

## Nano-peanut shell-OSO<sub>3</sub>H: Green and natural-based renewable nanocatalyst for one-pot synthesis of dihydropyrano[3,2-*b*]chromenedione derivatives

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Received 28 October 2018; received in revised form 16 February 2019; accepted 10 March 2019

### ABSTRACT

Nano-peanut shell-OSO<sub>3</sub>H nanoparticles were simply prepared and characterized by the scanning electron microscopy (SEM), transmission electron microscope (TEM), fourier transform infrared (FT-IR), energy dispersive X-ray spectroscopy (EDX), and thermal analysis techniques (TGA/DTG). The catalytic activity of nano-peanut shell-OSO<sub>3</sub>H was studied in the synthesis of biologically important 4*H*-pyran derivatives. The reaction involves the use of Kojic acid, 5,5-dimethyl-1,3-cyclohexanedione, and various benzaldehydes. A wide range of benzaldehydes is compatible in this reaction, producing excellent yields in a short time. The successful synthesis of dihydropyrano[3,2-*b*]chromenedione derivatives shows that nano-peanut shell-OSO<sub>3</sub>H has high catalytic activity and can be effectively used as a green catalyst.

**Keywords:** Nano-peanut shell-OSO<sub>3</sub>H, Dihydropyrano[3,2-*b*]chromenedione, Kojic acid, Green nanocatalyst.

### 1. Introduction

Multicomponent reactions (MCRs) have attracted remarkable attention as a powerful means to synthesize medicinal and natural compounds [1,2]. The MCR strategy presents an efficient and straightforward path to produce complexity and diversity in a single operation [3,4]. A large number of multicomponent reactions demonstrate benefits in atomic economy, environmental friendliness, simplified steps, optimal and useful application of resources [5-7]. Synthesis of kojic acid derivatives such as dihydropyrano [3,2-*b*]chromenediones are some examples of multicomponent reactions. Kojic acid derivatives are attractive, because they generally show biological activities and pharmacological properties [8-9].

Nowadays, replacing liquid acids with solid acids is desirable as a “green” approach to process the chemicals in industrial transformations [10]. Recently, sulfated and sulfonated solid acid catalysts were used as important catalysts for various transformations in the fine and industrial production of chemicals [11].

The peanut shell (PS) is an agricultural waste and would be an excellent bio-resource for preparing solid acids because of its high supply, low cost, and easy sustainability. Much like other agricultural lignocellulosic biomass, peanut shells are composed mainly of lignin, cellulose and hemicellulose. Consequently, peanut shells consist of a wide variety of hydroxyl groups that can be used as active sites for the preparation of solid acid catalysts. Several papers [12-14] have reported the preparation of solid acid catalysts by the sulfonation of the partially carbonized peanut shell and shown their high activity and excellent recyclability for biodiesel production and esterification reactions.

In this study, the peanut shell (PS) has been applied as adsorbent for the preparation of nano-peanut shell-OSO<sub>3</sub>H (nano-PS-OSO<sub>3</sub>H) whose average size is small and is well distributed. The presence of functional groups on the surface of PS-OSO<sub>3</sub>H resulted in a dramatic increase of surface polarity and acidity, thereby improving the catalytic efficiency of the nano-PS-OSO<sub>3</sub>H.

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In recent years, several methods have been reported for the synthesis of dihydropyrano[3,2-*b*]chromenedione using catalysts such as indium(III) chloride [15], alum [16], silica-supported ferric chloride [17], ceric ammonium nitrate [18].

Despite the efficiency of these procedures, some methods suffer from drawbacks such as the use of an expensive, toxic, water intolerant catalyst, low yield, long reaction time, harsh reaction conditions, complex working and purification procedures. Therefore, the development of a facile, efficient, and environmentally benign method for the preparation of 4*H*-pyrans has become an interesting challenge.

On the basis of our successful experiments on the use of solid acids in organic synthesis [19–25], we prepared peanut shell-OSO<sub>3</sub>H nanoparticles and used it as an efficient and green catalyst for the synthesis of dihydropyrano[3,2-*b*]chromenedione derivatives (Scheme 1).

## 2. Experimental

### 2.1. General

All chemicals were purchased from Fluka or Merck Chemical Companies. The known products were identified by comparison of their melting points and spectral data with those reported in the literature. Melting points were determined with an Electrothermal 9100 apparatus. <sup>1</sup>H and <sup>13</sup>C NMR spectra were recorded on Bruker DRX-400 Avance spectrometer for solutions in CDCl<sub>3</sub>. The morphologies of the nanoparticles were observed using FESEM of a MIRA3 TESCAN microscope with an accelerating voltage of 15 kV. Transmission electron microscopy (TEM) experiment was conducted on a Philips CM10 microscope. The IR spectra of the catalyst were recorded using a model Bruker Tensor 27 FT-IR. The EDX analysis was done using a SAMx-analyzer. Thermogravimetric analysis was performed using TG 209 F1 (Netzsch Germany).

### 2.2. Preparation of nano-peanut shell-OSO<sub>3</sub>H

Peanut shells were washed several times to remove adhering dirt and then dried at 60 °C for 24 h. The dried peanut shells were ground to pass through a 1 mm sieve and labeled as peanut shell. To prepare nano-PS-

OSO<sub>3</sub>H, chlorosulfonic acid (2.33 g) was added dropwise over 15 min via a syringe to peanut shell powder (5 g) in a 50 ml flask at 0 °C. The reaction mixture was stirred and, after 40 min, the ashy powder was separated and washed with water (15 mL) and ethyl acetate (15 mL) respectively. The obtained solid was dried in an oven at 80 °C and then pulverized at the mortar.

### 2.3. General procedure for the preparation of dihydropyrano[3,2-*b*]chromenediones (4*a*-4*j*)

Nano-PS-OSO<sub>3</sub>H (0.08 g) was added to a stirred mixture of the aromatic aldehyde (1 mmol), dimedone (1 mmol) and kojic acid (1 mmol) at 120 °C for a specified time (Table 3). After the completion of the reaction (as monitored by TLC), ethyl acetate (15 mL) was added to the reaction mixture. The mixture was filtered to remove the catalyst. Then water (15 mL) was added to the solution and extracted with ethyl acetate (2×10 mL). The organic layer was dried over sodium sulfate and concentrated under vacuum. The crude product was chromatographed in silica gel (70:30 *n*-hexane/ethyl acetate) and afford the pure product 4.

## 3. Results and Discussion

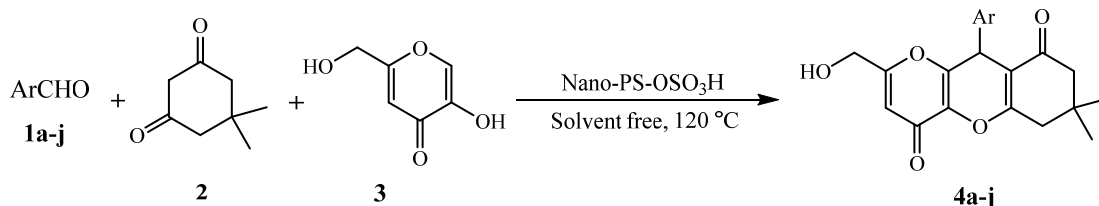
### 3.1. Preparation and characterization of nano-PS-OSO<sub>3</sub>H

Initially, nano-PS-OSO<sub>3</sub>H was synthesized using chlorosulfonic acid and peanut shells (PS) as a green, inexpensive and available surface and characterized by SEM, TEM, FT-IR, TGA and EDX analyses.

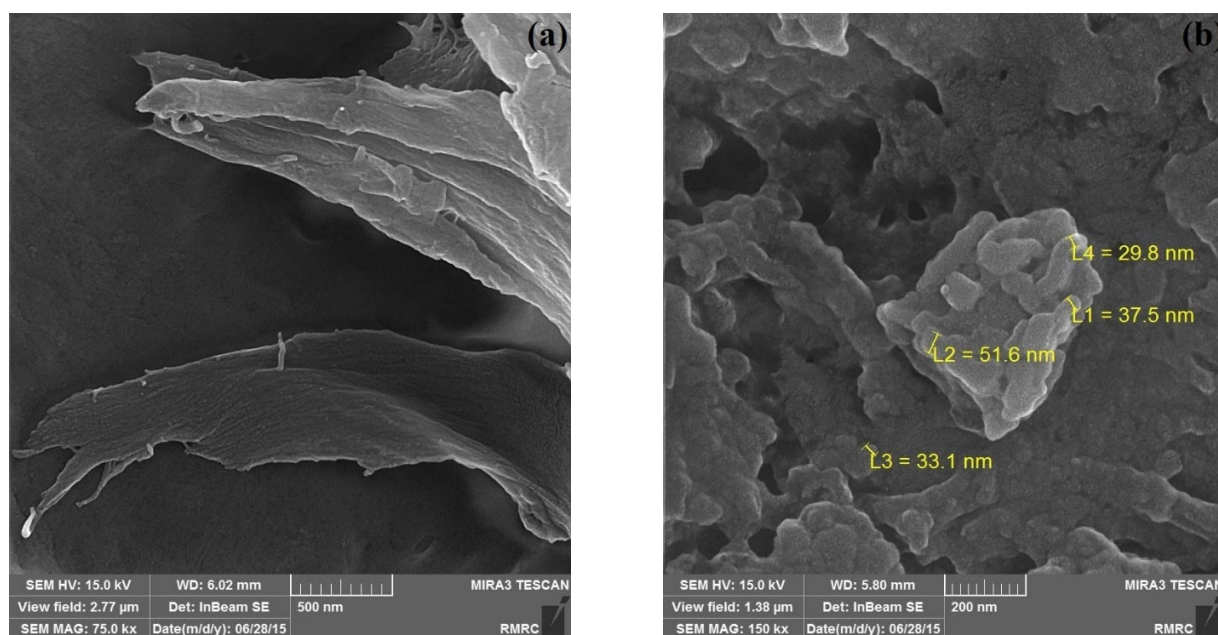
The morphology and size of PS and nano-PS-OSO<sub>3</sub>H were observed by scanning electron microscopy (SEM) images (Fig. 1). The SEM of nano-PS-OSO<sub>3</sub>H indicates that nanoparticles are homogeneous and small with an average size about 40 nm (Fig. 1b).

TEM image of nano-PS-OSO<sub>3</sub>H is shown in Fig. 2. It is clearly seen on the TEM image of catalyst that the average size of nanoparticle is below 30 nm.

In FT-IR spectrum of nano-PS-OSO<sub>3</sub>H (Fig. 3), the hydroxyl bands of peanut shells and sulfonic acid appeared at 3446 cm<sup>-1</sup>. The C–H stretching vibrations of the aliphatic systems for cellulose and hemicelluloses units were observed at 2938 cm<sup>-1</sup>.



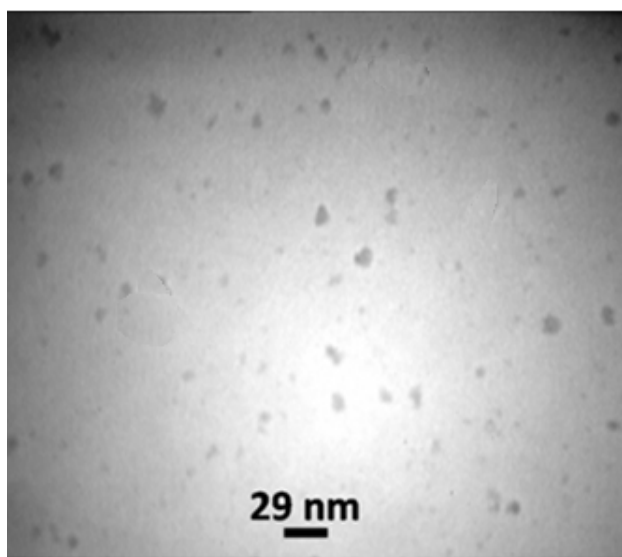
**Scheme 1.** Synthesis of dihydropyrano[3,2-*b*]chromenedione derivatives in the presence of nano-PS-OSO<sub>3</sub>H as the catalyst.



**Fig. 1.** SEM micrograph of (a) peanut shells (PS) and (b) nano-PS-OSO<sub>3</sub>H.

The C=C stretching vibrations of the phenyl rings in the lignin unit were viewed at 1653 cm<sup>-1</sup>. The symmetric and asymmetric O=S=O stretching vibrations appeared at 1058 and 1233 cm<sup>-1</sup>. The S-O stretching vibrations were observed at 880 cm<sup>-1</sup>.

The results of EDX analyses of the peanut shells and nano-PS-OSO<sub>3</sub>H were given in Table 1. The EDX analysis show how the different elements are distributed. Chemical analysis of peanut shells gave carbon and oxygen as the major elements (Fig. 4a). The elemental analysis by EDX identified the high peak of sulfur (Fig. 4b), that reveals the chemical interaction of chlorosulfonic acid with the surface of peanut shells.



**Fig. 2.** TEM image of nano-PS-OSO<sub>3</sub>H.

Thermal gravimetric analysis (TGA) and derivative thermogravimetric analysis (DTG) of the nano-PS-OSO<sub>3</sub>H were shown in Fig. 5. The loss in the weight of the nano-PS-OSO<sub>3</sub>H particles with rising temperature up to 540 °C was performed under nitrogen atmosphere. The first decrease of weight (mass) was assigned to the catalyst moisture removal (at 183 °C, 9.9% weight loss) while the second decrease showed the decomposition of cellulose in the nanocatalyst (309 °C, 13.12% weight loss). The third decrease showed the decomposition of lignin (446 °C, 19.31% weight loss). This catalyst did not show any degradation below 200 °C; therefore, it can act as an effective resistant biodegradable nano-catalyst in the organic reaction.

### 3.2. Catalytic activity of nano-PS-OSO<sub>3</sub>H

After synthesis and characterization of the catalyst, we investigated the catalytic activity of solid acid nanocatalyst for synthesis of dihydropyrano [3,2-*b*]chromenedione, by the condensation of various benzaldehydes, 5,5-dimethyl-1,3-cyclohexanedione (dimedone), kojic acid (Scheme 1).

To optimize quantity of nano-PS-OSO<sub>3</sub>H and reaction conditions, a model experiment was carried out using kojic acid, dimedone and benzaldehyde. When the model reaction was examined in the absence of catalyst at 120 °C, no desired product was formed even after 4 h (Table 2, entry 1), but in the presence of nano-PS-OSO<sub>3</sub>H (0.1 g) after 120 min a high yield of the expected dihydropyrano[3,2-*b*]chromenediones **4a** was obtained, showing the role of catalyst in this reaction (Table 2, entry 2).

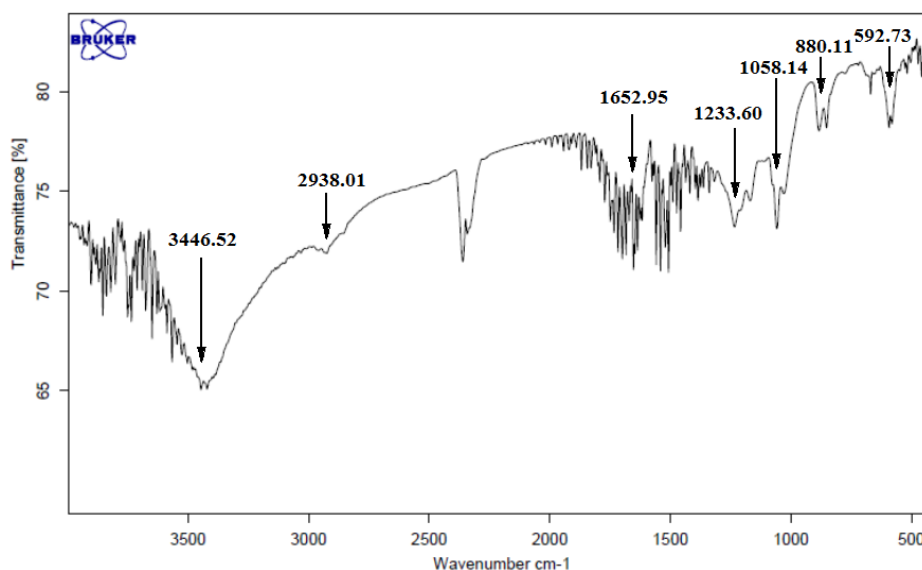


Fig. 3. FT-IR spectra of nano-PS-OSO<sub>3</sub>H.

Table 1. Chemical analysis of PS and nano-PS-OSO<sub>3</sub>H.

Element	PS		Nano-PS-OSO <sub>3</sub> H	
	W %	A %	W %	A %
C	62.94	69.68	50.87	60.56
O	36.06	29.97	39.22	35.06
S	1.01	0.34	9.49	4.23

The model reaction was also studied in the presence of chlorosulfonic acid and peanut shells; no satisfactory result was achieved under these catalytic conditions (Table 2, entries 3-4). The above reaction was also examined in various solvents such as CH<sub>2</sub>Cl<sub>2</sub>, EtOH, CH<sub>3</sub>CN and H<sub>2</sub>O in reflux conditions (Table 2, entries 5–8). Most of these solvents required a longer time and gave lower yields than solvent free conditions (Table 2, entry 2). Similar reactions were then attempted

using 0.05, 0.08 and 0.15 g of nano-PS-OSO<sub>3</sub>H (Table 2, entries 9–11). It was found that utilizing 0.08 g nano-PS-OSO<sub>3</sub>H under solvent free condition at 120 °C is sufficient to complete the reaction with the maximum yield (Table 2, entry 10). Increasing the amount of catalyst did not change the yield, whereas reduction of it significantly decreased the product yield (Table 2, entry 11). The effect of temperature was also investigated.

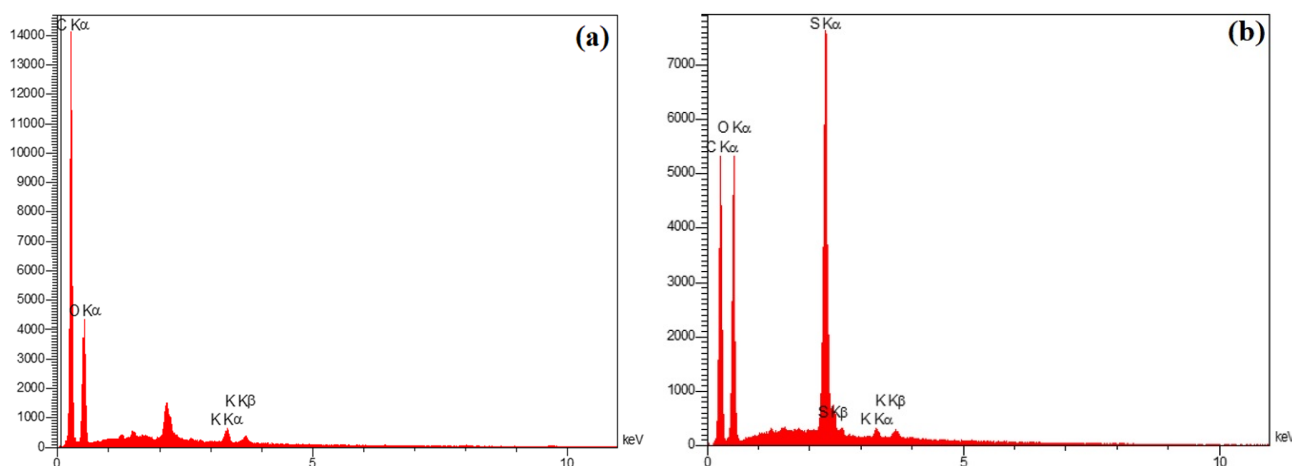


Fig. 4. EDX patterns of (a) peanut shells and (b) EDX of nano-PS-OSO<sub>3</sub>H.

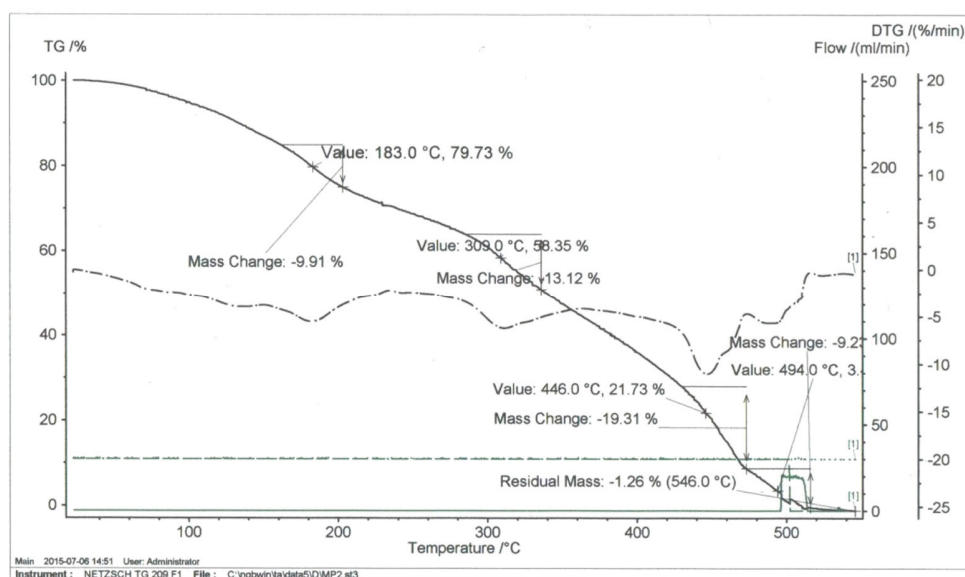
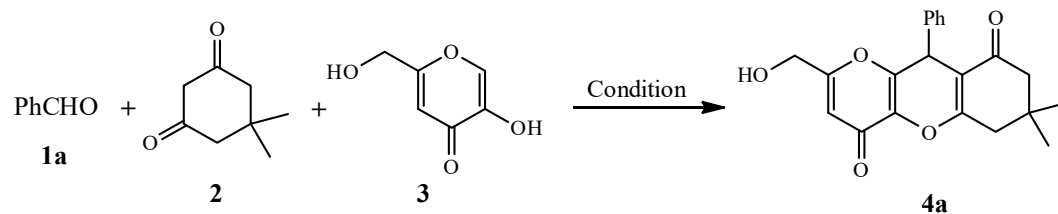


Fig. 5. The TGA/DTG curves of nano-PS-OSO<sub>3</sub>H.

Table 2. Optimization of reaction conditions for the synthesis of dihydropyrano[3,2-*b*] chromenedione **4a**.<sup>a</sup>



Entry	Catalyst (g)	Solvent/Condition	Time (h)	Yield (%) <sup>b</sup>
1	-	-/120 °C	4	-
2	Nano-PS-OSO <sub>3</sub> H (0.1)	-/120 °C	2	90
3	HSO <sub>3</sub> Cl	-/120 °C	2	60
4	Peanut shell	-/120 °C	2	trace
5	Nano-PS-OSO <sub>3</sub> H (0.1)	CH <sub>2</sub> Cl <sub>2</sub> <sup>c</sup> /reflux	2	35
6	Nano-PS-OSO <sub>3</sub> H (0.1)	EtOH <sup>c</sup> /reflux	2	40
7	Nano-PS-OSO <sub>3</sub> H (0.1)	CH <sub>3</sub> CN <sup>c</sup> /reflux	2	45
8	Nano-PS-OSO <sub>3</sub> H (0.1)	H <sub>2</sub> O <sup>c</sup> /reflux	2	10
9	Nano-PS-OSO <sub>3</sub> H (0.15)	-/120 °C	2	90
10	Nano-PS-OSO <sub>3</sub> H (0.08)	-/120 °C	2	90
11	Nano-PS-OSO <sub>3</sub> H (0.05)	-/120 °C	2	70
12	Nano-PS-OSO <sub>3</sub> H (0.08)	-/80 °C	2	60
13	Nano-PS-OSO <sub>3</sub> H (0.08) <sup>2nd run</sup>	-/120 °C	2	87
14	Nano-PS-OSO <sub>3</sub> H (0.08) <sup>3rd run</sup>	-/120 °C	2	83
15	Nano-PS-OSO <sub>3</sub> H (0.08) <sup>4th run</sup>	-/120 °C	2	80

<sup>a</sup>Reaction conditions: Reactants **1a**, **2** and **3** (1.0 mmol).

<sup>b</sup>Yields refer to isolated products.

<sup>c</sup>7 mL solvent.

It was observed that decreasing temperature to 80 °C is not appropriate for nano-PS-OSO<sub>3</sub>H catalyzed reaction and leads to reducing the reaction rate and increasing the reaction time (Table 2, entry 12). Therefore, the best result was obtained by the use of substrates in the equimolar ratio and 0.08 g nano-PS-OSO<sub>3</sub>H as catalyst under solvent free conditions at 120 °C.

Encouraged by these remarkable results, and in order to investigate the generality and scope of this new protocol, a variety of dihydropyrano[3,2-*b*]chromenedione derivatives were synthesized by treating substituted benzaldehyde with kojic acid and dimedone in presence of the catalytic amount of nano-PS-OSO<sub>3</sub>H under solvent free conditions at 120 °C.

When kojic acid and dimedone reacted with benzaldehydes including halogen group and electron-withdrawing group on phenyl ring, the corresponding product was formed with 85-93% yields under reaction conditions (Table 3, entries 2-6 and 10). Aromatic aldehydes bearing electron-donating gave high yields of the corresponding products (Table 3, entries 7-9), but within longer reaction time than benzaldehydes containing electron-withdrawing group.

An interesting feature of this method is that the catalyst can be regenerated at the end of the reaction and can be used several times without losing its activity. To recover the catalyst after completion of the reaction, ethyl acetate was added to the reaction mixture. The resulting heterogeneous mixture was filtered and the solid residue was washed with ethyl acetate and dried in an oven at 80 °C for 2 h. The recovered catalyst was used for model reaction. This process was repeated for three cycles and the yield of product **4a** did not significantly change (Table 2).

According to the literature survey [15], the mechanism suggested for the formation of dihydropyrano [3,2-*b*]chromenedione derivatives have been shown in Scheme 2. The more important point is that nano-PS-OSO<sub>3</sub>H facilitates the Knoevenagel-type coupling by coordinating with the oxygen of carbonyl group of benzaldehyde. Complex **I** is produced by the reaction of benzaldehyde **1** with nano-PS-OSO<sub>3</sub>H. In next step, by the Knoevenagel reaction of complex **I** and dimedone, intermediate **II** is generated. Subsequent Diels Alder-type addition of kojic acid **3** to intermediate **II** followed by dehydration afford the product **4a-4j** (Scheme 2).

To show the merits and drawbacks of this catalyst, our results were compared with other catalysts results reported in the literature (Table 4). In Table 4, dihydropyrano[3,2-*b*]chromenedione **4d** has been considered. Although the reaction temperatures and yields of the product in the present protocol are comparable with other methods, our method requires a smaller amount of catalyst than most methods (Table 4, entries 2-3, 6 and 9). So, the use of a cheap, green and natural-based renewable heterogeneous nanocatalyst, which is prepared easily, is great advantage of this method than other approaches.

#### 4. Conclusions

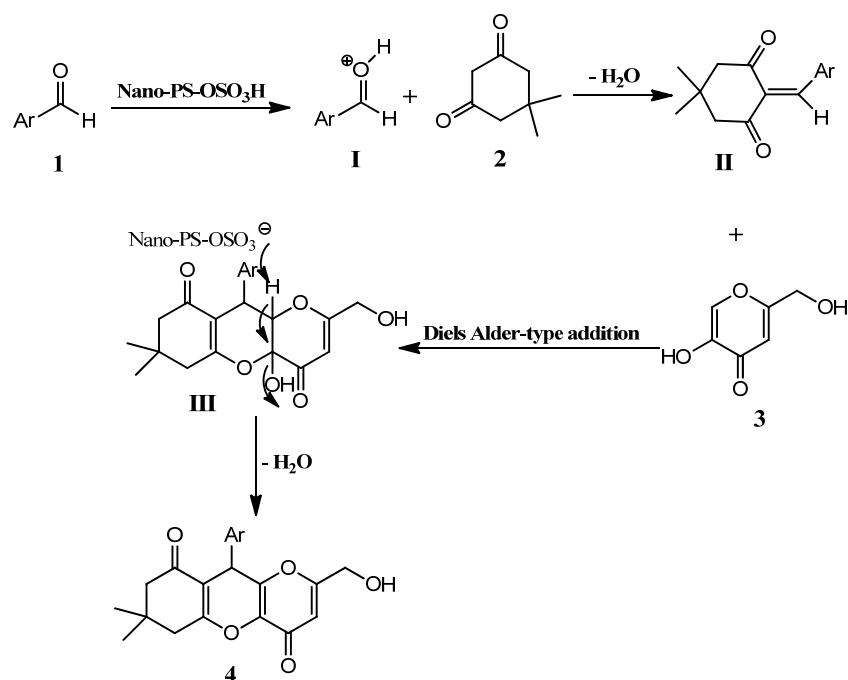
In conclusion, for the first time nano-PS-OSO<sub>3</sub>H was introduced as a heterogeneous eco-friendly highly powerful solid acid catalyst for the synthesis of dihydropyrano[3,2-*b*]chromenediones under solvent-free conditions. All the reactions work easily for a variety of aldehydes with both electron-donating and electron-withdrawing groups to give corresponding products in high yields.

**Table 3.** Synthesis of dihydropyrano[3,2-*b*]chromenedione derivatives **4**.<sup>a</sup>

Entry	Ar	Product	Time (min)	Yield (%) <sup>b</sup>	m.p. (°C)	Ref.
1	C <sub>6</sub> H <sub>5</sub>	<b>4a</b>	120	90	185-187	[15]
2	2-Cl- C <sub>6</sub> H <sub>4</sub>	<b>4b</b>	120	87	215-217	[16]
3	3-Cl- C <sub>6</sub> H <sub>4</sub>	<b>4c</b>	127.5	93	197-199	[15]
4	4-Cl- C <sub>6</sub> H <sub>4</sub>	<b>4d</b>	120	93	204-206	[16]
5	3-NO <sub>2</sub> - C <sub>6</sub> H <sub>4</sub>	<b>4e</b>	108	93	210-212	[16]
6	4-NO <sub>2</sub> - C <sub>6</sub> H <sub>4</sub>	<b>4f</b>	96	89	227-229	[16]
7	4-CH <sub>3</sub> -C <sub>6</sub> H <sub>4</sub>	<b>4g</b>	144	87	213-215	[16]
8	4-OCH <sub>3</sub> - C <sub>6</sub> H <sub>4</sub>	<b>4h</b>	168	85	179-181	[15]
9	2,5-(OCH <sub>3</sub> ) <sub>2</sub> - C <sub>6</sub> H <sub>3</sub>	<b>4i</b>	144	83	190-192	[16]
10	2,4-(Cl) <sub>2</sub> - C <sub>6</sub> H <sub>3</sub>	<b>4j</b>	120	89	164-166	[16]

<sup>a</sup>All products were identified by their melting points, <sup>1</sup>H NMR and <sup>13</sup>C NMR spectra.

<sup>b</sup>Isolated yield.



**Scheme 2.** Plausible mechanism for the formation of dihydropyrano[3,2-*b*]chromenedione derivatives.

**Table 4.** Comparison of nano-PS-OSO<sub>3</sub>H with other catalysts for the synthesis of dihydropyrano[3,2-*b*] chromenedione **4d**.

Entry	Catalyst	Reaction condition	Time (min)	Yield (%) <sup>b</sup>	Ref.
1	nano-PS-OSO <sub>3</sub> H	Solvent free/120 °C/7.55 mol %	120	93	-
2	CAN	Solvent free/120 °C/10 mol %	30	95	[18]
3	InCl <sub>3</sub>	Solvent free/120 °C/10 mol %	80	95	[15]
4	CeCl <sub>3</sub> ·7H <sub>2</sub> O	Solvent free /110 °C/5 mol %	40	96	[26]
5	FeCl <sub>3</sub> -SiO <sub>2</sub>	Solvent free/100 °C/5 mol %	60	91	[17]
6	Alum	Solvent free/100 °C/10 mol %	50	94	[16]
7	Bi(OTf) <sub>3</sub>	Solvent free /120 °C/5 mol %	20	91	[27]
8	Nano-Bi <sub>2</sub> O <sub>3</sub> ·ZnO	Solvent free/100 °C/ 2.8 mol%	60	82	[28]
9	[SiPrPy]AlCl <sub>4</sub> @MNPs	Solvent free/110 °C/10 mol %	20	95	[29]

The excellent catalytic performance, easy preparation and separation of the catalyst make it to be a good heterogeneous solid acid nanocatalyst for organic synthesis and transformations. In addition, good yields, low-cost, short reaction times, nontoxicity, and solvent-free conditions are supporting methods toward the green chemistry.

#### Acknowledgments

The Research Council of the Islamic Azad University of Yazd Branch is gratefully acknowledged for the financial support for this work.

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