

motecutes

1–30 November 2015 chaired by Dr. Julio A. Seijas Vázquez

A new synthesis of cellulose-based silver nanocomposite and its catalytic

performance

Ali Maleki,* Hamed Movahed, Reza Paydar

Catalysts and Organic Synthesis Research Laboratory, Department of Chemistry, Iran University of Science and Technology, Tehran 1684613114, Iran; Email: maleki@iust.ac.ir

Abstract: In the present work, cellulose-based silver nanocomposite is synthesized by a new method and used as a catalyst for the synthesis of heterocyclic compounds. To study the morphology and characterization of the nanocomposite, scanning electron microscopy (SEM) and X-ray diffraction (XRD) were obtained.

Keywords: Cellulose, nanocomposite, Ag, catalyst, imidazole.

Introduction

In the recent years, nonoscince has been one of the hottest sciences between chemists, physicist, biologists and other field scientists because of its novel and unique applications in various fields. Accordingly, preparing different composites in nanoscale for several applications seems to be essential [1]. Ag nanoparticles in recent years have attracted much attention due to their unique properties at the nanoscale and wide range of applicability for optical, catalytic, magnetic, electrical and antimicrobial devices [2]. Having such properties has been caused to create so much interest between chemists and

physicists [3]. In particular, chemists using polymers and especially biopolymers in their researches have been convinced that introducing metal nanoparticles such as Ag nanoparticles to polymers and biopolymers surfaces is an ideal way to evaluate polymers and biopolymers properties and applicability [4].

Cellulose fiber that is obtained from wood annual plants and agricultural by-products is an abundant renewable resource. Cellulose as a biopolymer and owing excellent chemical, physical and mechanical properties has created so much interest for scientists in the recent decades [5]. As a result, inducing Ag nanoparticles to cellulose surface seems to be essential. In connection with our previous works about introducing green nanocatalysts for important reactions [6], herein we have induced Ag nanoparticles to cellulose surface and we have proved that it is a green nonocomposite to catalysis synthesis of 2,4,5-trisubstituted imidazoles reaction under solvent free condition with excellent yields.

Experimental

General

All the solvents, chemicals and reagents were purchased from Merck, Fluka and Aldrich. Melting points were measured on an Electrothermal 9100 apparatus and are uncorrected.

Synthesis of cellulose/Ag nanocomposite

2 g of PEG-400 and 9 g of NaOH were loaded into 90 ml of ultrapure water to prepare an aqueous solution of PEG/NaOH. Then 1.5 g of cellulose was added to the mixture and swelled for 3 h at room temperature. Afterward, 0.1 g of formaldehyde added to the cellulose suspension. Then 0.3 g of AgNO₃ added to a PEG/H₂O (3 mL: 20 mL) mixture for preparing silver nitrate solution and the resultant solution was loaded to the cellulose suspension drop by drop in a period of 3h at room temperature.

Synthesis of 2,4,5-trisubstituted-1H-imidazoles

The mixture of benzoin or benzil (1 mmol), a benzaldehyde (1 mmol) and ammonium acetate (2.5 mmol) was heated at 100 °C (bath temperature) in the presence of cellulose/Ag (0.015 g) under solvent-free conditions. After the completion of the reaction, as indicated by TLC, the resulting reaction mixture was dissolved in 10 mL of EtOH and filtered to separate the catalyst. The filtered solution retaining product was placed in the refrigerator to obtain pure crystalline products in good-to-high yields.

Results and discussion

First, cellulose/Ag nanocomposite was prepared as described in the materials and method section. The schematic structure of the prepared nanocomposite is shown in Fig. 1.



Fig. 1. The schematic structure of the prepared nanocomposite.

To study the morphology and characterization of cellulose/Ag nanocomposite, scanning electron microscopy (SEM) and X-ray diffraction (XRD) patterns were used. SEM images shown in Fig. 2a and b represent cellulose/Ag nanocomposite synthesized in this work. The nanostructure of the nanocomposite is clearly evident with sizes ranging from 30 to 50 nm.



Fig. 2. SEM images of the prepared nanocomposite.

XRD patterns shown in Fig. 3 result further certify the formation of Ag NP in cellulose microcrystal. In these patterns the diffraction peaks at around $2^{\text{(f)}} = 15.2^{\circ}$, 16.8° , and 22.8° were attributable to cellulose microcrystals, and peaks at around $2^{\text{(f)}} = 38.4^{\circ}$, 44.2° , 64.4° , 77.3° , and 81.5° were assigned to the well-crystallized silver nanoparticles with face-centered cubic structure (JCPDS 04–0783).



Fig. 3. XRD pattern of the prepared nanocomposite.

To investigate the catalytic ability of cellulose/Ag nanocomposite, a pilot experiment was carried out from the reaction of benzil, benzaldehyde and ammonium acetate to produce 2,4,5-triphenyl-1H-imidazole.

After several tests, we realized that the optimized conditions for the reaction is using 0.015 g of cellulose/Ag nanocomposite at 100 °C under solvent-free conditions. This reaction was performed with various benzaldehydes (Table1).

 Table 1. Synthesis of 2,4,5-trisubstituted-1*H*-imidazoles using cellulose/Ag nanocatalyst under solvent-free conditions.

$ \begin{array}{c} O \\ O \\ O \\ O \\ O \\ O \\ H \end{array} $ or $ \begin{array}{c} O \\ O $						
Entry	R	Time (Time (min)		Yield (%)	
		Benzil B	Benzil Benzoin		Benzil Benzoin	
1	phenyl	10	15	90	90	
2	3-nitro phenyl	8	12	92	89	
3	3-hydroxy phenyl	10	15	84	81	
4	4-chloro phenyl	15	18	88	87	
5	4-methyl phenyl	13	20	85	83	
6	4-methoxy phenyl	20	23	89	85	

Conclusions

In summary, we have introduced a new green approach to synthesis of cellulose/Ag nanocomposite and then we used the prepared nanocomposite as a green and efficient nanocatalyst in the synthesis of various 2,4,5-trisubstituted imidazole.

Acknowledgements

The authors gratefully acknowledge the partial support from the Research Council of the Iran University of Science and Technology.

References

- [1] Parak, W. J.; Nel, A. E.; Weiss, P. S. ACS Nano 2015, 9, 6637.
- [2] Maneerung, T.; Tokura, S.; Rujiravanit, R. Carbohydr. Polym. 2008, 72, 43.
- [3] Kong, H.; Jang, J. Langmuir 2008, 24, 2051.
- [4] Herzog, R. O. J. Phys. Chem. 1925, 30, 457.
- [5] Lin, S.; Cheng, Y.; Liu, J.; Wiesner, M. R. Langmuir 2012, 28, 4178.
- [6] Maleki, A.; Paydar, R. RSC Adv. 2015, 5, 33177.