chemistry proceedings

MDPI

Proceedings

Investigation of Acridinedione Derivatives Synthesis with Versatile Morphologies of Bi_2O_3 Nanoparticles †

Hossein Ghafuri *, Bahareh Pourakbari, Peyman Hanifehnejad and Zeinab Tajik

Catalysts and Organic Synthesis Research Laboratory, Department of Chemistry, Iran University of Science and Technology, Tehran 16846-13114, Iran; bahareh_pourakbari@chem.iust.ac.ir (B.P.); p_hanifehnejad@chem.iust.ac.ir (P.H.); z_tajik@chem.iust.ac.ir (Z.T.)

* Correspondence: ghafuri@iust.ac.ir

† Presented at the 23rd International Electronic Conference on Synthetic Organic Chemistry, 15 November–15 December 2020; Available online: https://ecsoc-24.sciforum.net/.

Published: date

Abstract: In this study, the Bi_2O_3 nanoparticles have been successfully synthesized through a facile hydrothermal procedure. The structure of the Bi_2O_3 nanoparticles was analyzed by Fourier transfer infrared spectroscopy (FTIR) and scanning electron microscopy (SEM). The synthesized Bi_2O_3 nanoparticles have unique properties such as high activity, high purity, and high surface area. Hence, we have reported the Bi_2O_3 nanoparticles as an efficient, cost-effective and mild catalyst for the synthesis of acridinedione derivatives via one-pot four-component reaction. Also, the effect of morphology of Bi_2O_3 nanostructure was investigated on catalytic performance. Therefore, Bi_2O_3 nanoparticles were prepared and applied as a heterogeneous catalyst in the synthesis of acridinedione derivatives. The present approach offers several advantages such as excellent yields within short times, green catalyst and ease of recovery.

Keywords: Bi_2O_3 nanoparticles; acridinedione derivatives; organic synthesis

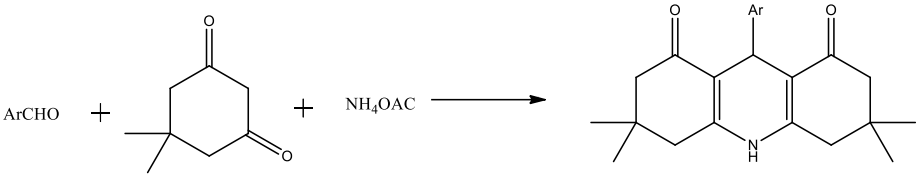
1. Introduction

Nowadays, for the synthesis of complex organic compounds use from multicomponent reactions that more than two components combine, and with high selectivity, the intended product will obtain. These reactions have attracted sizeable attention due to their many applications in various fields such as medicine, agriculture, and intermediates [1]. The different multicomponent reactions like Mannich, Biginelli, Strecker, Hantzsch, and acridinedione derivatives synthesis are significant organic transformation for the synthesis of pharmaceutical compounds [2]. Acridinedione compounds are a class of heterocyclic compounds due to their unique properties such as anticancer, antimicrobial, antibacterial, and their fluorescence properties, use in various fields, including pharmaceutical, biological, and laser dyes. Acridinedione derivatives have synthesized with different methods, which usually suffer from hazard solvents, expensive reagents, and high reaction time. Heterogeneous catalysts have a crucial role in determining the condition of reactions [3,4]. They are known as compounds or substances that speed up a chemical reaction without changing it. The advantages of heterogeneous catalysts are high activity, high surface area, long lifetime, thermal stability, selectivity, and non-toxicity [5].

As mentioned, the acridinedione derivatives are one of the attractive reactions in chemistry that have synthesized with various catalysts such as nano-ferrite, TiO_2 carbon nanotube, SiO_2, Rh (III), CTAB, and Amberlyst-15. Also, bismuth oxides have shown excellent catalytic properties. They have potential applications in solar cells, gas sensors, and piezoelectric-optical materials. The other attractive features of bismuth oxides included non-toxicity, ionic conductivity, high refractive index, and remarkable conductivity, making it possible to be used as an efficient catalyst to promote the

synthesis of acridinedione derivatives [6]. Besides these properties, the various morphologies of Bi_2O_3 nanoparticles, including nanowire, nanotube, nanoflake, nanofiber, and nanobelt, have successfully synthesized. The synthesis of Bismuth oxide nanoparticles has been reported with various protocols such as sol-gel, hydrothermal, and precipitated. These nanoparticles have extraordinary properties such as high activity, high purity, and high surface area [7].

In this research, we have reported the synthesis of Bi_2O_3 nanoparticles as a heterogeneous recyclable catalyst for the preparation of acridinedione derivatives through four multicomponent condensation reactions, as shown in Scheme 1.



Scheme 1. Synthesis of acridinedione derivatives catalyzed by Bi_2O_3 nanoparticles.

2. Experimental

2.1. General

All reagents were purchased from Fluka and Merck companies and used without further purification. Thin layer chromatography (TLC) was used for the purity determination of substrates, products and reaction monitoring over silica gel 60 F254 aluminum sheet. Melting points were measured in open capillary tubes with Electro thermal 9100 melting point apparatus. The FT-IR spectra were measured with a Shimadzu IR-100 spectrometer. MIRA3 TESCAN-XMU used for FE-SEM images.

2.2. Preaperation of Bi_2O_3 Nanoparticles:

2.2.1. Method (A)

20 mg Bi_2O_3 was added to 100 mL H_2O and raised PH to 11 using 1.0 M NaOH solution. While increasing the PH, a white powder has obtained. The powder was filtered and washed with water and acetone. After drying the powder in oven 50 °C, it has calcined at 400 °C with the rate of 10 °C /min and stayed for 30 min. After calcination, a yellow powder has obtained.

2.2.2. Method (B)

20 mg Bi_2O_3 was added to a solution of 40 mL H_2O and 40 mL ethanol and raised PH to 11 using 1.0 M NaOH solution. After a few hours in vigorous stirring, the solution poured into a Teflon-lined stainless autoclave and heated at 180 °C for 24 h. Then the products were filtered and washed with water and acetone and dried at 80 °C and a white powder has obtained.

2.3. General Procedure for the Preparation of Acridinediones Derivatives

1.0 mmol ammonium acetate, 1.0 mmol ethyl acetoacetate, 1.0 mmol dimedone, 1.0 mmol aromatic aldehyde, 3.0 mL ethanol as solvent, and 20.0 mg Bi_2O_3 nanoparticle as catalyst were mixed in a round bottom flask. They stirred under reflux condition for the appropriate time. After completing the reaction (monitored by TLC), the reaction mixture was filtered and recrystallized by ethanol to afford the pure product.

2.4. Spectral Data

3,3,6,6-tetramethyl-9-phenyl-3,4,6,7,9,10-hexahydroacridine-1,8(2H,5H)-dione (1a): mp: 274–276 °C, IR (KBr: ν/cm^{-1}): 3278, 3213, 2964, 1636, 1603, 1477, 1366, 1251, 1165 [8].

3,3,6,6-Tetramethyl-9-(4-chlorophenyl)-3,4,6,7,9,10-hexahydroacridine-1,8(2H,5H)-dione (2a): mp: 297–299 °C, IR (KBr: ν/cm^{-1}): 2976, 2902, 1648, 1606, 1490, 1366, 1220, 1149 [8].

3,3,6,6-Tetramethyl-9-(2,4-dimethoxyphenyl)-1,2,3,4,5,6,7,8,9,10-decahydroacridine-1,8-dione (3a): mp: 265–267 °C, IR (KBr: ν/cm^{-1}): 3190, 3065, 2954, 1636, 1602, 1480, 1395, 1363, 1292, 1261, 1217, 1144, 1124, 1041, 928, 825 [9].

9-(4-hydroxyphenol)-3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahydroacridine-1,8(2H,5H)-dione (4a): mp: 272–274 °C, IR (KBr: ν/cm^{-1}): 3273, 2963, 1645, 1394 [10].

3,3,6,6-Tetramethyl-9-(3-nitrophenyl)-3,4,6,7,9,10-hexahydroacridine-1,8(2H,5H)-dione(5a): mp: 296–298 °C, IR (KBr: ν/cm^{-1}): 3273, 3185, 3064, 2959, 1646, 1601, 1525, 1345 [11].

9-(4-nitrophenol)-3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahydroacridine-1,8(2H,5H)-dione(6a): mp: 272–274 °C, IR (KBr: ν/cm^{-1}): 3192, 2961, 1637, 1597 [10].

3. Results and Discussion

FE-SEM images and FT-IR spectrum have been used for the characterization of Bi_2O_3 nanoparticles. In Figure 1, the FT-IR spectrum in the range of 400–4000 cm^{-1} to investigate the chemical bonding of Bi_2O_3 nanoparticles is shown. 558 and 446 cm^{-1} peaks are related to Bi-O stretching vibration modes, 2364 cm^{-1} related to CO_2 of instrument, and the rest of FT-IR transmittance looks flat. The flat FT-IR transmittance is the evidence of the complete preparation of Bi_2O_3 nanoparticles [12].

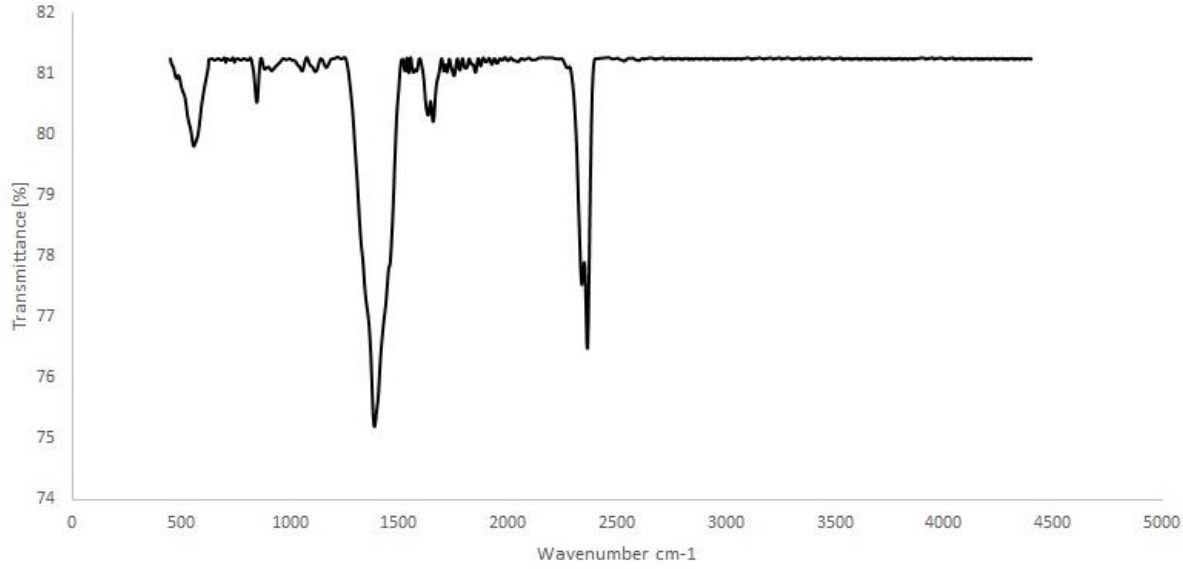
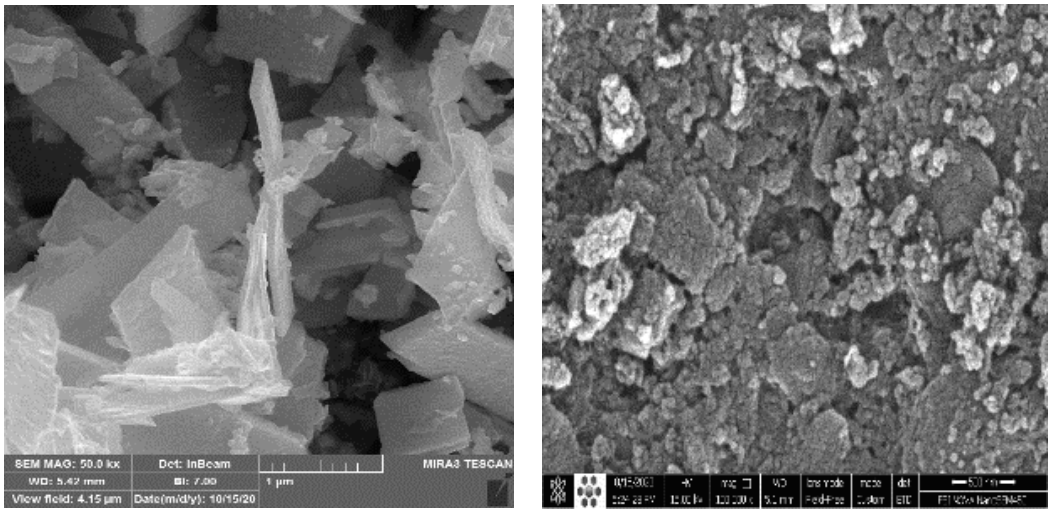


Figure 1. The FT-IR spectra of Bi_2O_3 nanoparticles.

The morphology of synthesized Bi_2O_3 nanoparticles has investigated using FE-SEM and shown in Figure 2.

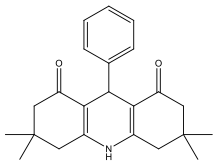
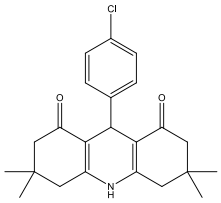
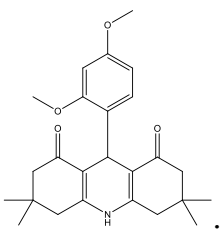
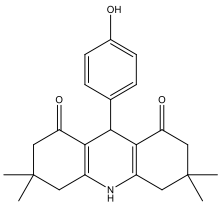


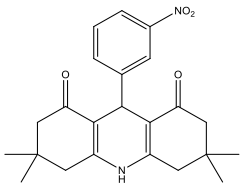
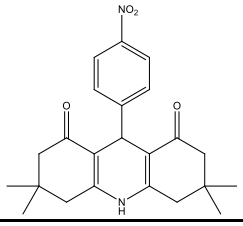
(**a**) (**b**)

Figure 2. The FE-SEM images of Bi_2O_3 nanoparticles. (**a**) Method A (1 µm) (**b**) Method B (500 nm).

We applied Bi_2O_3 nanoparticles as a catalyst in the synthesis of acridinedione derivatives through multicomponent reactions to indicate Bi_2O_3 nanoparticles' merits in organic synthesis. For that, we used six different aromatic aldehydes in optimum conditions, and the intended products have obtained in excellent yields. The results are shown in Table 1.

Table 1. Synthesis of acridinedione derivatives catalyzed by Bi_2O_3 nanoparticles [a].

Entry	Product	Time (min)	Yield (%) Method A Method B	Mp °C (Ref.)
1a		10	92 88	274–276 [13]
2a		12	86 80	297–299 [13]
3a		18	88 82	265–267 [9]
4a		15	90 82	272–274 [14]

5a		10	88 83	296–298 [13]
6a		8	90 85	272–274 [13]

As shown in Table 1, in comparison with method (B), the Bi_2O_3 nanoparticles synthesized by method (A) have more yield for acridinedione derivatives synthesis. This result indicates that the morphology of Bi_2O_3 nanoparticles is one of the important agents for catalytic activity.

4. Conclusions

We synthesized two different morphologies of Bi_2O_3 nanoparticles and used them as a catalyst in organic reactions, acridinedione derivative synthesis. Short reaction time, high yield, use of non-toxic solvent and mild condition reaction are the advantage of using Bi_2O_3 nanoparticles. Comparing results from two morphologies demonstrated that the catalyst's morphology is among the most critical catalytic activity agents.

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

Conflicts of Interest: The authors declare no conflict of interest.

References

1. Das, D. Multicomponent Reactions in Organic Synthesis Using Copper-Based Nanocatalysts *ChemistrySelect* **2016**, *1*, 1959–1980.
2. Domling, A.; Wang, W.; Wang, K. Chemistry and biology of multicomponent reactions. *Chem. Rev.* **2012**, *112*, 3083–3135.
3. Su, Q.; Li, P.; He, M.; Wu, Q.; Ye, L.; Mu, Y. Facile synthesis of acridine derivatives by ZnCl2-promoted intramolecular cyclization of o-arylaminophenyl Schiff bases. *Organic Lett.* **2014**, *16*, 18–21.
4. Gensicka-Kowalewska, M.; Cholewiński, G.; Dzierzbicka, K. Recent developments in the synthesis and biological activity of acridine/acridone analogues. *RSC Adv.* **2017**, *7*, 15776–15804.
5. Ramos, L.P.; Cordeiro, C.S.; Cesar-Oliveira, M.A.F.; Wypych, F.; Nakagaki, S. Applications of heterogeneous catalysts in the production of biodiesel by esterification and transesterification. In *Bioenergy Research: Advances and Applications*; Elsevier: Amsterdam, The Netherlands, 2014; pp. 255–276.
6. Li, W. Facile synthesis of monodisperse Bi2O3 nanoparticles. *Mater. Chem. Phys.* **2006**, *99*, 174–180.
7. Zhang, X.B.; Zhang, L.; Hu, J.S.; Huang, X.H. Facile hydrothermal synthesis and improved photocatalytic activities of Zn 2+ doped Bi 2 MoO 6 nanosheets. *RSC Adv.* **2016**, *6*, 32349–32357.
8. Eyvazzadeh-Keihan, R.; Bahrami, N.; Taheri-Ledari, R.; Maleki, A. Highly facilitated synthesis of phenyl (tetramethyl) acridinedione pharmaceuticals by a magnetized nanoscale catalytic system, constructed of GO, Fe3O4 and creatine. *Diam. Relat. Mater.* **2020**, *102*, 107661.
9. Shi, D.Q.; Ni, S.N.; Shi, J.W.; Dou, G.L.; Li, X.Y.; Wang, X.S. An efficient synthesis of polyhydroacridine derivatives by the three-component reaction of aldehydes, amines and dimedone in ionic liquid. *J. Heterocycl. Chem.* **2008**, *45*, 653–660.
10. Chavan, P.N.; Pansare, D.N.; Shelke, R.N. Eco-friendly, ultrasound-assisted, and facile synthesis of one-pot multicomponent reaction of acridine-1, 8 (2H, 5H)-diones in an aqueous solvent. *J. Chin. Chem. Soc.* **2019**, *66*, 822–828.

11. Javid, A.; Khojastehnezhad, A.; Heravi, M.; Bamoharram, F.F. Silica-supported preyssler nanoparticles catalyzed simple and efficient one-pot synthesis of 1, 8-dioxodecahydroacridines in aqueous media. *Synth. React. Inorg. Metal-Org. Nano-Met. Chem.* **2012**, *42*, 14–17.
12. Abu-Dief, A.M.; Mohamed, W. α-Bi2O3 nanorods: Synthesis, characterization and UV-photocatalytic. Activity. *Mater. Res. Express* **2017**, *4*, 035039.
13. Zeynizadeh, B.; Gilanizadeh, M. Microwave-promoted three-component Hantzsch synthesis of acridinediones under green conditions. *Curr. Chem. Lett.* **2020**, *9*, 71–78.
14. Patil, D.; Chandam, D.; Mulik, A.; Patil, P.; Jagadale, S.; Kant, R.; Gupta, V.; Deshmukh, M. Novel Brønsted acidic ionic liquid ([CMIM][CF 3 COO]) prompted multicomponent hantzsch reaction for the eco-friendly synthesis of acridinediones: An efficient and recyclable catalyst. *Catal. Lett.* **2014**, *144*, 949–958.