Green Synthesis of ZnO Nanoparticles via Sol-gel Method and Investigation of Its Application in Solvent-free Synthesis of 12-Aryl-tetrahydrobenzo[a]xanthene-11-one Derivatives Under Microwave Irradiation

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ABSTRACT
In this work, zinc oxide (ZnO) nanoparticles were fabricated using Arabic gum as a reducing and stabilizing agent by the novel sol-gel method without adding any surfactants. The synthesized nanoparticles were characterized by Fourier transform infrared spectroscopy (FTIR), powder X-ray diffraction (XRD), and scanning electron microscopy (SEM). Subsequently, ZnO nanoparticles as efficient catalysts were consumed for the three-component coupling of 2-naphthol, aldehydes, and dimedone under microwave irradiation and solvent-free conditions in order to furnish the corresponding synthesis of 12-aryl-tetrahydrobenzo[a]xanthene-11-one derivatives in high yields.
Introduction

Recently, literature has highlighted the importance of nanosized materials in several scientific and technological areas, and many research organizations have intensified investments in nanotechnology for the coming years [1-4]. Moreover, the development of efficient green-chemistry procedures for the synthesis of metal nanoparticles has become a major focus of researchers. They have perused to find an eco-friendly method for the production of well-characterized nanoparticles [5-9]. One of the most considered methods is the production of metal nanoparticles using organisms [10-15]. Between these organisms, plants seem to be the excellent candidates as they are appropriate for large-scale biosynthesis of nanoparticles [16-18]. These approaches have many benefits over chemical, physical, and microbial synthesis since there is no demand for the elaborated process of culturing and maintaining the cell, using hazardous chemicals, high-energy and wasteful purifications [19-23]. Nowadays, the oxides of transition metals represent an important class of semiconductors, which have applications in catalysis, gas sensors, electronics, solar energy transformation, etc. [24-26]. Among all the metal oxides, ZnO is of great interest due to its wide band gap (3.37 eV), non-toxicity, high photosensitivity physical chemical stability, and large excitation binding energy (60 m eV). It is worth mentioning that zinc oxide (ZnO) is an inorganic semiconductor with a hexagonal Wurtzite crystal structure [27]. The ZnO NPs have numerous applications such as catalysis [28], piezoelectric devices [29], photocatalytic [30, 31], chemical sensors [32] and cosmetic material especially for transparent UV protection [33]. Lately, multicomponent reactions (MCRs) have concerned notable attention due to extensive benefits such as simplicity of operation, reduction of purification steps and isolation, and decreases of time, expenses and waste production [34-38]. MCRs are especially beneficial in preparing expedient approaches to a broad range of biological and pharmaceutical compounds [39-43]. The utilization of microwave energy for conducting organic reactions at
widely accelerated rates is an emerging technique. In recent years, microwaves have become significantly popular among synthetic organic chemists due to the better shortening reaction times, classical organic reactions, and improving yields, and because of the fact that it promotes new reactions [44-46]. The derivatives of xanthene are the most important heterocycles that are known to possess multiple activities such as antiviral [47], antiplasmodial and anti-inflammatory [48]. In this work, the synthesized ZnO nanoparticles using Arabic gum (AG) by the sol-gel method, as an efficient catalyst, were consumed for the three-component coupling of 2-naphthol, aldehydes, and dimedone under microwave irradiation and solvent-free conditions to furnish the corresponding synthesis of 12-aryl-tetrahydrobenzo[α]xanthene-11-one derivatives in excellent yields (Scheme 1).

![Scheme 1. Synthesis of 12-aryl-tetrahydrobenzo[α]xanthene-11-one](image)

**Experimental**

**General**

Starting materials were obtained from Fluka (Switzerland) and Merck (Germany) and were used without further purification. The Arabic gum (AG) was obtained from a local health food store. Melting points were measured on an Electrothermal 9100 apparatus and are uncorrected. The microwave-assisted procedures were carried out in a milestone microwave oven operating at 1600 W. All obtained products are known compounds and are identified by comparing their melting points with those reported in the literature. The progress of the reactions was monitored by thin layer chromatography (TLC). $^1$H (DMSO-d$_6$) and $^{13}$C NMR (DMSO-d$_6$) spectra were recorded on a Bruker DRX-250 avance spectrometer at 250.13 and 62.90 MHz, respectively. The IR spectra were measured on a Jasco 6300 FT-IR spectrometer (KBr disks). The structural properties of synthesized nanoparticles were investigated by X-ray powder diffraction (XRD) pattern on a X’Pert-PRO advanced diffractometer using Cu (Kα) radiation the (wavelength: 1.5406 Å) at 40 kV and 40 mA at room temperature in the range of 2θ from 20 to 80°. The particle size and morphology of the surfaces of the sample were analyzed by a scanning electron microscopy (LEO Co., England, model: 1455VP).
Preparation of ZnO nanoparticles using Arabic gum

In a generic synthesis, 0.3 g of the Arabic gum (AG) was dissolved in 40 mL of deionized water and stirred for 120 min at 75 °C to attain a clear Arabic gel (AG) solution. Then, 2 g of Zn(NO$_3$)$_2$.6H$_2$O was added to the AG solution, and the container was placed in a sand bath. The temperature of the sand bath was fixed at 75 °C and stirring was continued for 12 h to obtain a brown color resin. The ultimate product was calcined at 500 °C temperatures in air for 4 h to obtain a white powder of ZnO.

General procedure for the synthesis of 12-aryl-8,9,10,12-tetrahydrobenzo[α]xanthen-11-one derivative

A mixture of benzaldehyde (1 mmol), 2-naphthol (1 mmol), dimeredone (1 mmol), and ZnO (0.01 g) under solvent-free condition was placed for 15-18 min with the power of 400 W in the microwave. The progress of the reaction was checked by TLC (petroleum ether: EtOAc, 10:2). After the completion of the reaction which was followed by TLC, hot ethanol was added and the catalyst was separated by centrifugation. Then, the residue was poured into crushed ice and stirred for a few minutes. The solid product was filtered and the pure product was obtained by recrystallization from hot ethanol.

The selected spectral data

9,9-Dimethyl-12-phenyl-8,9,10,12-tetrahydro-benzo[α]xanthene-11-one (4a): White solid, yield, 92%, $^1$H NMR (250 MHz, DMSO-d$_6$), $\delta$: 8.00 (d, $J$=7.75 Hz, 1H), 7.85 (d, $J$=8.25 Hz, 2H), 7.43—6.99 (m, 8H), 5.57 (s, 1H), 2.54 (s, 2H), 2.08 and 2.27 (AB system, $J$=16.2 Hz, 2H), 0.96 (s, 3H), 0.82 (s, 3H). $^{13}$C NMR (62.90 MHz, DMSO-d$_6$): $\delta$: 196.23, 164.15, 147.64, 145.31, 131.53, 131.12, 129.49, 128.96, 128.56, 127.52, 126.61, 125.36, 123.68, 117.73, 117.56, 113.70, 50.57, 34.59, 32.23, 29.25, 26.62.

Results and discussion

Catalyst characterization

Figure 1. displays the FTIR spectra of the catalyst in the range of 4000–400 cm$^{-1}$. The broad band at 3416 cm$^{-1}$ is the stretching vibration of O–H group. The peak at 1599 cm$^{-1}$ is due to the O–H bending of water. The peak at 541 cm$^{-1}$ is allotted to Zn–O [49] which is clearly represented in the Figure.
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Figure 1. FT-IR spectrum of ZnO NPs

Figure 2. XRD pattern of ZnO NPs

Figure 2. shows the XRD pattern of the synthesized ZnO NPs. The very strong diffraction lines have been observed in the XRD pattern that are indexed as hexagonal phase of ZnO (Wurtzite structure) and lattice constants a=b=3.258 Å and c=5.205 Å based on the JCPDS card No: 89-7102. The 2θ characteristic lines around at 31.28°, 33.88°, 35.71°, 46.98°, 56.08°, 62.36°, 65.89°, 67.44°, 68.69°, 72.02° and 76.38° were assigned to (100), (002), (101), (102), (110), (103), (200), (112), (201), (004) and (202) crystal planes of hexagonal ZnO, respectively. In addition, the average crystallite size of ZnO NPs was calculated using the Debye–Scherrer formula [50]: D=0.9λ/βcos θ in this regard, D, λ, θ and β are the crystallite size, wavelength of the X-ray beam, diffraction angle and full width half maximum (FWHM) of the peak, respectively. The calculated crystallite size was found to be 10 nm.
The SEM image shows the particle size and external morphology of the ZnO nanoparticles that calcined at 500 °C for 4 h (Figure 3). It can be seen from the SEM image that the ZnO nanoparticles have a uniform spherical morphology and narrow size distributions.

![Figure 3. SEM micrograph of the ZnO NPs](image)

The synthesized ZnO NPs were studied as catalysts in the synthesis of 12-aryl-tetrahydrobenzo[α]xanthene-11-one derivatives under microwave irradiation. The reaction conditions such as catalyst dosage, microwave power, and reaction times were optimized. First, the efficiency and amount of the ZnO catalyst were investigated in the model reaction of benzaldehyde 1a (1 mmol) 2-naphthol 2 (1 mmol) and dimedone 3 (1 mmol) for the synthesis of compound 4a (Table 3, entry 1). As shown in Table 1, the optimum yield of the product was obtained when 10 mg of ZnO was used (Table 1, entry 1).

| Entry | Catalyst (mg) | Microwave power (W) | Time (min) | Yield (%) |
|-------|---------------|---------------------|------------|-----------|
| 1     | 5             | 400                 | 15         | 74        |
| 2     | 10            | 400                 | 15         | 92        |
| 3     | 15            | 400                 | 15         | 92        |

**Table 1. Effect of the catalyst dosage on the synthesis of 4a**

Reactions conditions: benzaldehyde (1mmol), 2-naphthol (1mmol) and dimedone (1 mmol).

The effect of microwave power inputs from 300 to 450 W on the synthesis of compound 4a (Table 3, entry 1) as the model reaction was investigated (Table 2). The reaction yield improved with the microwave power at 400 W in comparison to 300 and 350 W.

| Entry | Catalyst (mg) | Microwave power (W) | Time (min) | Yield (%) |
|-------|---------------|---------------------|------------|-----------|
| 1     | 10            | 300                 | 15         | 76        |
| 2     | 10            | 350                 | 15         | 86        |
| 3     | 10            | 400                 | 15         | 92        |
| 4     | 10            | 450                 | 15         | 92        |

**Table 2. Effect of microwave power on the synthesis of 4a**

Reactions conditions: benzaldehyde (1 mmol), 2-naphthol (1 mmol), dimedone (1 mmol).

aIsolated yields
Using this optimized condition, a varied type of aldehydes, dimedone and 2-naphthol were subjected to undergo three-component condensation in the presence of ZnO under solvent-free conditions (Table 3). Then, various aromatic aldehydes carrying electron-withdrawing and electron-donating groups on the aromatic ring in the ortho, meta, and para positions and heterocyclic aldehydes were evaluated. Yields of all reactions were good to excellent. Comparison of catalytic ability of catalysts reported in the literature for the synthesis of 12-aryl-tetrahydrobenzo[α]xanthene-11-one derivatives is demonstrated in Table 4. A plausible mechanism for the formation of products (4a–m) in the presence of ZnO NPs is suggested in Scheme 2.

**Table 3.** ZnO catalyzed the synthesis of 12-aryl-tetrahydrobenzo[α]xanthene-11-one derivatives

| Entry | Aldehydes | Product | Time (min) | Yield (%) | M.P. (°C) | M.P. (°C) [Ref.] |
|-------|-----------|---------|------------|-----------|-----------|-----------------|
| 1     | PhCHO     | 4a      | 15         | 92        | 148-150   | 149-150 [51]    |
| 2     | 4-ClC₆H₄CHO | 4b     | 15         | 93        | 186-187   | 187-188 [52]    |
| 3     | 2-ClC₆H₄CHO | 4c     | 16         | 90        | 178-179   | 179-180 [53]    |
| 4     | 2,4-Cl₂C₆H₄CHO | 4d     | 16         | 91        | 184-186   | 183-184 [54]    |
| 5     | 4-MeC₆H₄CHO | 4e     | 18         | 88        | 174-176   | 175-176 [51]    |
| 6     | 4-OC₆H₄CHO | 4f     | 18         | 86        | 151-152   | 150-151 [54]    |
| 7     | 2-NO₂C₆H₄CHO | 4g     | 15         | 88        | 169-170   | 168-170 [53]    |
| 8     | 3-NO₂C₆H₄CHO | 4h     | 15         | 89        | 175-177   | 175-176 [55]    |
| 9     | 4-NO₂C₆H₄CHO | 4i     | 15         | 92        | 174-176   | 175-176 [51]    |
| 10    | 4-BrC₆H₄CHO | 4j     | 17         | 89        | 183-185   | 184-186 [56]    |
| 11    | 4-MeOC₆H₄CHO | 4k     | 18         | 88        | 207-208   | 208-209 [52]    |
| 12    | 4-FC₆H₄CHO | 4l     | 15         | 94        | 185-187   | 185-186 [57]    |
| 13    | 4-CN₆H₄CHO | 4m     | 15         | 93        | 168-170   | 168-169 [58]    |

Reaction conditions: aldehyde 4a–m (1 mmol), 2-naphthol (1 mmol), dimedone (1 mmol) and 10 mg of the catalyst under microwave irradiation (400 W) in solvent-free conditions.

*Isolated yields

**Table 4.** Comparison of catalytic ability of ZnO NPs with other catalysts

| Entry | Catalyst | Conditions | Time (min) | Yield (%) | [Ref.] |
|-------|----------|------------|------------|-----------|--------|
| 1     | Fe₂O₄@SiO₂-SO₃H | Solvent-free, 110 °C | 30         | 95        | [59]   |
| 2     | DSIMHS   | Solvent-free, 55 °C | 20         | 93        | [56]   |
| 3     | CAN      | Microwave irradiation, 120 °C | 120       | 85        | [60]   |
| 4     | NaHSO₄/SiO₂ | CH₂Cl₂, Reflux | 300        | 91        | [52]   |
| 5     | Sr(OTf)₂ | 1,2-Dichloroethane, 80 °C | 300       | 85        | [61]   |
| 6     | NO₂FePc/C | EtOH, Reflux | 30         | 91        | [62]   |
| 7     | ZnO      | Solvent-free, 50 °C | 45         | 70        | [58]   |
| 8     | ZnO      | CH₂Cl₂, Reflux | 240        | 35        | [58]   |
| 9     | ZnO      | Solvent-free, Microwave irradiation | 15         | 92        | This work |

Based on the three-component reaction of aldehyde (1 mmol), 2-naphthol (1 mmol), dimedone (1 mmol)
**Conclusions**

In conclusions, we have described an efficient and environmentally friendly synthesis of ZnO nanoparticles that was accomplished by the sol-gel method in Arabic gum (AG) as a reducing and stabilizing agent. This efficient procedure has many advantages such as economic viability, nontoxic, ease of scale up, environmentally friendly and less time consuming approach for the synthesis of ZnO nanoparticles without using any harmful chemicals. Then, the synthesized ZnO nanoparticles as an impressive catalyst were used for the Three- component coupling of 2-naphthol, aldehydes, and dimedone under microwave irradiation and solvent-free conditions to provide the corresponding synthesis of 12-aryl-8,9,10,12-tetrahydrobenzo[α]xanthen-11-one derivatives in excellent yields. Simple procedure, mild reaction conditions, short reaction times, high yields of products and ease of separation are the main features of the protocol presented here.

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