Original Research Paper

A novel silicotungstic acid-containing ionic liquid immobilized on graphene oxide nanosheets: Preparation, characterization, and first catalytic application in the synthesis of 1,8-dioxo-octahydroxanthenes

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Abstract

A novel functionalized graphene oxide (GO) nanosheets containing silicotungstic anion $H_3SiW_{12}O_{40}^-$ (H_3SiW), was successfully prepared by grafting of (3-chloropropyl)triethoxysilane (3-CPTS) on GO nanosheets followed by reaction with an excess amount of imidazole and finally interaction with silicotungstic acid ($H_4SiW_{12}O_{40}$, denoted as H_4SiW). The prepared material, denoted as GO-Imid-H_3SiW, was characterized using scanning electron microscopy (SEM), transmission electron microscopy (TEM), and energy-dispersive X-ray (EDX) analysis. The activity of this new material as a novel catalyst was also evaluated in the synthesis of 1,8-dioxo-octahydroxanthenes by reaction of dimedone with various aromatic aldehydes under solvent-free conditions. The results demonstrated a significant catalytic performance of the catalyst for this transformation under solvent-free conditions, giving high yields of the desired products over relatively short reaction times. In addition, the catalyst could be easily recovered from the reaction mixture by simple filtration and can be reused many times with no significant loss of its catalytic activity.

1. Introduction

Graphene is a novel material that has emerged as a rapidly rising star in the field of material science.[1] It possesses unique physical properties, such as high thermal and electronic conductivity, high thermal stability, and high structural strength,[2-4] and therefore has diverse applications in various fields.[5-7] Graphene is easily produced from graphene oxide (GO) by reduction using reducing agents such as $NaBH_4$[8] or hydrazine.[9] In recent years, GO which can be prepared from graphite powder using Hummers method,[10] has attracted great interest as a promising ideal support for a number of metals, nanoparticles and organic compounds.[11-13] Heteropoly acids (HPAs) are an important class of catalysts having both redox and acid properties.[14] Among them; silicotungstic acid, $H_4SiW_{12}O_{40}$ (H_4SiW), with Keggin structure has received the most attention due to its unique structure and strong acidity.[15] Several advantages of supported HPAs compared to homogeneous examples include easier recovery and recycling after carrying out reactions, and simpler product separation.[16] The ionic immobilization of H_4SiW on GO can be employed to derive a novel heterogeneous catalyst system that possesses both a high separation efficiency and a relatively high surface area to maximize catalyst loading and activity. The synthesis of xanthenes, in particular xanthenediones, has received significant attention because of their wide range of biological and pharmaceutical properties.[17-19] Xanthenediones are generally synthesized by condensation of aromatic aldehydes with 5,5-dimethyl-1,3-cyclohexanedione (dimedone) using various promoting agents.[20-26] Nevertheless, development of new efficient recyclable catalysts for the synthesis of these compounds was of certain demand. In the last decade, acidic ionic liquids (ILs), especially those based on imidazolium cation, have attracted much interest as green substitute catalysts for conventional acids.[27-29] However, the danger of catalyst leaching and the difficulties of extracting products from ILs when polar products are produced are still big challenges to overcome. In this view and in line with our interest in catalysis,[30-37] in this paper, a novel functionalized imidazolium-based IL containing a silicotungstic counter-anion $H_3SiW_{12}O_{40}^-$ (H_3SiW) immobilized on GO was prepared and characterized (Scheme 1). The

catalytic activity of this new material which was denoted as GO-Imid-H$_3$SiW was investigated in the synthesis of 1,8-dioxo-octahydroxanthenes by reaction of dimedone with aromatic aldehydes under solvent-free conditions (Scheme 2).

2. Results and Discussion

### 2.1. Preparation and characterization of the catalyst GO-Imid-H$_3$SiW

At first, GO nanosheets were prepared from natural graphite powder by modified Hummers method.[10] GO nanosheets were allowed to react with 3-CPTS and then with an excess amount of imidazole to give GO-Imid. Finally, the interaction of obtained GO-Imid nanosheets with H$_4$SiW gave the final material which is denoted as GO-Imid-H$_3$SiW (Scheme 1) and characterized using different techniques including SEM, TEM, and EDX.

The surface morphology of the GO-Imid-H$_3$SiW was characterized using SEM image. As seen in Figure 1, the SEM image exhibits a nanosheet-like structure in disordered phase with crumpled and wrinkled edges. These folded edges owing to sp^3-carbon in GO-Imid-H$_3$SiW, carry Imid-H$_3$SiW on both sides of the GO nanosheets. These sites may serve as reactive catalytic sites to provide the desired chemical transformation. On the other hand, the granular-like particles on the surfaces of GO nanosheets, confirm efficient grafting of new imidazole functionalized GO containing H$_3$SiW anion on the GO nanosheets..
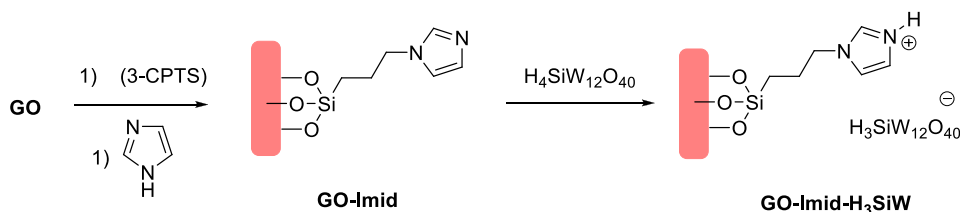
Scheme 1. Preparation of GO-Imid-H$_3$SiW nanosheets
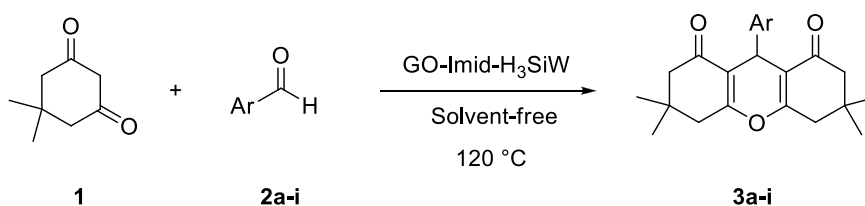
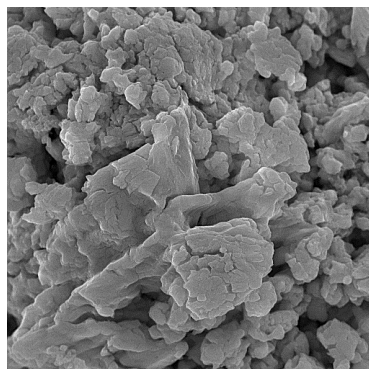**Scheme 2.** GO-Imid-H$_3$SiW nanosheets catalyzed synthesis of 1,8-dioxo-octahydroxanthenes

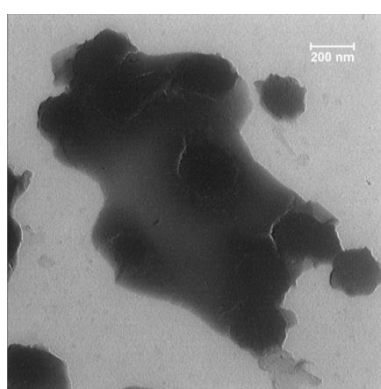**Figure 1.** The SEM images of GO-Imid-H_3SiW nanosheets

Figure 2. The TEM images of GO-Imid-H_3SiW nanosheets
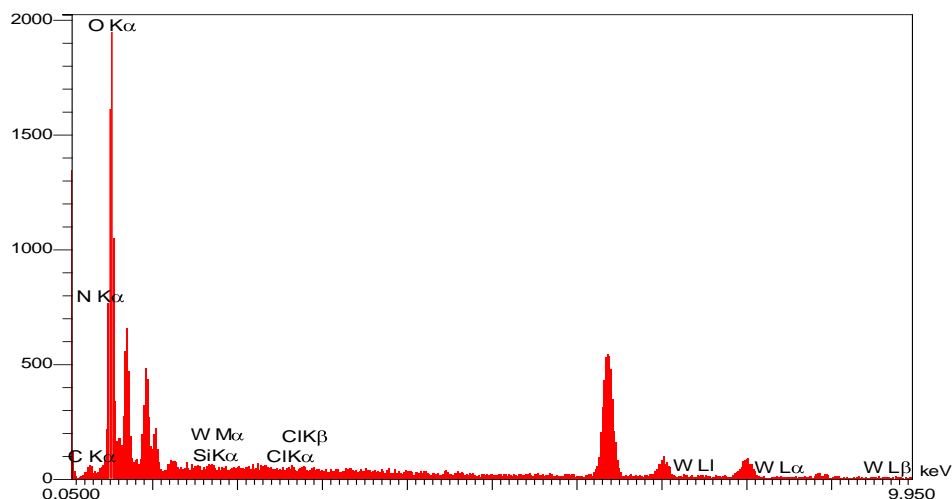
Figure 3. EDX analysis of GO-Imid-H_3SiW nanosheets

In the TEM image of the GO-Imid-H_3SiW shown in Figure 2, some recognizable nanoparticles supported on the surface of the GO nanosheets can be seen. In order to get more evidences about the chemical structure of GO-Imid-H_3SiW catalyst, EDX analysis was provided. As depicted in Figure 3, the EDX spectrum clearly shows the presence of oxygen, carbon, silicon, nitrogen, and tungsten elements in the catalyst, confirming the successful functionalization of GO.

2.2 Evaluation of the catalytic activity of GO-Imid-H$_3$SiW

The catalytic activity of the GO-Imid-H$_3$SiW was tested in the synthesis of 1,8-dioxo-octahydroxanthenes. For optimization of the reaction conditions, various parameters like catalyst amount, effect of solvent and influence of temperature were studied for the formation of compound **3b**. These results are summarized in Table 1. When the reaction was carried out without catalyst (Table 1, entry 1) under solvent-free conditions at high temperature, a low yield of the product was formed even after 90 min. On the contrary, we were pleased to see that the reaction was efficiently catalyzed by GO-Imid-H$_3$SiW.

Solvent-free condition proved to be a much better media than the others tested including H$_2$O, MeOH, EtOH, and CH$_3$CN. The best result was achieved at 120 °C in the presence of 0.03 g of GO-Imid-H$_3$SiW (entry 10). Thereafter, applicability of the method was evaluated for the synthesis of other 1,8-dioxo-octahydroxanthenes using a variety of aryl aldehydes (Table 2). As can be seen, in all cases the products were isolated in high yields within short reaction time showing high catalytic activity of GO-Imid-H$_3$SiW. Under the same conditions however, no reaction occurred when aliphatic aldehydes were used.

Table 1. Synthesis of **3b** in the presence of the GO-Imid-H$_3$SiW catalyst under different reaction conditions[a]

Entry	Catalyst (g)	Solvent	T (°C)	Time (min)	Yield (%)
1	-----	-----	120	90	22
2	0.01	-----	80	45	57
3	0.01	-----	100	40	66
4	0.01	-----	120	25	69
5	0.02	-----	80	38	66
6	0.02	-----	100	30	75
7	0.02	-----	120	18	81
8	0.03	-----	80	23	75
9	0.03	-----	100	12	81
10	0.03	-----	120	8	91
11	0.03	-----	140	8	91
12	0.04	-----	120	8	90
13	0.03	H$_2$O	Reflux	180	32
14	0.03	MeOH	Reflux	90	57
15	0.03	EtOH	Reflux	90	67
16	0.03	CH$_3$CN	Reflux	90	53

[a]Reaction conditions: dimedone **1** (2 mmol), 4-chlorobenzaldehyde **3b** (1 mmol).

Table 2. GO-Imid-H$_3$SiW catalyzed synthesis of 1,8-dioxo-octahydroxanthenes **3a-i**[a]

Entry	R	Product	Time (min)	Isolated Yield (%)	mp (°C)	
					Find	Reported
1	C$_6$H$_5$	**3a**	8	90	201-203	203-205 [20]
2	4-ClC$_6$H$_4$	**3b**	8	91	228-230	230-233 [38]
3	2-ClC$_6$H$_4$	**3c**	10	89	231-233	230-232 [38]
4	4-HOC$_6$H$_4$	**3d**	13	86	245-247	247-249 [25]
5	4-BrC$_6$H$_4$	**3e**	8	90	239-241	238-239 [39]
6	3-O$_2$NC$_6$H$_4$	**3f**	7	92	173-175	171-173 [38]
7	4-O$_2$NC$_6$H$_4$	**3g**	5	93	225-226	227-228 [25]
8	4-MeC$_6$H$_4$	**3h**	15	88	212-213	210-215 [26]
9	4-MeOC$_6$H$_4$	**3i**	15	87	237-239	240-243 [26]

To further evaluate the overall utility of the current methodology, we compared our results with those of the other methods reported for the synthesis of 1,8-dioxo-octahydroxanthenes. This comparison is shown in Table 3. As can be seen, the catalyst gave high yields of the products in shorter reaction time than the other methods.

In view of the green chemistry, the catalyst was further explored for the reusability using the model reaction. After completion of the reaction, the mixture was cooled to room temperature and hot ethanol was added. The catalyst was easily separated by a simple filtration, washed with ethanol, dried at 60 °C under vacuum for 1 h, and used for the next cycle. The catalyst could be used at least ten times with only a slight reduction in activity (91, 91, 90, 88, 88, 87, 87, 87, 86, 86 % yields for first to tenth

use, respectively) which clearly demonstrates the practical reusability of this catalyst.

To show the catalyst's role, a plausible mechanism for the present reaction may proceed as depicted in Scheme 3. It is proposed that the reaction occurs *via* initial formation of the intermediate [**II**], prepared by condensation of dimedone **1** with aryl aldehydes **2a-i** via the intermediate [I]. Subsequent Michael addition of the intermediate [**II**] with enolic form of dimedone **1** followed by addition of the hydroxyl moiety in intermediate [**III**] to the carbonyl group provides cyclic hemiketal intermediate [**IV**], which on dehydration afforded final products **3a-i**. As shown in Scheme 3, we propose that the GO-Imid-$H_3SiW \equiv HA$ activate the reactants and the intermediates in this reaction. Under these conditions, attempts to isolate the intermediates failed even after careful monitoring of the reactions.

Table 3. Comparison of the efficiencies of different catalysts for the synthesis of 1,8-dioxo-octahydroxanthenes

Catalyst	Conditions			Time (min)	Yield (%)	Ref.
	Solvent	T (°C)	Other			
FeNP@SBA-15	-----	80	-----	20-60	90-99	[40]
L-Proline	ClCH$_2$CH$_2$Cl	60	-----	360	59-90	[22]
CAN/HY-zeolite	-----	80	-----	45-160	72-93	[24]
Trimethylsilyl chloride	CH$_3$CN	reflux	-----	480-600	72-84	[41]
Fe$_3$O$_4$@SiO$_2$@(CH$_2$)$_3$-Imidazole-SO$_3$H]Cl	-----	80	-----	10-40	89-98	[42]
Sulfated zirconia	EtOH	70	-----	480	84-95	[43]
SmCl$_3$	-----	120	-----	480-1440	20-98	[38]
Silica/Preyssler NPs	H$_2$O	reflux	-----	180	82-96	[20]
β-CD-BSA	H$_2$O	reflux	-----	15-60	88-97	[44]
GO-Imid-H$_3$SiW	-----	120	-----	5-15	86-93	This work
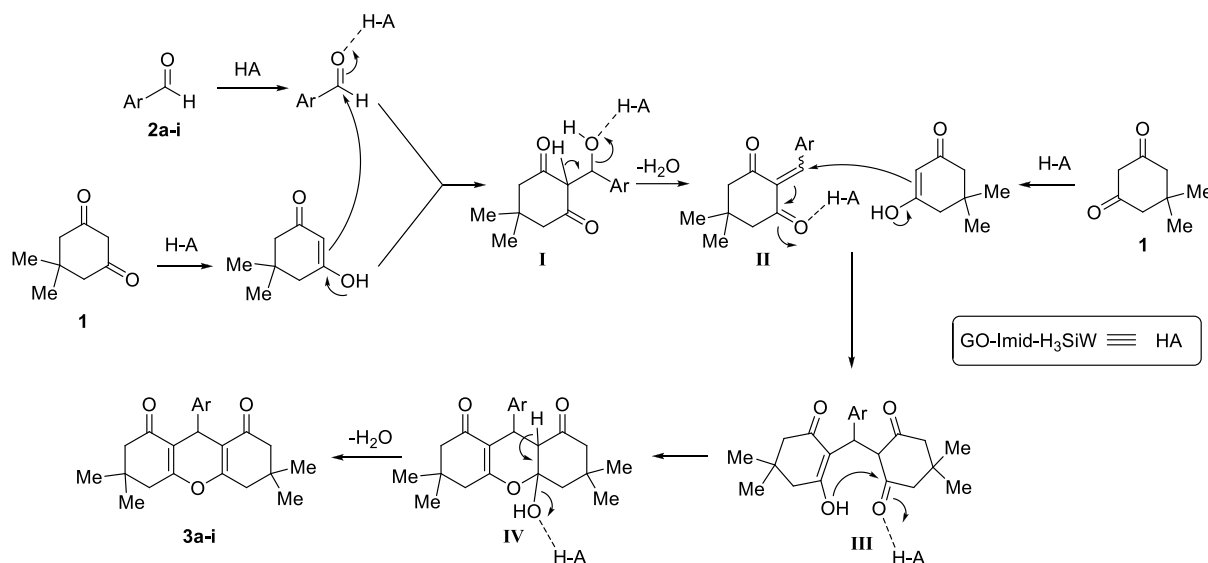
Scheme 3. Plausible mechanism for the formation of 1,8-dioxo-octahydroxanthenes in the presence of GO-Imid-$H_3SiW \equiv HA$ as catalyst

3.   Experimental

All chemicals were purchased from Merck and Aldrich and used without further purification. Melting points were recorded with a Stuart SMP3 melting point apparatus. Ultrasonication was performed using a Soltec sonicator at a frequency of 40 kHz and a nominal power of 260 W. SEM analyses was done using a TESCAN BRNO-MIRA3 LMU. TEM analysis was performed using a Leo 912 AB microscope with an accelerating voltage of 120 kV. EDX analysis and elemental mapping were performed using a SAMX model instrument.

3.1.   Preparation of GO nanosheets

GO nanosheets were prepared from natural graphite using Hummers method[10] with some modification. A mixture of graphite powder (5.0 g), sodium nitrate ($NaNO_3$, 2.5 g), and concentrated sulfuric acid (115 mL, 98% H_2SO_4) was stirred in an ice bath at 0-5 °C for 15 min and then potassium permanganate ($KMnO_4$, 15.0 g) was slowly added. The stirring was continued for 2 h while the temperature was kept in the range of 0-10 °C. The mixture was then transferred to a water bath and stirred at 35 °C for 30 min, forming a brownish grey thick paste. Afterwards, deionized water (230 mL) was slowly added to the paste and the suspension, now brown in color, was stirred at 95-98 °C for 15 min. The suspension was further diluted with warm deionized water (700 mL, 40 °C), followed with a drop by drop addition of 30% hydrogen peroxide (H_2O_2, 50 mL). The mixture was centrifuged and the isolated yellow-brown cake was washed with diluted HCl (5 wt%) and deionized water several times until the pH became neutral. The solid graphite oxide was separated by centrifugation and dried at 60 °C under vacuum for 12 h. The obtained graphite oxide (0.4 g) was dispersed in distilled water (400 mL) and sonicated in an ultrasonic bath cleaner (100 W) for 1 h to exfoliate graphitic oxide. The complete exfoliation of graphite oxide is confirmed with the formation of light brown coloured homogeneous dispersion GO. Afterwards, the GO solution was centrifuged for 20 min and then the supernatant was removed (to remove any unexfoliated graphitic oxide). The GO nanosheets were obtained after drying the sediment in a vacuum oven at 80 °C for 24 h.

3.2.   Preparation of GO-Imid

The synthesized GO (0.5 g) was ultrasonically dispersed in anhydrous toluene (10 ml) at room temperature for 20 min and then 3-CPTS (0.5 ml) was added. The mixture was heated under reflux for 12 h. The resultant solid was filtered and washed with toluene, and dried at 70 °C under vacuum for 8 h. The later material was ultrasonically dispersed in dry toluene (7 ml) for 15 min at 70 °C and then imidazole (10 mmol) was added and the mixture was heated under reflux for 12 h. After cooling to room temperature, the solid was collected and repeatedly washed with toluene and diethyl ether and dried under vacuum at 80 °C for 2 h to form GO-Imid nanosheets.

3.3.   Preparation of GO-Imid-H_3SiW

GO-Imid (0.5 g) was ultrasonically dispersed in absolute ethanol (15 ml) at 70 °C for 15 min. H_4SiW (2 mmol) was added to the suspension and sonication continued for another 1 h at same temperature. The mixture was then refluxed for 12 h. After cooling to room temperature, the solid was collected by filtration and repeatedly washed with absolute ethanol and dried under vacuum at 70 °C for 8 h to form GO-Imid-H_3SiW.

3.4. General procedure for the synthesis of 1,8-dioxo-octahydroxanthenes 3a-i catalyzed by GO-Imid-H$_3$SiW

To a mixture of dimedone **1** (2 mmol), and an aromatic aldehyde **2a-i** (1 mmol), GO-Imid-H$_3$SiW (0.03 g) was added. The mixture was heated in the oil bath at 120 °C for 5-15 min and the reaction was monitored by TLC. Upon completion, the reaction mixture was cooled to room temperature and hot ethanol was added. The catalyst was insoluble in hot ethanol and it could therefore be recycled by a simple filtration. The product was then collected from the filtrate after cooling to room temperature and recrystallized from ethanol to give compounds **3a-i** in high yields.

9-(4-Chlorophenyl)-3,3,6,6-tetramethyl-1,8-dioxo-octahydroxanthene (3b). ^{1}H NMR (δ, ppm): 1.13 (s, 6H, 2CH$_3$), 1.24 (s, 6H, 2CH$_3$), 2.30-2.55 (m, 8H, 4CH$_2$), 5.50 (s, 1H, CH), 7.04 (d, J = 8.1 Hz, 2H, arom-H), 7.25 (d, J = 8.1 Hz, 2H, arom-H); FT-IR (v, cm^{-1}): 2952, 1661, 1625, 1489, 1469, 1413, 1361, 1198, 1166, 1140, 1089, 1003, 852.

9-(2-Chlorophenyl)-3,3,6,6-tetramethyl-1,8-dioxo-octahydroxanthene (3c). ^{1}H NMR (δ, ppm): 1.04 (s, 6H, 2CH$_3$), 1.12 (s, 6H, 2CH$_3$), 2.13-2.29 (m, 4H, 2CH$_2$), 2.47 (s, 4H, 2CH$_2$), 5.02 (s, 1H, CH), 7.08 (t, J = 6.9 Hz, 1H, arom-H), 7.19 (t, J = 7.2 Hz, 1H, arom-H), 7.24 (d, J = 7.8 Hz, 1H, arom-H), 7.45 (d, J = 7.2 Hz, 1H, arom-H); FT-IR (v, cm^{-1}): 3070, 2960, 1664, 1625, 1468, 1356, 1203, 1156, 1036, 1006, 840, 793.

9-(4-Nitrophenyl)-3,3,6,6-tetramethyl-1,8-dioxo-octahydroxanthene (3g). ^{1}H NMR (δ, ppm): 0.91 (s, 6H, 2CH$_3$), 1.04 (s, 6H, 2CH$_3$), 2.09 (d, J = 16.5 Hz, 2H, CH$_2$), 2.18 (d, J = 16.2 Hz, 2H, CH$_2$), 2.42 (s, 4H, 2CH$_2$), 4.74 (s, 1H, CH), 7.39 (d, J = 8.7 Hz, 2H, arom-H), 8.01 (d, J = 8.7 Hz, 2H, arom-H); FT-IR (v, cm^{-1}): 2959, 1662, 1515, 1470, 1361, 1344, 1201, 1166, 1139, 1002, 868.

4. Conclusions

In summary, GO-Imid-H$_3$SiW, as a new functionalized GO containing a silicotungstic counter-anion, was successfully prepared and characterized. This new functionalized GO performed well as a catalyst in the synthesis of 1,8-dioxo-octahydroxanthenes by reaction of dimedone with several aromatic aldehydes. The reactions occur under solvent-free conditions and furnish the expected products in high yields within short reaction times. Also, the catalyst can be easily recovered by simple filtration and reused for subsequent reactions without noticeable deterioration in catalytic activity.

Acknowledgement

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