



The first gold nanoparticles supported SBA 15 functionalized aminopropylsilane as an efficient catalyst for the synthesis of β -acetamido ketone derivatives

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Abstract

Gold nanoparticles were deposited into the mesoporous aminopropylsilane functionalized SBA-15 by impregnation. The catalyst was characterized by XRD, UV–Vis, BET, MET, and IR. The characterization demonstrates that the gold nanoparticles situated in the SBA-15 pores with small sizes in an average of 1–8 nm. The catalyst exhibited a highest catalytic performance in the synthesis of β -acetamido ketones molecules with a short time reaction.

Keywords Acetamido ketones · Gold nanoparticles · SBA-15-NH₂ · Heterogeneous catalyst

Introduction

The SBA-15 is a highly ordered two-dimensional hexagonal structure synthesized in 1998 by stucky et al. [1] which has attracted particular attention because of its properties as a high specific surface [2], large pore size, thick silica walls favoring thermal and hydrothermal stability [3, 4], and a high density of hydroxyl on the surface. It is easily synthesized in acidic medium, using as structural agent as the amphiphilic triblock copolymer [1]. Indeed, based on these typical characteristics, SBA-15 has an exciting field of research of great scientific importance and different applications, as a heterogeneous catalyst in waste water treatment, hydro-cracking, biodiesel production and drug delivery [5, 6].

Many researchers have been carried out in order to develop SBA-15 where it was necessary to modify it in order to widen its application for the design of new

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catalysts. However, various metals were used such as Al-SBA-15 [7], Vx-SBA-15 [8] Zr-SBA-15 [9], Ag/SBA-15 [10], Au/SBA-15 [11], and MoO₃/SBA-15 [12].

Mesoporous silica functionalized by introduction of organic group is one of the approaches achieved by scientists; the organic active species are oriented towards the surface and facilitate the modification and introduction of new physicochemical characteristics. The organo-functionalization on the surface of mesoporous silica can be carried out generally either by integrating functionalities directly by co-condensation also called direct synthesis [13–15], or by the grafting method also called post-synthesis [16], these methods present advantages for example the control of morphology, functionalities, and the dimension of the pores but also disadvantages such as the reduction of the structural properties and the manufacture on a large scale. The grafting method consists in modifying the surface of silica with organic groups by creating covalent bonds between the active function and the internal surface of the pores by means of silanol (Si–OH) groups by using trichloro or trialkoxy-organosilane or silylamines as organic precursors [17]. On the other hand, co-condensation is a simpler and faster synthesis and allows the obtain a more homogeneous distribution of the organic groups in the material compared to grafting method, in addition it avoids the possibility of pore blocking. Nevertheless, this method also has some disadvantages because there is an antagonistic effect between the degree of mesoscopic order and the concentration of the organosilanes in the reaction medium. For this purpose, in the presence of a structuring agent, an organosilane precursor (R'O)₃SiR is combined with a silica precursor salt of the type Si(OR')₄, generally tetraethylorthosilicate TEOS (R'=ethyl group) was used [18]. However, organo-functionalized SBA-15 has shown very promising results in various applications such as adsorption [19, 20], biodiesel production [21], drug delivery [22], water decontamination [23], and corrosion inhibitor [24]. In addition, supported SBA-15 catalysts were also used in organic synthesis such as the synthesis of propargylamines [25, 26], imidazoles [27], and pyrroles [28].

Acetamido ketones derivatives are suitable mediators for a variety of pharmaceutically and biologically essential compounds [29]. Also, they have an important moiety used as precursors in organic synthesis like 1,3-amino alcohols [30], 1,3-aminoacids [31] as well as their for the preparation of diverse bioactive molecules such as the antibiotics nikkomycins or neopolixins [32] and other different antibiotic drugs [33]. Generally, the Dakin-West reaction is one of the most popular tools used for the synthesis of β-acetamido ketones [34]. In 1994, a simple procedure for preparation of β-acetamido ketone was developed by Bhatia et al. [35], which represents a one-pot multi-component coupling condensation of an enolizable ketone, aromatic aldehyde, and acetonitrile in the presence of acetyl chloride and catalyst, so this transformation became a model where different catalysts was developed like H₇SiV₃W₉O₄₀ [36], Fe₃O₄@SiO₂-GA-M(Pc) [37], NiFe₂O₄@SiO₂-PPA [38], SBA-15-Pr-SO₃H [39], IL [(PS)₂pi][OTf]₂ [40], H₅PW₁₀V₂O₄₀ [41], Cellulose sulfuric acid [42], and Mg(HSO₄)₂ [43]

Gold is known to have an exceptionally rich and exciting chemistry especially as homogeneous [44] and heterogeneous [45] catalysts. However, gold nanoparticles catalysts were used in organic synthesis several times due to their capacity to activate C–H and C–C bands. Their activity is directly related to the particles size,

dispersion, and the characteristics of the support, which can control the metal-support interaction [26, 46, 47]

The development of solid heterogeneous catalysts with various organic functionalities is considered to be an attractive topic due to the advantage of easier separation, recovery and environmental improvement compared with homogeneous catalyst. In this context, this paper demonstrates for the first time, the synthesis and characterization of heterogeneous catalyst; amine-modified mesoporous silica SBA-15 (Au/NH₂-SBA-15) and their activity in the synthesis of β -acetamido ketones via one pot condensation of an enolizable ketone, an aromatic aldehyde, and acetyl chloride at room temperature.

Experimental

Preparation of SBA-15

The synthesis of SBA-15 was realized using the Zhao's method reported previously [48]. At room temperature under stirring, a total of 4 g of block copolymer surfactant Pluronic 123 (EO₂₀PO₇₀EO₂₀) used as a template, was dissolved in a mixture of ultra pure water (120 mL, 6.67 mol) and 7.5 mL of HCl (0.24 mol, pH=0.83). Next, the solution was heated up to 40 °C followed by addition of 9.15 mL of tetraethyl orthosilicate TEOS. The mixture stirred for 24 h at the same temperature. Then, this later was placed into a Teflon-lined autoclave for 48 h at 100 °C. Finally, the solid product was filtered, dried at room temperature overnight, and calcined in air at 500 °C for 4 h.

SBA-15 functionalization with aminopropylsilane

Before the SBA-15 surface functionalization, 3 g of calcined SBA-15 was refluxed in 100 mL of distilled water for 6 h, and then the mixture was centrifuged and dried at room temperature for 24 h. The product was mixed with 1.5 g of n-octadecyl-trimethoxysilane and 100 mL of chloroform under stirring at 25 °C for 24 h. The solid was washed abundantly with chloroform and dried at room temperature. Next, 1.5 g of APTES was slowly added to the obtained sample diluted by 100 mL of chloroform under stirring for 24 h under. The mixture was then filtered and washed several times with chloroform and finally the product was dried overnight at 80 °C. The catalyst was denoted SBA-15-NH₂.

Preparation of 1 wt% Au/SBA-15-NH₂:

1 g of the functionalized SBA-15-NH₂ support was stirred for 3 h in 139 mL of chloroauric acid (HAuCl₄·3H₂O). After filtration and washing with deionized water of mixture product, the recovered solid was reduced under hydrogen atmosphere at 100 °C for 2 h.

General procedure for synthesis of β -acetamido ketones

In a round-bottomed flask, 2 mmol of acetyl chloride, 1 mmol of aromatic aldehyde, and 1 mmol of acetophenone derivative in the presence of 0.03 g of Au-SBA-15-NH₂ were stirred in 3 mL of acetonitrile at room temperature for appropriate time. Progress of the reaction was monitored by TLC. After the reaction was completed, the catalyst was separated and the reaction mixture was extracted with dichloromethane and water and dried over Na₂SO₄. After evaporation of the solvent under reduced pressure, the crude product was purified by column chromatography (n-hexane/n-ethyl acetate).

Results and discussions

Catalyst characterization

Wide-angle XRD of support and final catalyst are described in (Fig. 1). A broad peak is observed at $2\theta = 23^\circ$ corresponding to the formation of amorphous silica mesopores [49]. No visible diffraction peaks attributed to the Au NPs could be identified, which reveals that gold Au nanoparticles are well dispersed on the surface of the support or are in the form of non-crystal phases [50]. Also, the samples present the same diffraction peaks even after the organo-functionalization. In addition, the low-angle powder X-ray diffraction of the catalyst Au/SBA-15-NH₂ gave 2θ values of 1.03° , 1.65° and 1.88° which are indexed to the (100), (110) and (200) reflections of an ordered hexagonally SBA-15 materials [51]. The highest peak refers to the

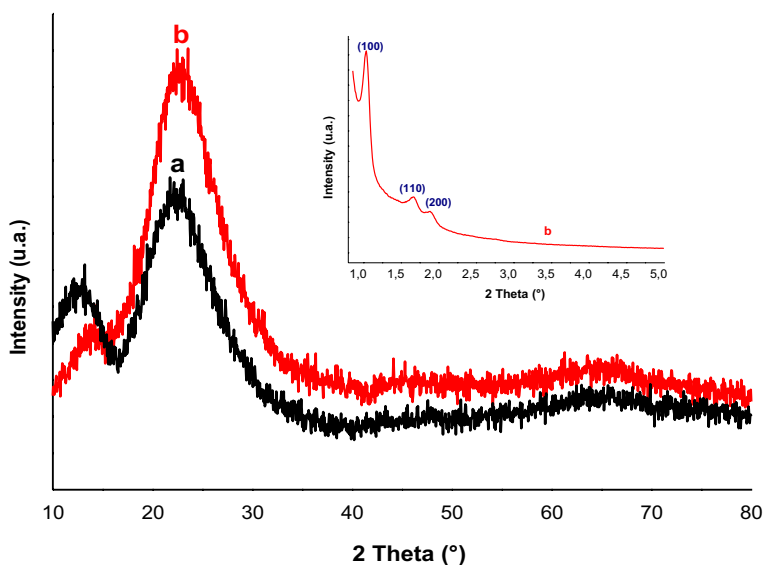


Fig. 1 Powder X-ray diffraction patterns of **a** SBA-15-NH₂, and **b** Au/SBA-15-NH₂

periodic inter-space area between the walls of mesoporous channels and the two lower peaks which is due to the elevated order of SBA-15 structure channels [52]

Fig. S1 shows the UV–Vis spectrum of Au/SBA-15-NH₂. From 200 to 350 nm spectral zone represents the structure of the support. Moreover, the surface plasmon resonance (SPR) band at ~520 nm thus identifying small gold nanoparticle with spherical shape and size superior than 2 nm. Also, that certifying that Au NPs have been incorporated in the support [53, 54]

Fig. 2 demonstrates the FTIR spectrum of the support and the catalyst, a strong peak situated around 1087 cm⁻¹ and 803 cm⁻¹ assigned to the asymmetric vibrations of the Si–O–Si bond. Another peaks reveals at 1640 cm⁻¹ could be attributed to H₂O adsorbed in mesoporous structure [55]. This spectrum exhibits bending and stretching vibrations of the Si–OH bond as a small band at about 956 cm⁻¹ [51]. Furthermore, the appearance of the C–H stretching of methyl groups containing amino silane at 2800–3000 cm⁻¹, indicates that the NH₂ groups have been incorporated into the backbones of the mesoporous silica (–NH₂) to the SBA-15 support. Moreover, the presence of –NH₂ after the APTES modification can also be confirmed by the vibration peak of N–H stretching at 500–750 cm⁻¹ in the pattern of SBA-15-NH₂. At the same time, the N–H stretching vibration peak at 500–750 cm⁻¹ was no visible in Au/SBA-15-NH₂, which assigned to the coordination interaction between –NH₂ and Au [56].

In order to understand the structure of the catalysts, low temperature adsorption–desorption isotherms at 77 K and pore size distribution analyses (Fig. 3) were done. The analysis showed that all samples belong to type IV with H1 type hysteric loop (P/P0 > 0.4), which represents capillary condensation of the ordered mesoporous structure. The resulting textural properties of these isotherms are summarized in (Table 1). We note a decrease in specific surface area which can be explained by the partial obstruction of the mesopores by the gold particles.

Fig. 4 shows the HRTEM images and particle size histogram for supported gold nanoparticles. The image of the support (Fig. 4a) shows well-ordered hexagonal pore structure and regular periodicity of SBA-15, this indicating that the addition

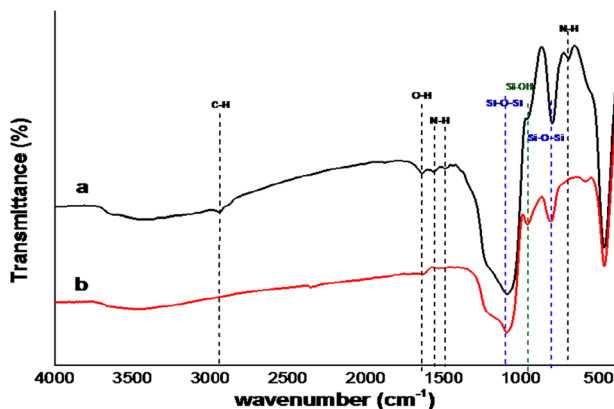


Fig. 2 FTIR spectra of samples a support SBA-15-NH₂, and b Au/SBA-15-NH₂

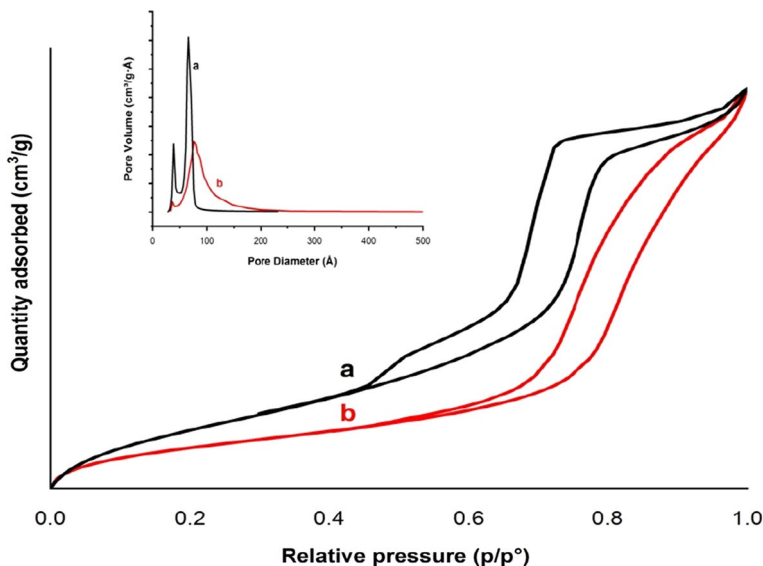


Fig. 3 Nitrogen adsorption isotherm and pore distribution curves (BJH method) of **a** support SBA-15-NH₂, **b** Au/SBA-15-NH₂

Table 1 Textural characteristic of SBA-15, SBA-15-NH₂ and Au/SBA-15-NH₂

Sample	BET surface area (m ² g ⁻¹)	Pore volume (cm ³ g ⁻¹)	Pore size (Å)
SBA-15	871.857	1.693	70.946
SBA-15-NH ₂	282.017	0.513	61,319
Au/SBA-15-NH ₂	208.485	0.627	94,461

of organic function group to the SBA-15 materials has no distinct influence on their morphology. The TEM analysis reveals also a highly dispersed and uniformly sized of Au NPs. The structure of SBA-15 mesoporous silica is retained after gold deposition and the Au NPs were clearly observed mainly inside porous channels of SBA-15 (Fig. 4c). As can be seen, the histogram of the particles size distribution (Fig. 4d) indicates that particle size of gold in Au/SBA-15-NH₂ range from 2 to 8 nm with an average size of 3 nm.

Catalytic activity

In order to find the optimal reaction condition for the synthesis of β -acetamido ketones, the reaction of benzaldehyde, acetophenone, and acetyl chloride was selected as a model reaction (Table 2). Initially, the reaction was performed in the absence of catalyst Au/SBA-NH₂ for 3 h. Result indicates that the reaction could not bring about any yield product and no reaction occurred even after an extended reaction time to 24 h

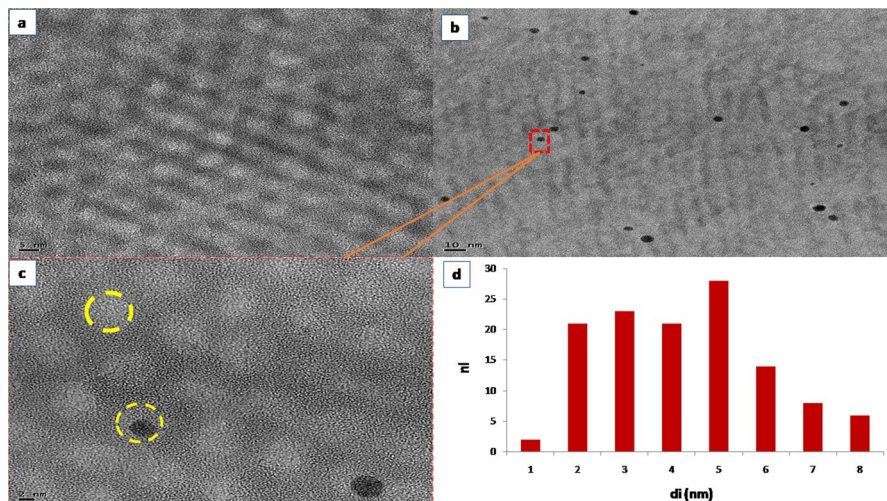
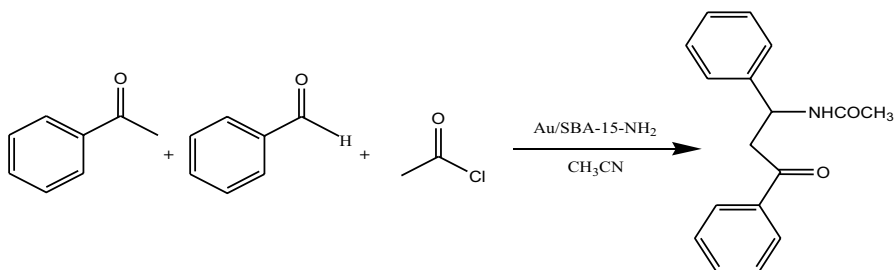


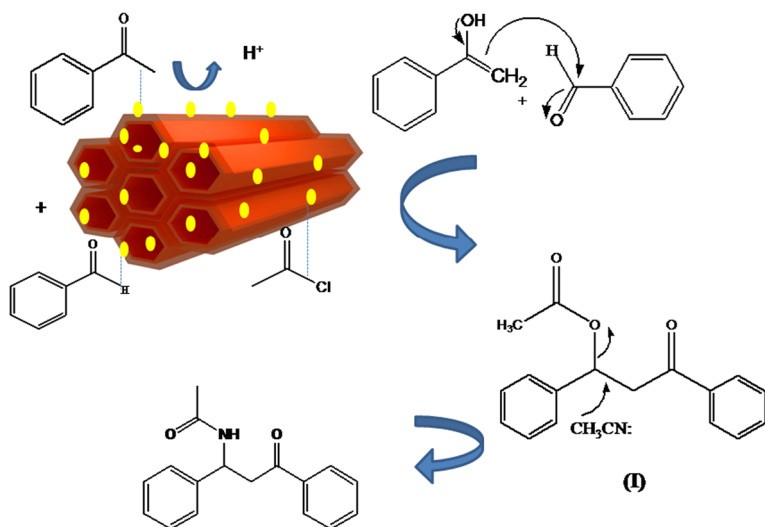
Fig. 4 TEM micrograph images of **a** SBA-15-NH₂, **b** and **c** Au/SBA-15-NH₂, **d** gold particles size distribution

Table 2 Effect of the time and catalyst amount on the synthesis of β-(phenyl)propiophenone in the presence gold catalyst



Entries	Catalysts	Mass (g)	Time (h)	Yield (%)
1	Au/SBA-15-NH ₂	–	3	–
2	Au/SBA-15-NH ₂	–	24	–
3	SBA-15	0.01	3	–
4	SBA-15-NH ₂	0.01	3	–
5	Au/SBA-15-NH ₂	0.01	3	58
6	Au/SBA-15-NH ₂	0.01	6	42
7	Au/SBA-15-NH ₂	0.01	24	39
8	Au/SBA-15-NH ₂	0.02	3	61
9	Au/SBA-15-NH ₂	0.04	3	77
10	Au/SBA-15-NH ₂	0.06	3	87
11	Au/SBA-15-NH ₂	0.08	3	94

Reaction conditions: Benzaldehyde (1 mmol), acetyl chloride (2 mmol), acetophenone (1 mmol), acetonitrile (3 ml), catalyst, Room Temperature.



Scheme 1 The plausible mechanism for the β -acetamido carbonyl compounds synthesis in the presence of gold catalyst

(Table 2, entries 1 and 2). Then, the supports SBA-15, SBA-15-NH₂ were used with amount equal to 0.01 g, but no product was afforded (Table 2, entries 3 and 4). After introduction of gold NPs, the reaction achieved with a yield of 58% (Table 2, entry 5). This result revealed that gold nanoparticles were responsible on the catalyst activity.

To increase the product yield, the reaction was carried for 6 h and 24 h, with yields equal to 42 and 39 (Table 2, entries 6 and 7). The increase of catalyst amount to 0.08 g, leads to the decreases of yield to 94%. Using these conditions, several β -acetamido ketones derivatives were prepared with high yields 80–90% (Scheme S1).

The result above demonstrates that gold nanoparticles are responsible on the catalyst activity, this due to their contribution in the reaction as Lewis acid [57], so according to this information and the literature [38, 58], a proposed mechanism presented in Scheme 1 for the β -acetamido ketone synthesis via aldehyde, acetophenone, and acetylchloride condensation in acetonitril. In the first, the gold nanoparticles activate the C–H band of aldehyde and ketone by enolization of the acetophenone producing a proton (H⁺). Also, the gold nanoparticles were known for the C–Cl band activation, so the reaction between acetylchloride and intermediate given by the reaction between aldehyde and ketone will be faster to produce intermediate I. Then, this later reacts with acetonitrile to give the final desired product.

Conclusion

In the present paper we demonstrate for the first time that gold nanoparticles supported on modified SBA-15 is an effective catalyst for the one pot synthesis of the β -acetamido carbonyl compounds in short time. However, the use of SBA -15 or modified SBA-15 does not give product because the reaction needs an acidic sites

which were given by supported gold nanoparticles. Also, the characterization reveals that gold nanoparticles were supported with good dispersion and small sizes and affect directly on the catalyst activity.

Supplementary Information The online version contains supplementary material available at <https://doi.org/10.1007/s11144-023-02552-3>.

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Data Availability Data available for this research in Supplementary information.

Declarations

Conflict of interest Conflict of interest the authors declare that they have no conflict of interest.

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