^{-1}. The FT-IR data and the presence of these elements in the EDX vividly approved the actual event of chemical interaction of titanium tetrachloride with the surface area of pistachio hull.

The XRD patterns can be used to describe the crystallinity of nano-pistachio hull-O_xTiCl_{4-x} catalyst. The X-ray diffraction pattern for the nano-pistachio hull-O_xTiCl_{4-x} catalyst is shown in Fig. 5. The XRD pattern for the nano-pistachio hull-O_xTiCl_{4-x} catalyst displayed distinct peaks at $2\theta = 28.6°$, 40.7°, 50.4° and 66.6°, respectively. As can be seen, peaks indicate the presence of crystalline nano-pistachio hull structure and prove the bonding of Ti to the cellulose backbone. The XRD analysis of the prepared sample of TiO_2 nanoparticles was done using a Bruker make diffractometer, Cu-Kα X-rays of wavelength $(\lambda) = 1.5406$ Å, and data were taken for the 2θ range of 10° to 70° with a step of 0.1972° [27]. The results confirmed the nano-pistachio hull-O_xTiCl_{4-x} catalyst and are not TiO_2.

Brunauer–Emmett–Teller (BET) theory aims to explain the physical adsorption of gas molecules on a solid surface and serves as the basis for an important analysis technique for the measurement of the specific surface area of materials. In order to further understand the nature and geometry of the pores formed, BET adsorption–desorption isotherms were studied and analyzed for the as-synthesized nano-pistachio hull-O_xTiCl_{4-x} catalyst. The adsorption–desorption curves show a pores following H2 hysteresis of the IUPAC classification. The adsorption isotherm increases gradually until a relative P/Po of 0.6 followed by a change in slope at the

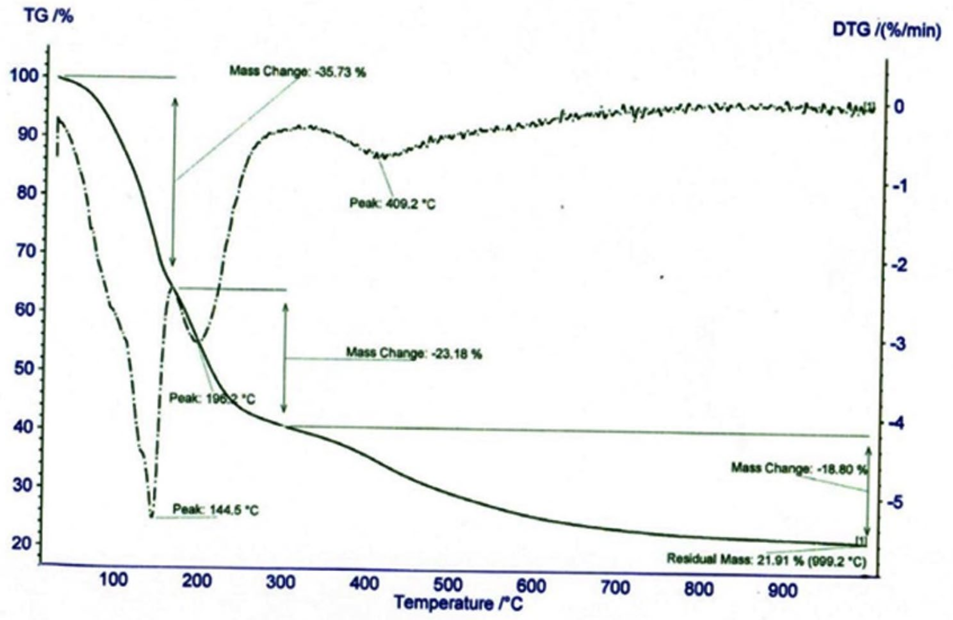


Fig. 3 TGA/DTG curves of nano-pistachio hull-O_xTiCl_{4-x} samples

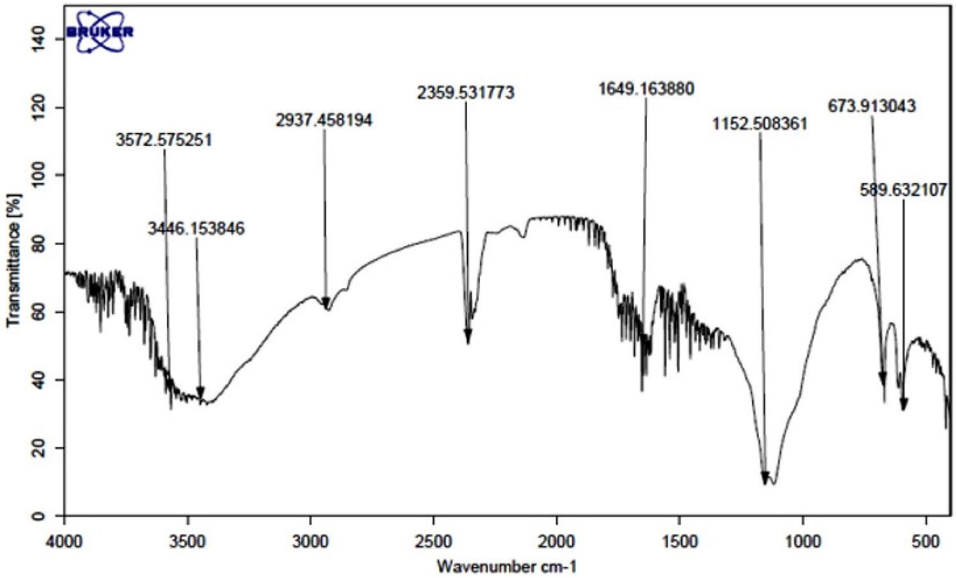


Fig. 4 FT-IR spectrum of nano-pistachio hull-O_xTiCl_{4-x} catalyst

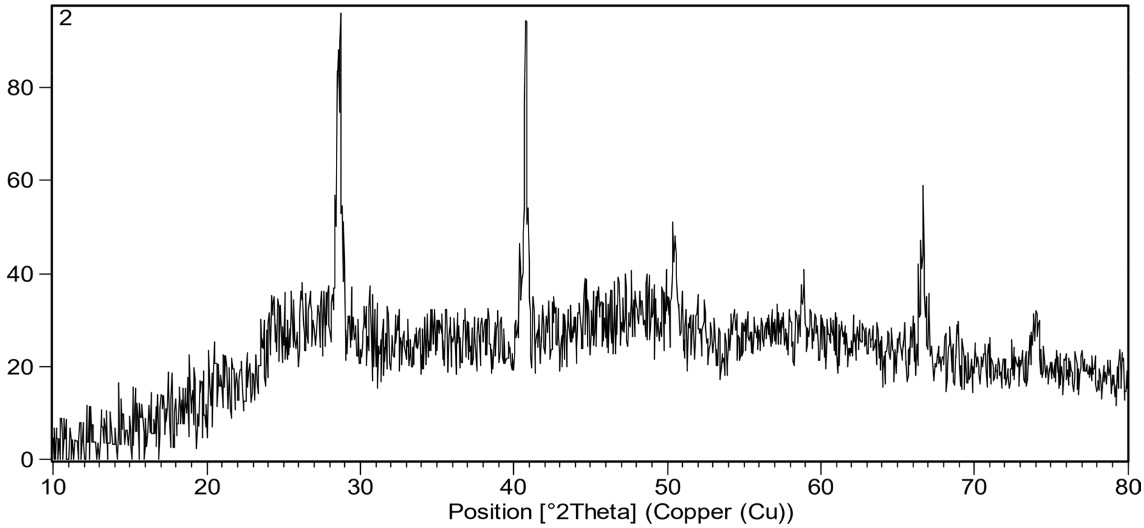


Fig. 5 X-ray diffraction pattern of nano-pistachio hull-O_xTiCl_{4-x} catalyst

curve beyond 0.6 which is attributable to the condensation taking place at the neck and the interconnected inner-bottled structure. However, the desorption curves show pores following H4 hysteresis of the IUPAC classification (Table 2, Fig. 6).

After synthesis and characterization of the nano-pistachio hull-O_xTiCl_{4-x} as a novel, stable and eco-friendly nano-catalyst, we investigated the performance of nano-pistachio hull-O_xTiCl_{4-x} in the synthesis of dihydropyrano[3,2-*b*] chromenediones. First, a test model reaction was done with 1.0 mmol benzaldehyde, 1.0 mmol kojic acid and 1.0 mmol dimedone in the existence of various amounts of nano-pistachio hull-O_xTiCl_{4-x} using different solvents. The results of these experiments are given in Table 3. In the absence of nano-pistachio hull-O_xTiCl_{4-x}, no desired product was formed (Table 3, entry 1). The favorable result was found

Table 2 BET results of nano-pistachio hull-O_xTiCl_{4-x} catalyst

Sample weight (g)	0.0109
Adsorption temperature (k)	77
Saturated vapor pressure (KPa)	88
Average pore diameter (nm)	14.481
Total pore volume (cm^3 g^{-1})	1.1368
AS, BET (m^2 g^{-1})	313.99

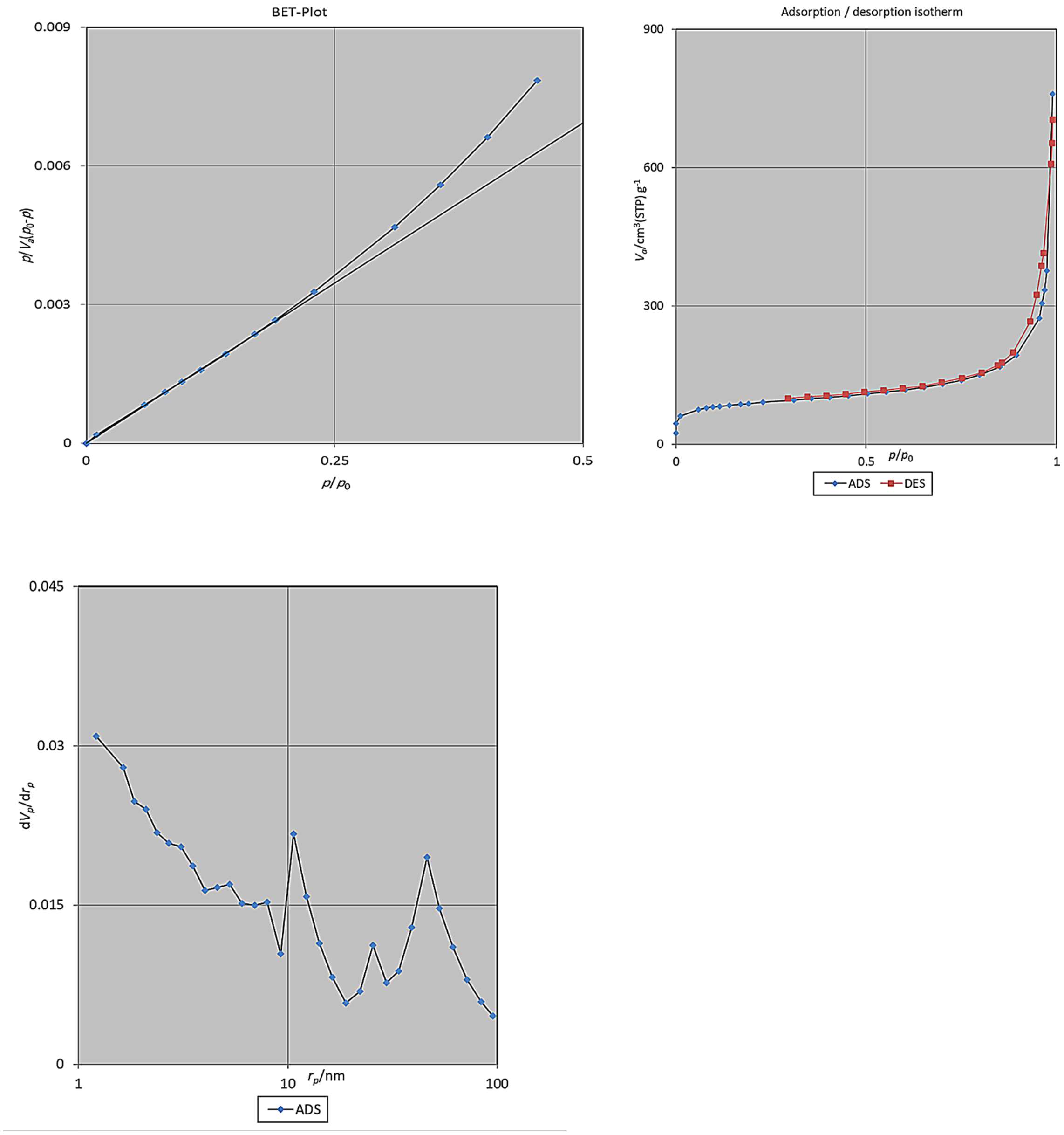


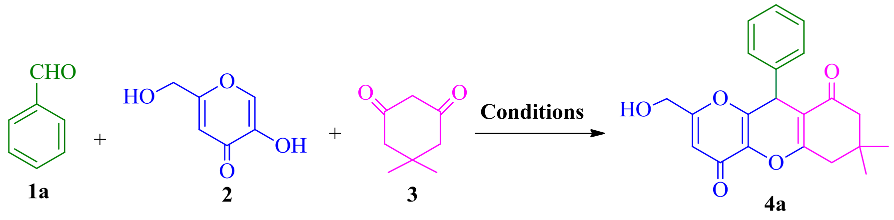
Fig. 6 BET adsorption–desorption isotherm and pore size desorption curve

with 7.0 mg nano-pistachio hull-O_xTiCl_{4-x} in refluxing ethanol (Table 3, entry 2). A remarkable advantage of this technique is that the catalyst can be recovered at the end of the reaction and can be used several times without losing its activity. To regenerate the nano-pistachio hull-O_xTiCl_{4-x}, after completion of the reaction, the mixture was filtered and recrystallized from hot ethanol; catalyst was separated and washed with ethanol and then dried to obtain the solid remainder. This procedure repeated for two cycles and the yield of product **4a** did not change significantly (Table 3, entries 9, 10).

After optimizing tests, we conducted reaction of kojic acid and dimedone with a variety of substituted aldehydes under the optimized condition for the synthesis of dihydropyrano[3,2-*b*] chromenedione derivatives (Scheme 2). Results are given in Table 4 indicating that different aromatic aldehydes substituted with either electron-donating or electron-withdrawing groups could react with kojic acid and dimedone smoothly and give products in excellent yield.

A detailed reaction mechanism for the synthesis of dihydropyrano[3,2-*b*]chromenediones(**4**) using nano-pistachio hull-O_xTiCl_{4-x}, which act as a solid acid catalysis in all steps is outlined in Scheme 3 [30, 31]. Initially, nano-pistachio hull-O_xTiCl_{4-x} activates the dimedone and aldehyde to form the olefin (**5**). The Michael addition of kojic acid

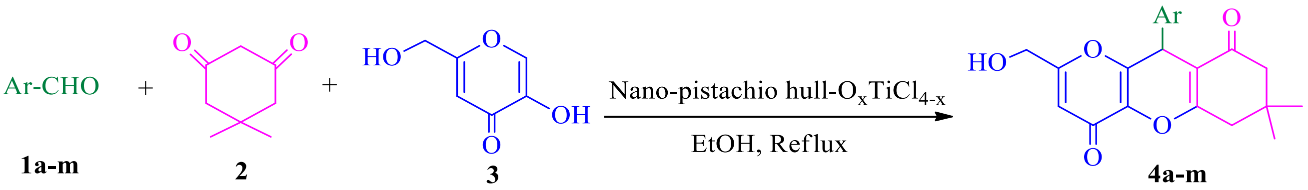
Table 3 Optimization of the reaction conditions for synthesis of **4a**



Entry	Catalyst (mg)	Solvent	Time (min)	Yield[a] (%)
1	Nano-pistachio hull-O_xTiCl_{4-x}(0.0)	EtOH	120	0
2	Nano-pistachio hull-O_xTiCl_{4-x}(7.0)	EtOH	20	95
3	Nano-pistachio hull-O_xTiCl_{4-x}(7.0)	*n*-Hexane	20	Trace
4	Nano-pistachio hull-O_xTiCl_{4-x}(7.0)	H_2O	20	90
5	Nano-pistachio hull-O_xTiCl_{4-x}(7.0)	CH_3CN	20	45
6	Nano-pistachio hull-O_xTiCl_{4-x}(3.0)	EtOH	20	67
7	Nano-pistachio hull-O_xTiCl_{4-x}(5.0)	EtOH	20	79
8	Nano-pistachio hull-O_xTiCl_{4-x}(9.0)	EtOH	20	95
9	Nano-pistachio hull-O_xTiCl_{4-x}(7.0) 2nd run	EtOH	20	92
10	Nano-pistachio hull-O_xTiCl_{4-x}(7.0) 3rd run	EtOH	20	89

Reaction condition: benzaldehyde (1.0 mmol), kojic acid (1.0 mmol) and dimedone (1.0 mmol) under reflux in various solvents

[a] Isolated yields



Scheme 2 Synthesis of dihydropyrano[2,3-*b*] chromenedione derivatives in the presence of nano-pistachio hull-O_xTiCl_{4-x} as a catalyst

(**3**) with olefin (**5**) in the presence of nano-pistachio hull-O_xTiCl_{4-x} finally gives intermediate (**7**), which then makes the inner molecular ring (**8**) to be formed after dehydration in the existence of catalyst to produce the corresponding product (**4**).

To show the merit of the present work in comparison with the reported results in the literature, we compared the results of nano-pistachio hull-O_xTiCl_{4-x} as catalyst with Alum [28], $InCl_3$ [29], $CeCl_3{\cdot}7H_2O$ [32], $Bi(OTf)_3$ [33], $FeCl_3{\cdot}SiO_2$ [34] and nano-Bi_2O_3-ZnO [35] in the synthesis of dihydropyrano[3,2-*b*]chromenediones. As shown in Table 5, entry 1, this new catalytic system compared to previous similar works has a number of advantages such as shorter reaction time, higher yields, lower reaction temperature, use of agricultural waste and most importantly, avoidance of hazardous or toxic catalysts and organic solvents. In all these cases, our work has introduced green and more economically cost-effective relative to previous similar works.

Conclusions

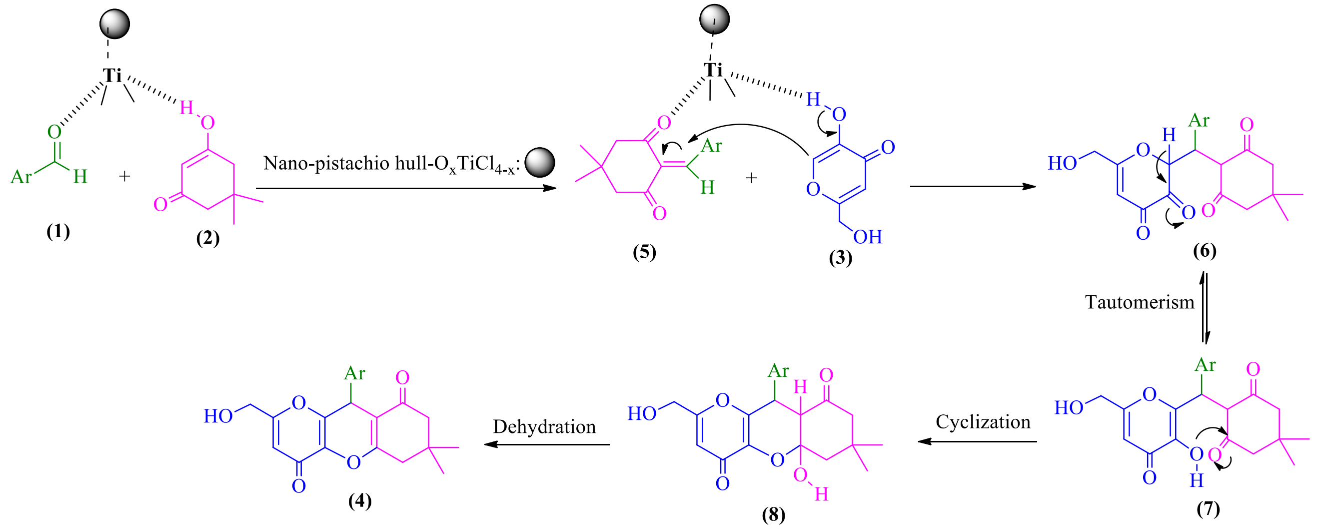
To conclude, we have introduced a well-organized, eco-friendly, and simple procedure for the efficient synthesis of dihydropyrano[3,2-*b*]chromenedione derivatives in high-to-excellent yields via a one-pot three-component reaction by using nano-pistachio hull-O_xTiCl_{4-x} as a mild, effective, non-toxic and inexpensive solid acid catalyst in ethanol without the addition of organic co-solvent. The promising points for the presented methodology are environmental acceptability, economic viability, easy work-up, short reaction time, high atom economy and finally compliance with the green chemistry protocols. Meanwhile, solid-phase acidic catalyst could be reused for a number of times without appreciable loss of activity. Moreover, our work is expected to show interesting pharmacology activities and may act as potential drug candidates, since

Table 4 Synthesis of dihydropyrano[3,2-*b*] chromenedione derivatives

Entry	Ar	Product	Time (min)	Yield[a]	M.P.(ºC) [Ref.]
1	C_6H_5	**4a**	20	95	185–186 [28]
2	4-Cl-C_6H_4	**4b**	20	92	204–206 [28]
3	3-Cl-C_6H_4	**4c**	20	91	196–197 [29]
4	2-Cl-C_6H_4	**4d**	20	93	215–217 [28]
5	4-F-C_6H_4	**4e**	18	92	160–162 [28]
6	3-NO_2-C_6H_4	**4f**	15	96	212–213 [28]
7	4-NO_2-C_6H_4	**4g**	15	95	230–232 [28]
8	2,4-Cl_2-C_6H_3	**4h**	20	90	164–166 [28]
9	2,4,6-$(Me)_3$-C_6H_2	**4i**	25	86	197–199 [29]
10	4-Me-C_6H_4	**4j**	25	89	216–218 [28]
11	3,4,5-$(MeO)_3$-C_6H_2	**4k**	30	87	173–175 [28]
12	4-MeO-C_6H_4	**4l**	30	87	176–178 [28]
13	2,5-$(MeO)_2$-C_6H_3	**4m**	30	88	190–192 [28]

Reaction condition: aldehyde (1.0 mmol), kojic acid (1.0 mmol), dimedone (1.0 mmol) and nano-pistachio hull-O_xTiCl_{4-x} (7.0 mg) in ethanol (5 mL) under reflux condition

[a] Isolated yields



Scheme 3 Suggested mechanism for the synthesis of dihydropyrano[2,3-*b*] chromenediones in the presence of nano-pistachio hull-O_xTiCl_{4-x} as a catalyst

Table 5 Comparison of the catalytic performance of nano-pistachio hull-O_xTiCl_{4-x} with other catalysts to the production of dihydropyrano[3,2-*b*] chromenedione derivatives

Entry	Catalyst (amount)	Temp. (ºC)/solvent	Time (min)	Yield (%)	Ref.
1	Nano-pistachio hull-O_xTiCl_{4-x} (7.0 mg)	Reflux/EtOH	15–30	86–96	This work
2	Alum (0.1 mmol)	100/–	40–70	85–95	[28]
3	$InCl_3$ (10 mol%)	120/–	80–95	85–95	[29]
4	$CeCl_3{\cdot}7H_2O$ (0.05 mmol)	110/–	30–60	86–96	[32]
5	$Bi(OTf)_3$ (0.05 mmol)	120/–	20–45	85–95	[33]
6	$FeCl_3{\cdot}SiO_2$ (0.05 mmol)	100/–	40–80	84–96	[34]
7	Nano-Bi_2O_3-ZnO (0.03 g)	100/–	60–120	75–84	[35]

pyran and chromene motifs have a vast range of biological activities.

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