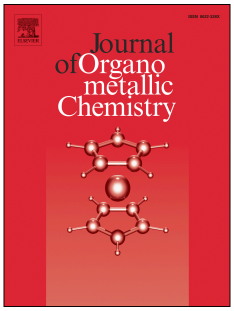

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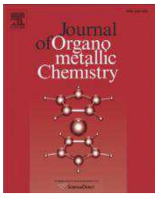

The facile approach to fabricate gold nanoparticles and their application on the hydration and dehydrogenation reactions

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abstract

A mild and practical strategy to prepare gold nanoparticles was developed. This gold particles supported mesoporous silica was fabricated from $AuPPh_3Cl$ under mild conditions and characterized through transmission electron microscopy, energy dispersive X-ray, X-ray power diffraction and X-ray photoelectron spectrometry. Interestingly, it was observed that this gold nanoparticle was effective to the hydration of alkynes and dehydrogenation of alcohols. The catalytic system can tolerate a variety of functional groups to afford the corresponding products in good to excellent yields.

1. Introduction

During the past few decades, noble-metallic nanoparticles have drawn great interest due to their fine electronic, optical, and catalytic properties.[1] These supported gold nanoparticles exhibited favorable catalytic activity such as in the classical CO oxidation, VOCs (Volatile Organic Compounds) oxidation, PROX (Preferential Oxidation) reaction of CO in hydrogen, and so on.[2] Traditionally, supported gold nanoparticles were prepared from $XAu(III)Cl_4$ (X = H, Na, K) under reducing atmosphere condition.[3-5] Considering the importance of the methyl ketone and hydroxy methyl ketone functional group in organic synthesis and medicinal chemistry,[6] we conducted an attempation to the direct alkyne hydration to synthesis of ketones with SBA-15 supported gold nanoparticles as the catalyst. However, the desired product was obtained with low yield even through a series of conditions screeningIn this paper, we developed an easy and practical strategy to prepare SBA-15 supported gold nanoparticles, which were synthesized from $Au(I)PPh_3Cl$ under mild conditions. Interestingly, it was observed that this phosphorus-involved gold nanoparticle was effective to the hydration of alkynes, and the dehydrogenation of alcohols. The catalytic system can tolerate a variety of functional groups to afford the corresponding products.

Our interest in gold catalysis was initiated by triazole ligands or NCAs (non-coordinating anions), which greatly tuned and changed the catalytic activity of gold catalysts in allene synthesis and borrowing hydrogenation reactions.[7] However, it was found that it couldn't recover these gold and iridium catalysts. For the sake of sustainable development and environmental friendly nature, we tuned our interest to the recyclable gold catalysts with high catalytic activity. In the beginning, SBA-15 was selected as the carrier to prepare gold catalyst with classical method.[8] However, only low yield of hydration product was achieved, when the prepared gold nanoparticles from chloroauric acid or potassium chloraurate were used. Interestingly, it is pleased that the gold nanoparticles, fabricated from $AuPPh_3OTf$ under mild conditions, showed enhanced catalytic activity in the hydration of alkynes.

2. Results and Discussion

2.1 Materials

All the chemicals (starting materials) were used without further distillation or purification. De-ionized water was used in all the experiments. Boranetertbutylamine complex (97%), triphenylphosphine (98%), alkynes, alcoholes were purchased from Energy Chemical, SBA-15 were purchased from Nanjing JI Cang Nano Technology Co. Ltd, toluene, chloroform, EtOH were purchased from Sinopharm Chemical Reagent Co., Ltd (China).

2.2 Synthesis of $AuPPh_3Cl$

PPh_3 (524 mg, 2.0 mmol) was dissolved in EtOH (8 mL) at 80 ^oC and then slowly added to $HAuCl_4·4H_2O$ (412 mg, 1.0 mmol) in EtOH (5 mL). The mixture was stirred for 0.5 h after the color changed from yellow to white. The crude product was filtered under reduced pressure. Then, the white solid was dissolved in 5 mL DCM and reprecipitated with 50 mL petroleum ether to give the pure product (Yield: 87%).

2.3 Synthesis of Au nanoparticles

A solution of $AuPPh_3Cl$ (495 mg, 1.0 mmol) was mixed with dodecanethiol (0.5 mL) in toluene (100 mL) to form a clear solution. Then, boranetertbutylamine complex (870 mg, 10 mmol) were added to above solution. The mixture was stirred at 55 ºC for 1 hour before it was cooled to rt (room temperature). Later, EtOH (100 mL) was added to the mixture, and gold nanoparticles were precipitated out.. The black gold nanoparticles were got by centrifuge and washed with EtOH for three times. Finally, the product was dried under vacuum condition.

2.4 Synthesis of Au@ SBA-15

To a 50 mL round-bottom bottle, Au nanoparticles (10 mg), SBA-15 (500 mg) and chloroform (30 mL) was added.The mixture solution was stirred for 1 h at rt (room temperature). Then the solvent was removed by centrifugation and the product was washed several times. Finally, the solid was dried in Freeze-dryer for 12 h to give Au@SBA-15 (2 wt.%). Au@SBA-15 (8 wt.%) was also synthesized with Au nanoparticles (30 mg), SBA-15 (500 mg) and chloroform (50 mL) in a 100 mL round-bottom bottle.

2.5 Dehydrogenation of alcohols

Benzyl alcohol (108 mg, 1.0 mmol), Au@SBA-15 (6 wt.%, 20 mg) and K_2CO_3 (829 mg, 6.0 mmol) was dissoveled in toluene (6.0 mL), the reaction mixture was stirred for 12 h at 110 ^oC. It was monitored by TLC. After the reaction was completed, the solvent was removed under reduced pressure and purified of the crude product by column chromatography on silica-gel afforded the title compound.

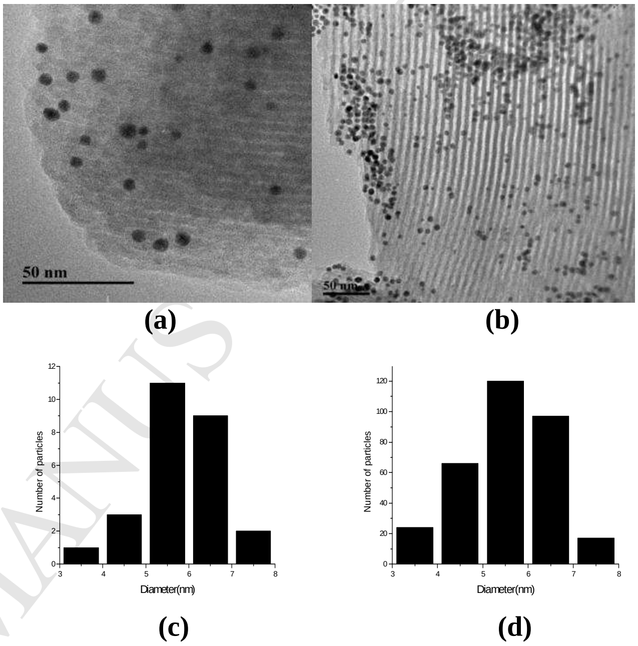
2.6 Hydration of alkynes

To a 25 mL Schlenk tube, Au@SBA-15 (6 wt.%, 20 mg), AgOTf (0.05 mmol) was added to a solution of phenylacetylene (1.0 mmol) in $HOAc/H_2O$ (3.0 mL, 15:1) under ambient air, the resulting mixture was stirred for 6 h at 110 ^oC. It was monitored by TLC. After the reaction was completed, the solvent was removed under reduced pressure and purified of the crude product by column chromatography on silica-gel afforded the desired compound.

Results and discussion

3.1. Characterization of the Au@ SBA-15 catalyst

The structural characteristices of the synthesized catalyst could be observed diametrically from the transmission electron microscopy (TEM). As shown in Fig.1a and Fig.1b, it was observed the gold particles supported mesoporous silica with a few particles. In addition, Fig.1c and Fig.1d showed a maximum number of size between 4-7 nm.



(a) **(b)**

(c) **(d)**

Fig. 1. TEM images of **(a)** Au@SBA-15 (0.8 wt.%), **(b)** Au@ SBA-15 (6 wt.%), **(c)** particles size distribution as figure out from **(a)** of Au@SBA-15 (0.8 wt.%), **(d)** particles size distribution as figure out from **(b)** of Au@SBA-15 (6 wt.%).

Fig. 2 showed the X-ray power diffraction (XRD) pattern of Au@SBA-15, the peak in the 2θ region of 15°-30° was attributed to mesoporous silica and exhibited diffraction lines at 77.6°, 64.6°, 44.4° and 38.2° assigned to the (311), (220), (200) and (111) crystal faces of Au nanoparticles. No other impurity diffraction peak has been observed.

In addition, X-ray photoelectron spectrometry (XPS) was measured to characterize the chemical elements on the surface of Au@SBA-15. As shown in the Fig. 3, XPS full-scan spectrum for the synthesized catalyst revealed the presence of elements such as Au, O, C and Si, which provides an efficient proof for the Au nanoparticles supported by mesoporous silica. Furthermore, the spectral peak at binding energy 87.68 and 83.98 eV for the Au4f5/2 and Au4f7/2 levels in the Fig. 4, indicated most of the Au^{1+} ions were reduced by boranetertbutylamine complex.

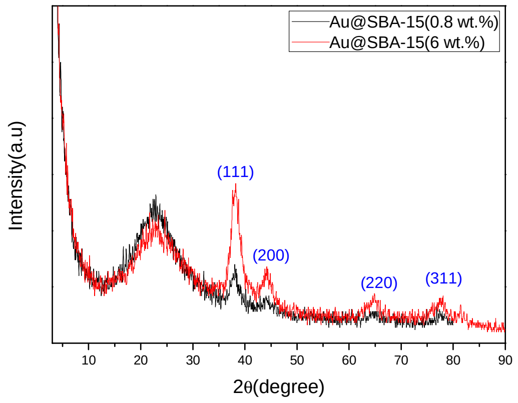


Fig. 2. XRD pattern of Au@SBA-15.

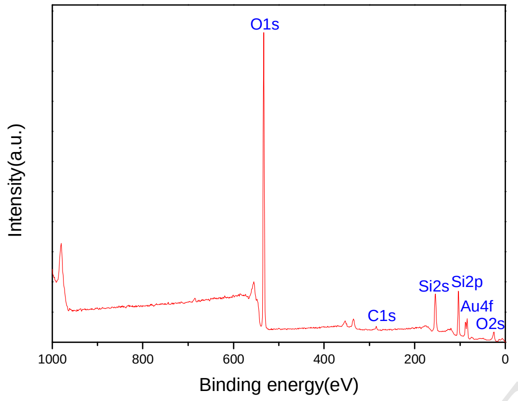


Fig. 3. XPS wide-scan spectrum of Au@SBA-15.

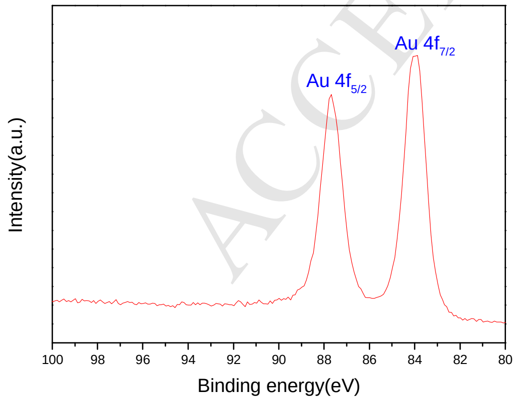
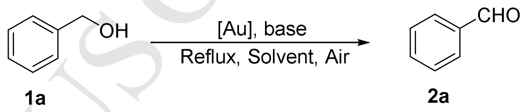


Fig. 4. High-resolution spectrum of Au4f.

With these Au@SBA-15 catalysts in hand, the standard oxidation reaction of alcohols was first explored to examine this catalyst. To our pleasure, the aldehyde product was smoothly achieved in 72% yield (Table 1, entry 1). For a better yield, the reaction conditions screening was conducted and the results were concluded in Table 1. First, the bases such as Cs_2CO_3, NaOH, *t*BuOK, NEt_3 were tested and the results showed the yields ranged from 34 to 92% (Table 1, entries 1-5). Next, solvent screening indicated that toluene is the best solvent, while other solvents like DCM, MeOH, EtOH, dioxane, THF and MeCN gave the yields from 11 to 42% (Table 1, entries 6-11). Additionally, the control experiments were conducted without catalyst or base, however, no desired product was detected (Table 1, entries 12-13).

Table 1. Optimization of reaction conditions ^{a,b}

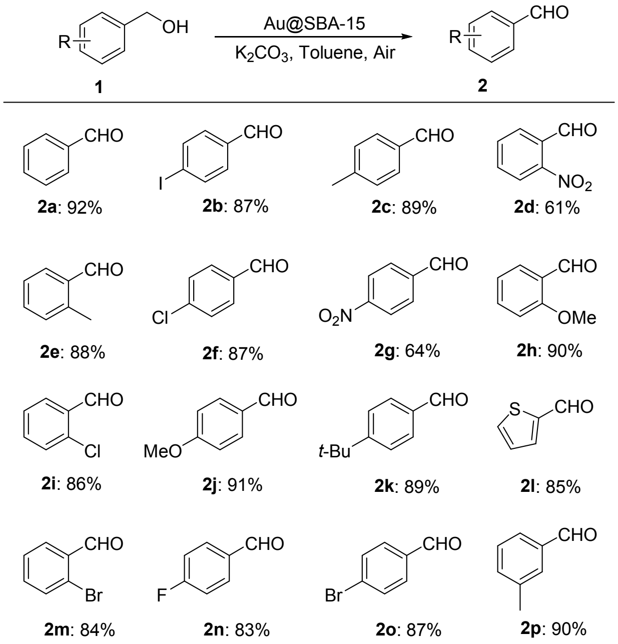


Entry	Catalyst	Base	Solvent	Yield[%]^b
1	Au@SBA-15	Cs_2CO_3	toluene	72
2	Au@SBA-15	NaOH	toluene	63
3	Au@SBA-15	*t*BuOK	toluene	65
4	Au@SBA-15	NEt_3	toluene	34
5	Au@SBA-15	K_2CO_3	toluene	92
6	Au@SBA-15	K_2CO_3	DCM	21
7	Au@SBA-15	K_2CO_3	MeOH	17
8	Au@SBA-15	K_2CO_3	EtOH	11
9	Au@SBA-15	K_2CO_3	Dioxane	42
10	Au@SBA-15	K_2CO_3	THF	14
11	Au@SBA-15	K_2CO_3	MeCN	27
12	-	-	toluene	<5
13	-	K_2CO_3	toluene	<5

^{a} Reagents and conditions: **1a** (1.0 mmol), catalyst (6 wt.%, 20 mg), K_2CO_3 (6.0 mmol), toluene (6.0 mL), air, 12 h, reflux. ^{b} Based on GC.

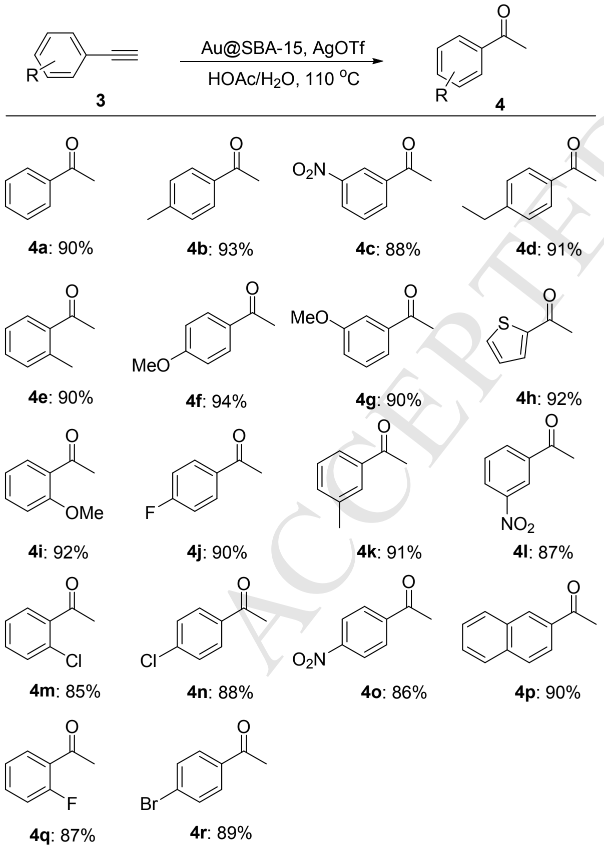
Next, other alcohols were investigated under the best reaction conditions, the results were shown in Table 2. Most of the alcohols were transformed into the corresponding aldehydes in good to high yields. Meanwhile, it is worth noting that the substrates with electron-donating and electron-withdrawing groups had light effect for this reaction. When (2-nitrophenyl)methanol was tested, only moderate yield was achieved. Interestingly, good result could be obtained with 2-thiophenemethanol as the substrate.

Table 2. Substrate expansion of dehydrogenation of alcohols ^{a,b}



[a] Conditions: **1** (1.0 mmol, 1.0 equiv.), Au@SBA-15 (6 wt.%, 20 mg), K_2CO_3 (6.0 mmol, 6.0 equiv.), toluene (6.0 mL), air, 12 h. [b] Based on GC.

Table 3. Substrate expansion of hydration of alkynes [a,b]



[a] Conditions: **3** (1.0 mmol, 1.0 equiv.), Au@SBA-15 (6 wt.%, 20 mg), AgOTf (5.0 mol%.), HOAc/H_2O (3.0 mL, 15:1), 6 h. [b] Isolated yields based on **3**.

The functionalized ketones represent an important class of organic materials and intermediates.[9] In order to demonstrate the importance of Au nanoparticles supported on mesoporous silica, the hydration reaction of phenylacetylene was explored. As shown in Table 3, this reaction could be performed smoothly in presence of gold nanoparticles and the corresponding products were isolated in good to high yields. It should be noted non-coordinating anion could slightly enhance the yields of hydration of alkynes (see Table S1 in Supporting Information for details), which was supported by previous results [10]. The alkynes with electro-withdrawing or electron-donating on the aromatic ring almost produced the same results.

Recycling

To a 25 mL Schlenk tube, Au@SBA-15 (6 wt.%, 40 mg), AgOTf (0.05 mmol) was added to a solution of phenylacetylene (2.0 mmol) in HOAc/H_2O (6.0 mL, 15:1) under ambient air, the resulting mixture was stirred for 6 h at 110 °C. After centrifugation, the recovered catalyst was dried in Freeze-dryer. The hydration of alkynes reaction was carried out for next time with the recovered catalyst from the frontal reaction. Generally, it was pleased to find that the reaction could perform even five times, although a slightly reduced yield was achieved (Fig. 5). To better illustrate this catalyst, XRD experiment was investigated to test the recovered gold catalyst and the result revealed that almost no loss of gold in the supporters were observed (Fig. 6).

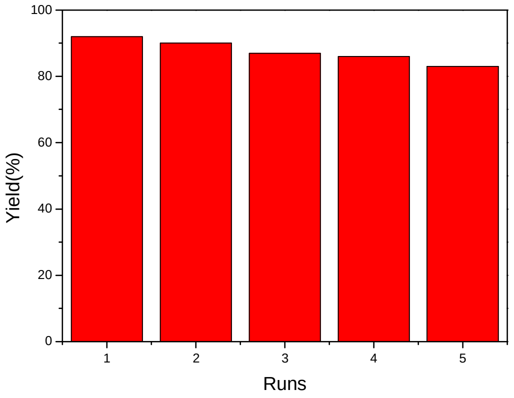


Fig. 5. Recyclability of Au@SBA-15 in the dehydrogenation of alcohols reaction.

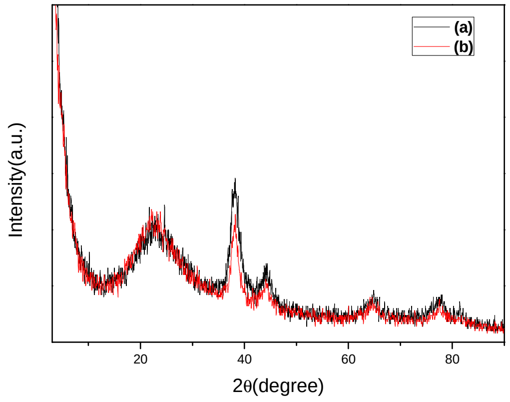


Fig. 6. XRD diffractograms: **(a)** fresh Au@SBA-15 (6 wt.%); **(b)** Au@SBA-15 (6 wt.%) after five runs.

Conclusions

In conclusion, we have developed a practical strategy to prepare gold nanoparticles supported by SBA-15, which was fabricated from $AuPPh_3Cl$ under mild conditions. It is worth noting that this gold nanoparticle was effective to the hydration of alkynes and dehydrogenation of alcohols. Compared to the literatures, this catalyst with Ph_3PAuCl as the precursor provided much higher yields than the previous reports. Ph_3PAuCl is easy to store for its stability, while $HAuCl_4$ was to hygroscopy and thus difficult to store in the air. The catalytic system can tolerate a variety of functional groups to afford the corresponding products in good to excellent yields. The catalyst system is recyclable at least five times without obvious catalytic activity loss.

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Graphical abstract

The facile approach to fabricate gold nanoparticles and their application on the hydration and dehydrogenation reactions

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Au@SBA-15: From AuPPh$_3$Cl

High catalytic activity

ABSTRACT: A mild and practical strategy to prepare gold nanoparticles was developed. This mesoporous silica-coated gold nanoparticle was fabricated from AuPPh$_3$Cl under mild conditions without high temperature and characterized through transmission electron microscopy (TEM), energy dispersive X-ray (EDX), X-ray power diffraction (XRD) and X-ray photoelectron spectrometry (XPS). Interestingly, it was observed that this gold nanoparticle was effective to the hydration of alkynes and dehydrogenation of alcohols. The catalytic system can tolerate a variety of functional groups to afford the corresponding products in good to excellent yields.

- A mild and practical strategy to prepare gold nanoparticles
- Gold particles supported silica was fabricated from $AuPPh_3Cl$
- This gold nanoparticle was effective to hydration and dehydrogenation