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Mini Review

Brief Review on Amino-Functionalized Mesoporous Silica Nanospheres for Nanoreactors by Nanoengineering

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ABSTRACT

For MSN Nanoengineering, mesoporous silica Nanospheres (MSNs) must be selectively functionalized. To create functionalized MSNs with externally attached amino groups, we have combined "surface-protected etching method" and "cationic surfactant assisted etching technique" in this study. The catalysts have high catalytic activity for the nitroaldol reaction between nitromethane and benzaldehyde thanks to the externally connected NH2 groups. These NH2-MSNs can also host gold nanoparticles, which have excellent catalytic activity for reducing 4-nitrophenol. It is conceivable that the synthesis technique created in this work might also be applied to nanoengineered MSNs, opening up possibilities for the construction of Nanoreactors.

Keywords: Nanoreactors; Nanoengineering; Nitroaldol; Organosilanes

INTRODUCTION

Mesoporous silica nanoparticles (MSNs) have drawn a lot of interest in the fields of adsorption, catalysis, and nanomedicine due to their tunable morphology, porosity, and functionalization. Numerous efforts have been made to functionalize MSNs with organic groups either on the exterior surface or the interior surface, which endows MSNs with a variety of properties and broadens the uses of these materials. Typically, co-condensation or post-grafting on the exterior surface of pre-silylanized materials can be used to create internally functionalized MSNs. The procedure of post-grafting organosilanes onto materials that include surfactants, followed by the removal of the surfactant, is the most common method of generating externally functionalized MSNs. However, this approach may result in pore clogging, particularly when the content of the functional groups is high, because the mesopores were produced before the inclusion of organo-functional groups [1]. Therefore, It is extremely desirable to develop novel techniques for making externally functionalized MSNs with a high loading of functional groups and for nanoengineering MSNs. MSNs' surfaces have been well-engineered utilising a variety of techniques. To create hollow, yolk-shell, and porous nanoparticles, silica-based etching method has been widely used. For instance, Yin and colleagues presented a simple surface-protected etching method based on suitable etching agents, such as hot water and basic media, to synthesise MSNs. However, because no surfactant was utilised to direct the pore-forming process, the distribution of pores was not uniform. An alternative method for creating hollow mesoporous silica spheres was "cationic surfactant assisted selective etching," which would allow for the etching of the silica shell without the creation of MSNs. In this study, we prepared externally functionalized MSNs and examined their catalytic properties by combining "surface-protected etching method" and "cationic surfactant assisted etching strategy." The resulting catalysts have a high amino group loading on the exterior surface, which is essential for adding more amino groups to the Au nanoparticles and other catalytic applications. According to the described procedure, Stöber nanospheres (SN) were created [2]. Without further treatment, a mixture of toluene (10 mL) and SN was prepared using additional reagents supplied from Sinopharm Chemical Reagent (0.30 g). The material was collected by centrifugation and three times rinsed with ethanol to eliminate unreacted organosilanes after being stirred for 24 hours at 110°C. The final product was identified as ASN.

Synthesis of Externally Functionalized MSNs with Amino Groups

ASN (0.05 g) and H_2O (9 mL) were added to the dispersion system in a typical synthesis, along with 1 mL of CTAB aqueous solution (12.5 mg mL1), and the mixture was agitated at room temperature for 1 h [3]. To get various samples, Na_2CO_3 (0.21 g) was then added and mixed at 35°C for various lengths of time (2 h, 4 h, 6 h, 15 h, and 48 h). According to the varying reaction times, the materials were given the names AMSN-n (2, 4, 6, 15, 48) after being extracted from the template by refluxing the material in 20 mL of ethanol with 1 mL of HCl.

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Catalytic Reaction Protocol

In nitromethane (3 mL), with benzaldehyde (100 L), AMSN (0.020 g), and 90°C for 24 hours, the nitroaldol reaction was carried out. GC was used to measure the conversion using an HP-5 capillary column [4].

4-Nitrophenol reduction reactions

The reaction was carried out in a cuvette and examined in-situ using UV/Vis. Typically, an aqueous dispersion of the catalyst (0.04 g/L), fresh NaBH4 aqueous solution (0.4 M, 1 mL), and water was added along with an aqueous solution of 4-nitrophenol (50 L, 0.005 M) (2 Ml) [5]. Using UV light, the reaction's development was timed at certain intervals.

RESULTS

The fundamental synthesis process and the underlying idea are schematically depicted. As the parent material, first the SN with a particle size of about 220 nm was synthesised. The amino functionality employed in base catalysis, the adsorption of heavy metal ions, and the immobilisation of biomolecules was selected as the surface-protected group for the surface nanoengineering of silica spheres [6]. The mesostructure of the materials formed at various etching times was first characterised in order to monitor the etching process using the nitrogen sorption technique. There was no discernible pore size distribution when the etching period was 2 hours; the surface area and pore volume were only 202 m^2/g and 0.21 cm^3/g, respectively. The surface area and pore size decreased when the etching duration was extended to 4 h. A modest pore size distribution peak could be seen as volume climbed to 388 m^2/g and 0.50 cm^3/g, respectively [7]. The pore size distribution peak becomes more visible with an additional increase in the etching time to 6 h given the surface area and pore volumes are 510 m^2/g and 0.46 cm^3/g. The highest surface area of 703 m^2/g and the biggest pore volume of 0.68 cm^3/g were obtained when the etching period was 15 h, resulting in the largest pore size distribution peak. The etching period is increased further to 48 hours, however the textural parameters are not further enhanced.

DISCUSSION

AMSN-15 has been chosen as the typical sample for TEM characterization in order to gain further understanding of the process of surface engineering and the creation of mesoporous structures. Mesopores that resemble worms can be seen on the surface of AMSN-15. The particle size is similar to the parent material at about 220 nm. ASN demonstrating that the etching reaction took place inside the material [8]. The surface protection effect is what causes the etching of the inner section and the preservation of the shell. Because the organic silane agents contain organic carbon chains, when the surface of the silica nanospheres was functionalized with them, a hydrophobic layer of the Si-O-Si framework may form next to the shell. The hydrophobic layer that had evolved might withstand attack from hydrophilic etching chemicals like Na_2CO_3 aqueous solution. Contrarily, it is believed that the etching agents will be able to reach and etch the inside of the silica spheres without the protection of organic silane agents [9].

The amino moieties on the surface of MSNs following cationic surfactant assisted etching method are still there because the surface was successfully shielded from the etching reaction. ICP analysis reveals that AMSN-15 has a N element content of approximately 1.0 mmol/g, demonstrating the viability of our technique to manufacture organo-functionalized MSNs with high organic group contents [10]. The AMSN-15 was used to support gold nanoparticles (Au@AMSN-15) for the catalyzing of the 4-nitrophenol (4-NP) reduction reaction and to catalyse the ni- tramadol reaction in order to test the preservation of the amino group's activity and that the amino group is on the external surface of silica nanoparticles. It has been demonstrated that AMSN-15 is particularly effective at catalyzing the crucial nitroaldol reaction, which produces nitro alkenes necessary for the synthesis of pharmaceutical drugs. The 95% conversion rate shows that the amino group of AMSN-15 added by our technique maintained high base catalytic activity [11]. However, when the catalyst was reused three times, less than 50% conversion was obtained, which has previously been noted by other researchers and may be caused by the irreversible creation of some by-products.

CONCLUSION

According to the TEM pictures, gold nanoparticles with a size of around 5 nm have virtually completely coated the materials' external surface after deposition, proving that our method successfully produced materials with external amino functionalization. As a model reaction for assessing the catalytic performance of metal nanoparticle catalysts, could also effectively catalyse 4-NP reduction processes [12]. Without the catalyst, no change was seen in the UV-Vis spectrum of the aqueous solution of 4-NP including NaBH4, which displayed an absorption peak at about 400 nm. Following the addition, a new peak at 295 nm formed, suggesting that the reduction of 4-NP was swiftly catalysed by. The absorption peak at 400 nm had been clearly decreasing with the reaction as it was happening. The catalyst converts 4-nitrophenol at a rate exceeding 95% in under 20 minutes. Additionally, a linear relationship between ln(Ct/C0) and reaction time is seen, indicating that the reaction follows first order reaction kinetics. Ct is the concentration at time t, and C0 is the initial concentration. As a result, it is determined that the catalytic reaction's apparent rate constant is roughly 0.14 min1. Our method is effective for creating externally functionalized MSNs since we were able to successfully surface engineer an amino-functionalized material to serve as a base catalyst and support for gold nanoparticles.

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None

Conflict of Interest

None

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