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Pt NPs@GO as Highly Efficient and Reusable Catalyst for One-Pot Synthesis of Acridinedione Derivatives

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Platinum nanoparticles decorated with graphene oxide (Pt NPs@GO) have been used for the first time as highly stable, exceptional reusable, isolable, bottleable, long-lived and highly efficient catalyst for the synthesis of acridinedione derivatives from dimedone, aromatic aldehydes and various amines with great catalytic performance. All acridinedione compounds were synthesized in DMF a single process through two successive reactions (Aldol condensation and Michael addition) in the presence of highly efficient Pt NPs@GO. The synthesized catalyst was characterized by transmission electron microscopy (TEM), the high resolution electron micrograph (HRTEM), X-ray diffraction (XRD), Atomic force microscopy (AFM) and X-ray photoelectron spectroscopy (XPS). All products obtained by Pt NPs@GO were characterized by FT-IR, ^{1}H-NMR, ^{13}C-NMR and HRMS techniques.

Introduction

Acridinedione molecules were discovered in 19th century and new derivatives of acridinedione were still continued to be explored today. Acridinedione derivatives of 1,4-dihydropyridine are compounds having interesting biological features which are similar to the structure of Nicotinamide Adenine Dinucleotide (NADH), an important co-enzyme found in cells [1]. Moreover, acridinedione derivatives are medicinally important compounds [2] and are used in laser dyes due to their high fluorescence efficiency [3, 4]. Furthermore, since acridinedione derivatives have a wide range of biological activities, for example antimicrobial [5, 6], antibacterial [7], fungicides [8], antitumor [9], anticancer [10-12] and also used for glaucoma treatment as carbonic anhydrase inhibitor [13-15] there are lots of reported methods for synthesis of acridinedione including multi component condensation (MCR) of aromatic aldehyde, cyclic diketones, and various aromatic amines in the presence of different catalysts, for example Amberlyst-15 [16, 17], CTAB [18], DBSA [19, 13], FSG-Hf(Npf$_2$)$_4$ [20], H_2SO_4 [15], Melamine-formaldehyde resin supported H^+ [21], Proline [22], SiO_2-Pr-SO_3H [23]. However, these homogeneous catalysts pose difficulties in reusability, recovery, separation and the sythesis procedure. For this reason, the heterogeneous alternatives have already captivated much attention. Due to the heterogeneous catalyst may be recovered and reusable, recently several heterogeneous catalytic systems were reported using, for example, CdO [24], Fe_2O_3@SiO_2-PW [25], Fe_3O_4 [26], Fe_3O_4@SiO_2 [27], $NiFe_2O_4$@SiO_2-FHS [28], $ZnFe_2O_4$ [29] as catalysts. However the problem in most of those catalytic systems is unsatisfactory yield, long reaction times, laborious work-up procedures, the requirement of special apparatus and harsh reaction conditions. To avoid these limitations and to improve the reaction conditions available for the synthesis of acridinedione, the discovery of new methodologies using new heterogeneous and reusable catalysts is still in demand. Herein we wish to report an efficient and novel method for the synthesis of acridinedione derivatives via one-pot MCRs of aromatic aldehyde, cyclic diketones, and various aromatic amines in high yields and short reaction times by using Pt NPs@GO as a highly efficient, robust, reusable and easily recoverable catalyst for synthesis of acridinedione derivatives.

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The current Pt NPs@GO has been used for the first time for these types of synthesis reactions.

Results and discussion

Pt NPs@GO has been prepared by sonochemical reduction method as given experimental section and their preliminary characterization was performed by TEM, HRTEM, XRD, AFM and XPS methods.

The high resolution electron micrograph (HRTEM) and particle size histograms of the prepared catalyst are demonstrated in Fig. 1. This also shows the information regarding the particle size and morphology of the Pt NPs@GO. A uniform distribution of the catalyst particles with a relatively narrow range on graphene oxide support was detected and most of the particles are found to be with and approximately spherical shape and average diameter of around 3.25 nm and no agglomerations were observed in the prepared catalyst. Moreover, the chosen area of HRTEM image of Pt NPs@GO displays the highly crystalline feature with a spacing of 0.229 nm which is very close to nominal Pt (111) spacing of 0.228 nm [30-35].

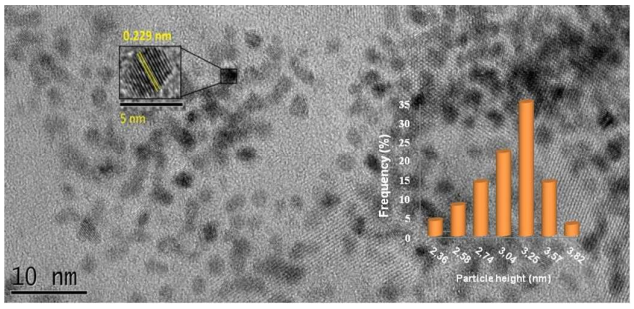


Fig. 1. High resolution transition electron micrograph and particle size histogram of a Pt NPs@GO

X-ray diffraction (XRD) measurements indicate that Pt NPs@GO exhibit distinct diffraction patterns, as illustrated in Fig. S1. The diffraction peaks at 2θ = 39.74, 46.32, 67.63, 81.58 and 85.79 are due to Pt (111), (200), (220), (311), and (320), respectively, planes of the face-centered cubic (fcc) crystal lattice of platinum (*JCPDS-ICDD, Card No. 04-802*). The peak at 10.8° is raised from the graphite (002) plane of GO. The Pt (220) diffraction peaks of the prepared catalyst are used to calculate the lattice parameters (αPt) and average crystallite sizes of the metal particles. The lattice constant was found to be 3.921 Å, which is in good agreement with 3.923 Å for pure Pt, by using the following equation [33].

$$\text{Sin } \theta = \frac{\lambda\sqrt{h^2+k^2+l^2}}{2a}$$ (for a cubic structure)

The average crystallite particle size of Pt NPs@GO were found to be about 3.49 ± 0.53, using the following equation [36-38];

$$d\ (\text{Å}) = \frac{k\lambda}{\beta cos\theta}$$

Where k= a coefficient (0.9); λ= the wavelength of X-ray used (1.54056 Å); β= the full width half-maximum of respective diffraction peak (rad); θ= the angle at the position of peak maximum (rad). Moreover, the Chemical Surface Areas (CSA) of the Pt NPs@GO were found to be 88.17 m^2/g by the use of the following equation, assuming homogenously distributed and spherical particles [39],

$$\text{CSA} = \frac{6 \times 10^3}{\rho \times d}$$

where d is the mean Pt crystalline size in Å (from the XRD results) and ρ is the density of Pt metal (21.4 g/cm^2) [40, 41].

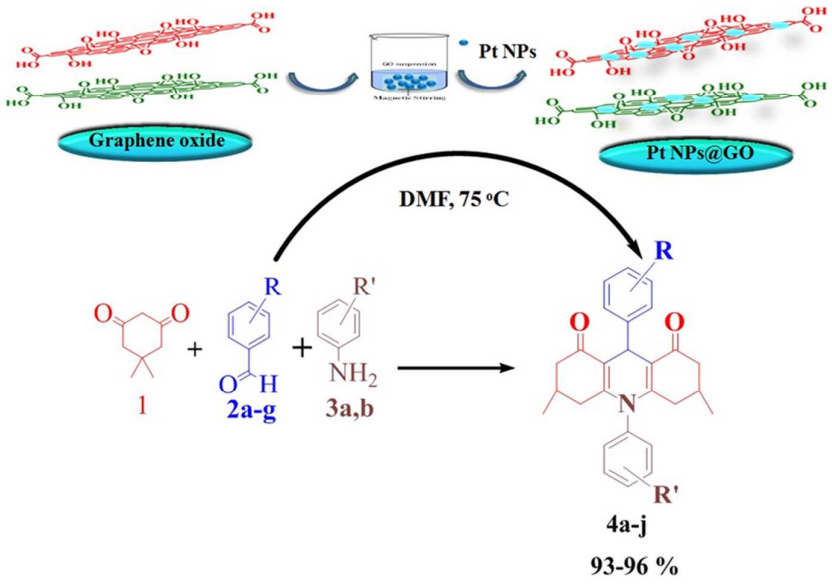
Atomic force microscopy (AFM) was also used to observe the height diameter distributions shown in Fig. S2a, b. The AFM height diameter of the Pt NPs@GO is found to be 3.27 nm which is in good agreement with the dimensions get by XRD and TEM.

X-ray photoelectron spectroscopy (XPS) was used for the determination of the surface compositions and chemical oxidation states of Pt in the prepared nanocatalyst. The Pt 4f region of the spectrum was analysed, and the fitting of the XPS peak was performed using the Gaussian-Lorentzian method. In the XPS spectrum, accurate binding energies (±0.3 eV) were determined by referencing to the C 1s peak at 284.6 eV. The Pt 4f photoelectron spectrum, consisting of two pairs of doublets, is illustrated in Fig. S3 for the prepared catalyst. The most intense doublet at about 71.2 and 74.0 eV is a signature of metallic platinum (23-30) and the other doublet at about 74.0 and 77.0 eV is most likely caused by a very small fraction of oxidized Pt^{4+} species possibly due to PtOx species formed during catalyst exposure to the atmosphere. The ratios of Pt (0) to Pt (IV) for the prepared catalyst were calculated from the relative peak area of the Pt 4f spectrum. The relative intensities of the Pt (0) and Pt (IV) species were estimated by calculating the integral of each peak after smoothing and

subtracting the Shirley-shaped background and Pt (0)/Pt (IV) ratio is found to be 6.81 for the Pt NPs@GO.

It is worth mentioning that Pt NPs@GO is often used as a catalyst in batteries, the hydrogen storage, sensors, super-capacitors [42-46] but its use as a catalyst for synthesizing acridinedione derivatives from different aromatic aldehydes and various amines has not been reported before.

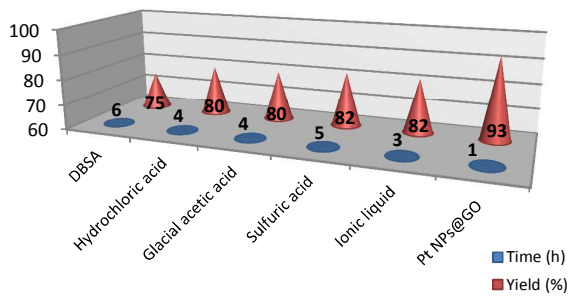
For the synthesis of acridinedione derivatives new, facile and effective method has been designed in the presence of Pt NPs@GO. The synthesis of acridinedione compounds were realized in DMF from dimedone **1**, different aromatic aldehydes **2a-g** and various amines **3a, b** in a single process through three successive reactions (Aldol condensation, Michael addition and cyclization) by using Pt NPs@GO as catalyst. Especially these acridinedione derivatives have been synthesized by using **1**, **2f**, **3a** compounds in the presence of **Pt NPs@GO** as a model reaction (Scheme 1).



Scheme 1. Synthesis of acridinedione derivatives in the presence of Pt NPs@GO.

In this line of work, we compared the results using Pt NPs@GO in the synthesis of acridinedione derivatives with those obtained using other catalysts such as DBSA, hydrochloric acid, glacial acetic acid [47], sulfuric acid, ionic liquid [48]. As shown in Table 1, Pt NPs@GO is the most efficient catalyst which gives the highest yields of products in the shortest time.

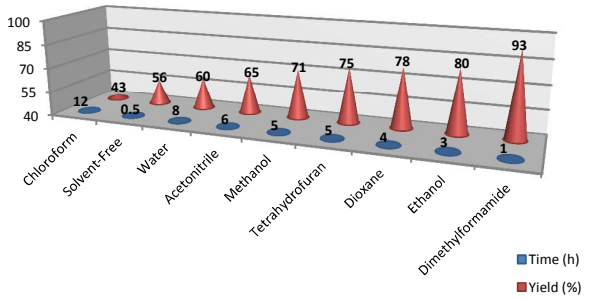
Table 1. Comparison of Pt NPs@GO with other catalysts



We also optimized our model reactions by different solvents, amount of catalyst and reaction conditions on the basis of different temperatures.

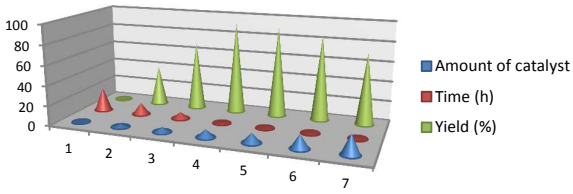
Firstly, the effect of different solvents such as chloroform, solvent-free, water, acetonitrile, methanol, tetrahydrofuran, dioxane, ethanol and dimethylformamide has been evaluated on the reaction rate under the same reaction conditions (Table 2). Since the reaction proceeded with the highest yield and shorter reaction time in the presence of DMF compared to other used solvents, we have chosen DMF as an optimum solvent for the model reaction.

Table 2. A comparison of the efficiency of various solvents employed in the synthesis of the acridinedione derivatives



We carried out seven sets of model reaction by varying the amount of catalyst to determine the exact catalyst concentration for the best possible yield. The first set where no catalyst was used led to no formation of product but the amount of catalyst increased gradually from 3 to 8 mg a steady increase was observed in the product yield (Table 3). Furthermore increase in catalyst concentration give rise to decrease in the product yield possibly due to the protonation of amine groups in the aromatic ring by using carboxylic acid groups of the graphene oxide.

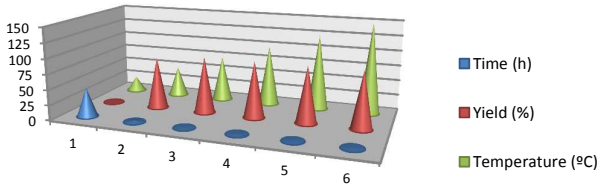
Table 3. The Optimization of Pt NPs@GO concentration for preparation of acridinedione derivatives in DMF



	1	2	3	4	5	6	7
Amount of catalyst	0	3	5	8	10	14	20
Time (h)	24	12	7	1	0.5	0.5	0.5
Yield (%)	0	40	69	93	91	86	72

After standardizing the solvent and catalyst concentration, we turned our attention toward examining the temperature effect on the model reaction. It was observed that at room temperatures no desired product was formed even after 48 h of stirring. The yield of desired product gradually increased with temperature from room temperature to 75 ^{o}C as shown in Table 4.

Table 4. The Optimization of temperature for preparation of acridinedione derivatives using Pt NPs@GO as catalyst in DMF



	1	2	3	4	5	6
Time (h)	48	3	1	1	1	1
Yield (%)	0	85	93	93	93	93
Temperature (ºC)	25	50	75	100	125	150

As a summary, the highest efficiency and fastest reaction time on the model reaction was observed at 75 ^{o}C by using 8 mg of **Pt NPs@GO** in DMF. The results of all reactions performed under these conditions are shown in Table 5.

Table 5. Synthesis of acridinedione derivatives 4a-j in the presence of 8 mg Pt NPs@GO[a]

Entry	R	R'	Time(min)	Yield[b] (%)	Mp. (^{o}C)	
					Found	Reported
4a	-H	4-Cl	50	94	300-302	309-311 [49]
4b	4-NO_2	4-Cl	75	93	315-317	>300 [50]
4c	3-NO_2	4-Cl	90	94	285-287	285-286 [51]
4d	4-Br	4-Cl	60	95	304-305	>300 [50]
4e	4-F	4-Cl	60	93	280-282	>300 [52]
4f	4-CH_3	4-Cl	60	93	262-265	268-269 [51]
4g	4-OCH_3	4-Cl	75	96	255-257	269-270 [53]
4h	-H	4-Br	60	95	303-305	269-272 [54]
4i	3-NO_2	4-Br	75	96	295-296	—
4j	4-Br	4-Br	120	94	318-320	—

[a] Reactions were carried out with dimedone, aromatic benzaldehyde and various amine, in 2 : 1 : 1 molar ratio.

[b] Yields refer to isolated pure products.

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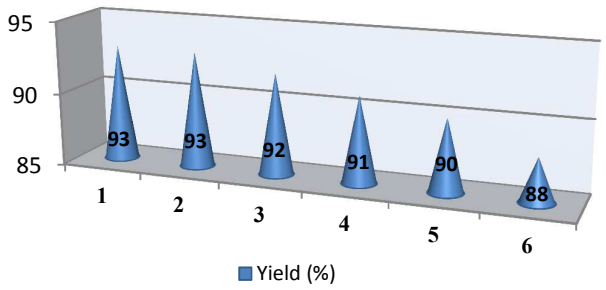
All the products were characterized by melting point, FT-IR, NMR, and HRMS analyses (Fig. S4-13).

The efficiency of the catalyst was also checked by investigation of its reusability. After completion of the reaction, Pt NPs@GO was separated, washed with acetone, dried and reused in another set of reaction. It was observed that the catalyst can be reused for six times with very high yields (Table 6). It should be pointed out that no extra care should be taken in order to store or handle the catalyst due to no air or moisture sensitivity. To the best of our knowledge Pt NPs@GO shows the best reusability performance in literature [24-29].

Table 6. Reusability performance of Pt NPs@GO in the model reaction.



Experimental

The preparation of Pt NPs@GO

Pt NPs was synthesized by sonochemical reduction method. Summarizing this method, ethanol and super hydride were used to reduce the mixture of 0.25 mmol of $PtCl_4$ dissolved in small amount of dehydrated tetrahydrofuran and 0.25 mmol of octylamine ligand by the help of ultrasonicator. The observation of a brown-black colour in the solution indicates the formation of amine-stabilized Pt NPs. Lastly, in the room temperature, the solid Pt NPs was dried under vacuum. Graphene oxide (GO) was synthesized from graphite powder using modified Hummer's method [Supplementary section]. The prepared platinum nanoparticles where mixed in a 1:10 ratio with GO which is used as a support.

General procedure for preparation of acridinedione derivatives (4a-j) using Pt NPs@GO *as the catalyst*

A mixture of 2 mmol dimedone **1**, 1 mmol 4-methyl benzaldehyde **2f**, 1 mmol 4-cloroaniline **3a** and Pt NPs@GO (8 mg) in 3 ml DMF was heated to 75°C continuously for 60 min. The reaction progress was monitored by TLC. The solid product was filtered, washed with 500 ml water, and recrystallized from ethanol (93-96%).

10-(4-bromophenyl)-3,3,6,6-tetramethyl-9-(3-nitrophenyl)-3,4,6,7,9,10-hexahydroacridine-1,8(2H,5H)-dione (4i)

As yellow crystals, (0.571 g, 96 %), mp. (295-296 °C) (ethanol). ^{1}H-NMR (300 MHz, DMSO-d_6) δ (ppm): 0.70 (s, 6H, 2x-CH_3), 0.90 (s, 6H, 2x-CH_3), 1.82 (d, 2H, *J*= 17.47 Hz, -CH_2), 2.02 (d, 2H, *J*= 16.10 Hz, -CH_2), 2.19-2.26 (m, 4H, -CH_2), 5.10 (s, 1H, -CH), 7.35-7.40 (m, 2H, Ar-H), 7.56-7.61 (m, 1H, Ar-H), 7.76-7.86 (m, 3H, Ar-H), 8.00-8.02 (m, 1H, Ar-H), 8.11-8.16 (m, 1H, Ar-H), 7.82 (d, 2H, *J*= 8.68 Hz, Ar-H); ^{13}C-NMR (75 MHz, DMSO–d_6) δ (ppm): 26.56, 29.65, 32.26, 32.45, 41.35, 49.95, 113.07, 119.25, 123.09, 130.33, 131.26, 132.56, 133.53, 138.10, 145.95, 150.74, 195.54; IR (cm^{-1}): 3052 w (Ar-H), 2959 s (-CH), 1635 s (C=O), 1574 s (C=C); HRMS (QTOF-ESI): m/z calcd. For $C_{29}H_{29}BrN_2O_2$: 548.1311: found: 549.1382 ($[M+H]^+$).

9,10-bis(4-bromophenyl)-3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahydroacridine-1,8(2H,5H)-dione (4j)

As yellow crystals, (0.549 g, 94 %), mp. (318-320 °C) (ethanol). ^{1}H-NMR (300 MHz, DMSO-d_6) δ (ppm): 0.70 (s, 6H, 2x-CH_3), 0.90 (s, 6H, 2x-CH_3), 1.78 (d, 2H, *J*= 17.13 Hz, -CH_2), 2.01 (d, 2H, *J*= 16.01 Hz, -CH_2), 2.16-2.22 (m, 4H, -CH_2), 5.00 (s, 1H, -CH), 7.24-7.44 (m, 6H, Ar-H),; ^{13}C-NMR (75 MHz, DMSO–d_6) δ (ppm): 26.47, 29.61, 32.53, 32.92, 41.34, 49.84, 112.71, 121.50, 122.56, 123.23, 130.21, 133.60, 134.75, 137.95, 147.88, 148.59, 151.32, 195.62; IR (cm^{-1}): 3051 w (Ar-H), 2954 s (-CH), 1634 s (C=O), 1575 s (C=C); HRMS (QTOF-ESI): m/z calcd. For $C_{29}H_{29}Br_2NO_2$: 581.0565: found: 582.0484 ($[M+H]^+$).

Conclusions

Easy, effective and practical synthetic method has been developed for the synthesis of acridinedione cycloaddition reactions of the various amine, aromatic aldehyde compounds and dimedone in the presence of highly stable, exceptional reusable, isolable, bottleable, long-lived and highly efficient Pt NPs@GO as the catalyst. Pt NPs@GO is the most efficient catalyst which gives the highest yields of products in the shortest time. Further, Pt NPs@GO shows the best reusability performance possible due to no air or moisture sensitivity of the catalyst. This reusability performance offers advantages like simple work-up and high yields.

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Pt NPs@GO as Highly Efficient and Reusable Catalyst for One-Pot Synthesis of Acridinedione Derivatives

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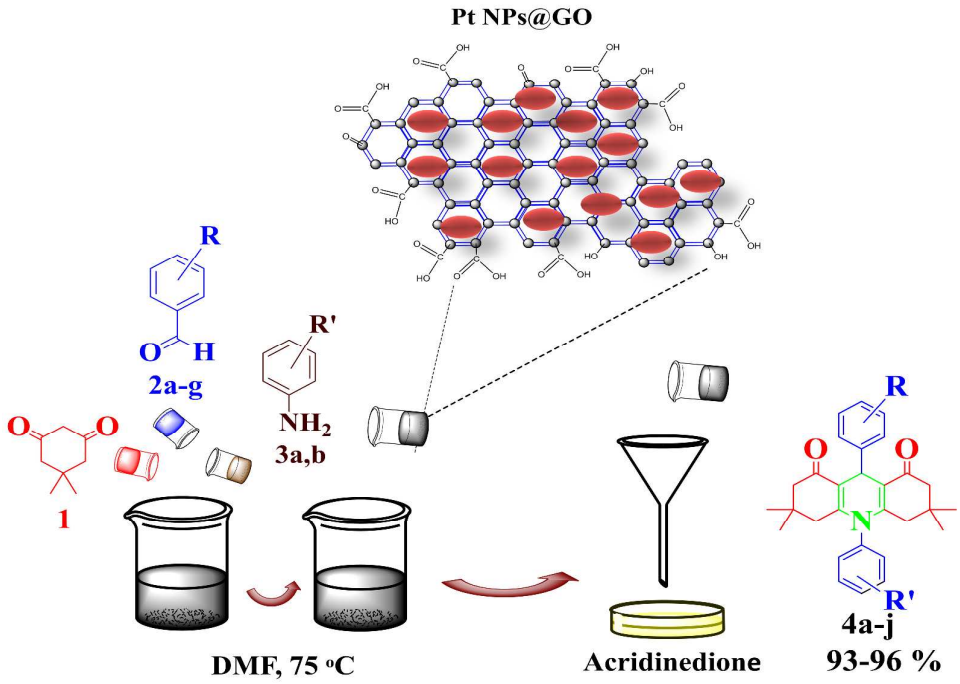

Pt NPs@GO has been used for the first time for synthesizing acridinedione from dimedone, aromatic aldehydes and various amines as a catalyst.