

Proceeding Paper

# Investigation of Acridinedione Derivative Synthesis with Versatile Morphologies of Bi<sub>2</sub>O<sub>3</sub> Nanoparticles †

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**Abstract:** In this study, Bi<sub>2</sub>O<sub>3</sub> nanoparticles were successfully synthesized through a facile hydrothermal procedure. The structure of the Bi<sub>2</sub>O<sub>3</sub> nanoparticles was analyzed by Fourier transfer infrared spectroscopy (FTIR) and scanning electron microscopy (SEM). The synthesized Bi<sub>2</sub>O<sub>3</sub> nanoparticles have unique properties, such as high activity, high purity, and high surface area. Hence, we have reported the Bi<sub>2</sub>O<sub>3</sub> nanoparticles as an efficient, cost-effective, and mild catalyst for the synthesis of acridinedione derivatives via a one-pot four-component reaction. In addition, the effect of the morphology of the Bi<sub>2</sub>O<sub>3</sub> nanostructure was investigated on catalytic performance. Therefore, Bi<sub>2</sub>O<sub>3</sub> 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<sub>2</sub>O<sub>3</sub> nanoparticles; acridinedione derivatives; organic synthesis

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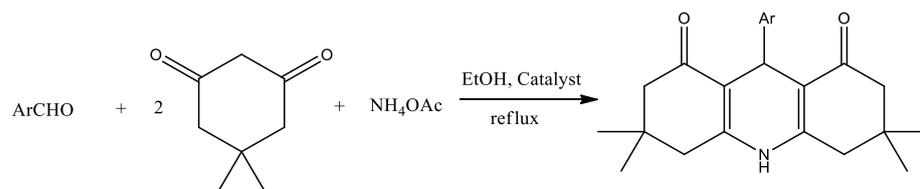
## 1. Introduction

Nowadays, scientists are using multicomponent reactions to synthesis complex organic compounds due to their high efficiency. 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 derivative synthesis, are significant organic transformations 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, their use in various fields, including pharmaceutical, biological, and laser dyes. Different methods have been used to synthesize acridinedione derivatives, which usually suffer from hazard solvents, expensive reagents, and high reaction times. Heterogeneous catalysts have a crucial role in determining the conditions 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 lifetimes, thermal stability, selectivity, and non-toxicity [5].

As mentioned, the acridinedione derivatives are one of the attractive reactions in chemistry that have been synthesized with various catalysts, such as nano-ferrite, TiO<sub>2</sub> carbon nanotube, SiO<sub>2</sub>, Rh (III), and Amberlyst-15. In addition, 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, a high refractive index, and remarkable conductivity, making it possible for them to be used as efficient catalysts to promote the

synthesis of acridinedione derivatives [6]. Besides these properties, the various morphologies of  $\text{Bi}_2\text{O}_3$  nanoparticles, including nanowire, nanotube, nanoflake, nanofiber, and nanobelt, have been 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 paper, we report the synthesis of  $\text{Bi}_2\text{O}_3$  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  $\text{Bi}_2\text{O}_3$  nanoparticles.

## 2. Experimental

### 2.1. General

All reagents were purchased from the 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 a silica gel 60 F254 aluminum sheet. Melting points were measured in open capillary tubes with an Electrothermal 9100 melting point apparatus. The FTIR spectra were measured with a Shimadzu IR-100 spectrometer. A MIRA3 TESCAN-XMU was used for FE-SEM images.

### 2.2. Preparation of $\text{Bi}_2\text{O}_3$ Nanoparticles:

#### 2.2.1. Method (A)

We added 20 mg of  $\text{Bi}_2\text{O}_3$  to 100 mL of  $\text{H}_2\text{O}$  and raised the pH to 11 using a 1.0 M NaOH solution. While increasing the pH, a white powder was obtained. The powder was filtered and washed with water and acetone. After drying the powder in an oven at 50 °C, it was calcined at 400 °C at the rate of 10 °C/min and remained there for 30 min. After calcination, a yellow powder was obtained.

#### 2.2.2. Method (B)

We added 20 mg of  $\text{Bi}_2\text{O}_3$  to a solution of 40 mL of  $\text{H}_2\text{O}$  and 40 mL of ethanol and raised the pH to 11 using a 1.0 M NaOH solution. After a few hours of vigorous stirring, the solution was 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 then dried at 80 °C and a white powder was obtained.

### 2.3. General Procedure for the Preparation of Acridinediones Derivatives

We mixed 1.0 mmol of ammonium acetate, 2.0 mmol of dimedone, 1.0 mmol of aromatic aldehyde, 3.0 mL of ethanol as a solvent, and 20.0 mg of  $\text{Bi}_2\text{O}_3$  nanoparticle as a catalyst in a round bottom flask. They were stirred in reflux conditions for an 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:  $\nu/\text{cm}^{-1}$ ): 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:  $\nu/\text{cm}^{-1}$ ): 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:  $\nu/\text{cm}^{-1}$ ): 3190, 3065, 2954, 1636, 1602, 1480, 1395, 1363, 1292, 1261, 1217, 1144, 1124, 1041, 928, 825 [9].

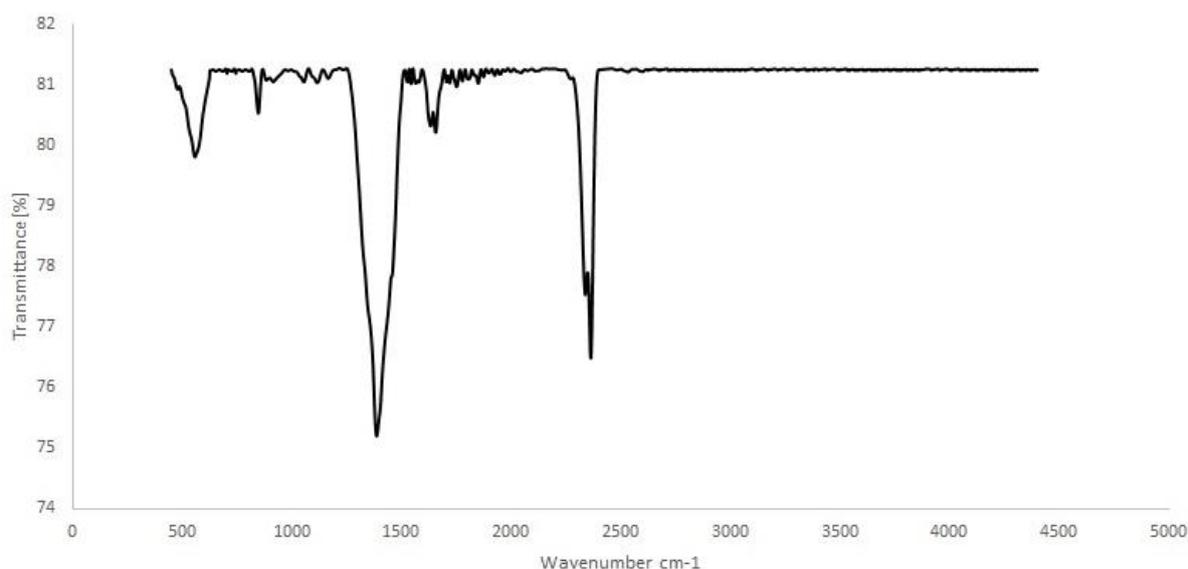
9-(4-hydroxyphenyl)-3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahydroacridine-1,8(2H,5H)-dione (**4a**): mp: 272–274 °C, IR (KBr:  $\nu/\text{cm}^{-1}$ ): 3273, 2963, 1645, 1394 [10].

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:  $\nu/\text{cm}^{-1}$ ): 3273, 3185, 3064, 2959, 1646, 1601, 1525, 1345 [11].

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

### 3. Results and Discussion

FE-SEM images and FTIR spectrum were used for the characterization of the  $\text{Bi}_2\text{O}_3$  nanoparticles. In Figure 1, the FTIR spectrum, in the range of 400–4000  $\text{cm}^{-1}$  to investigate the chemical bonding of the  $\text{Bi}_2\text{O}_3$  nanoparticles, is shown. The 558 and 446  $\text{cm}^{-1}$  peaks are related to Bi-O stretching vibration modes, the 2364  $\text{cm}^{-1}$  peak is related to the  $\text{CO}_2$  of the instrument, and the rest of the FTIR transmittance looks flat. The flat FTIR transmittance is evidence of the complete preparation of the  $\text{Bi}_2\text{O}_3$  nanoparticles [12].

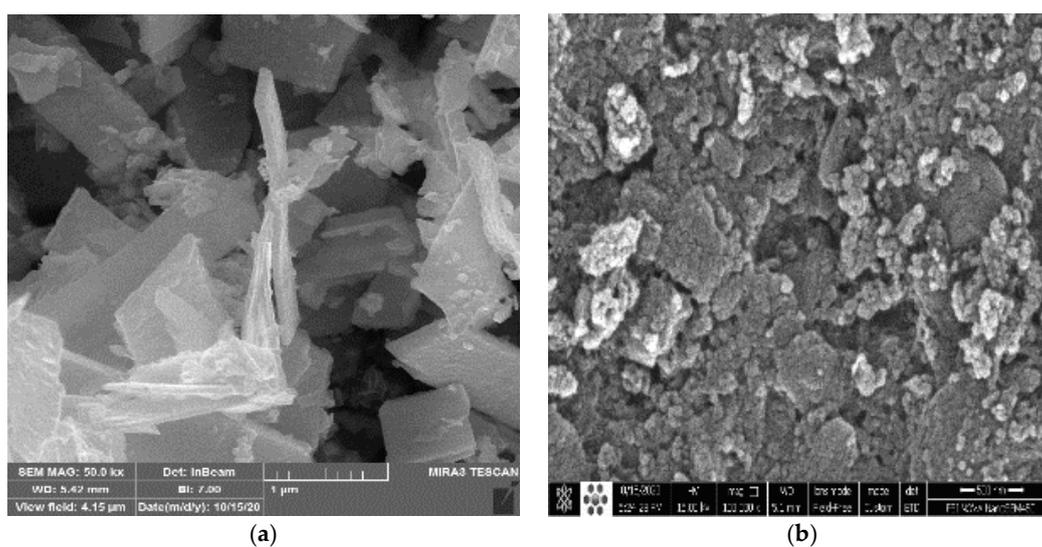


**Figure 1.** The FTIR spectra of  $\text{Bi}_2\text{O}_3$  nanoparticles.

The morphology of the synthesized  $\text{Bi}_2\text{O}_3$  nanoparticles was investigated using FE-SEM and is shown in Figure 2.

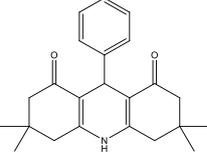
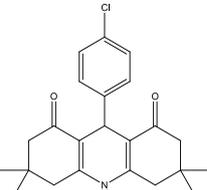
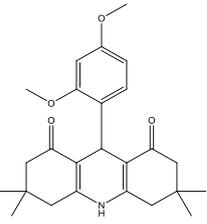
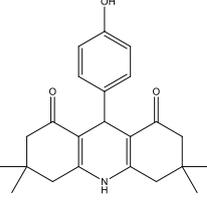
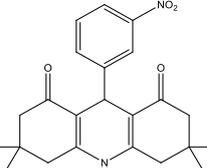
We applied  $\text{Bi}_2\text{O}_3$  nanoparticles as a catalyst in the synthesis of acridinedione derivatives through multicomponent reactions to indicate the  $\text{Bi}_2\text{O}_3$  nanoparticles' merits in organic synthesis. For this, we used six different aromatic aldehydes in optimum conditions, and the intended products were obtained in excellent yields. The results are shown in Table 1.

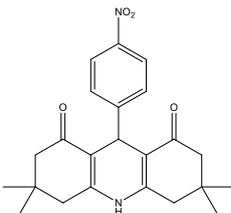
As shown in Table 1, in comparison with method (B), the  $\text{Bi}_2\text{O}_3$  nanoparticles synthesized by method (A) have more yield for acridinedione derivative synthesis. This result indicates that the morphology of  $\text{Bi}_2\text{O}_3$  nanoparticles is one of the important agents for catalytic activity.



**Figure 2.** The FE-SEM images of  $\text{Bi}_2\text{O}_3$  nanoparticles. (a) Method A (1  $\mu\text{m}$ ) (b) Method B (500 nm).

**Table 1.** Synthesis of acridinedione derivatives catalyzed by  $\text{Bi}_2\text{O}_3$  nanoparticles.

Entry	Product	Time (min)	Yield (%)		Mp °C (Ref.)
			Method A	Method B	
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]
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#### 4. Conclusions

We synthesized two different morphologies of Bi<sub>2</sub>O<sub>3</sub> nanoparticles and used them as a catalyst in organic reactions and acridinedione derivative synthesis. A short reaction time, high yield, use of non-toxic solvent, and a mild condition reaction are the advantages of using Bi<sub>2</sub>O<sub>3</sub> nanoparticles. The comparison of the results from the two morphologies demonstrates that the catalyst's morphology is among the most critical catalytic activity agents.

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

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