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ORIGINAL STUDY

Facile Synthesis of PEG- and Oleic Acid-modified ZnO Nanoparticles for Potential Skin UV-protection Applications

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Abstract

Zinc oxide (ZnO) has garnered significant attention due to its wide-ranging applications. ZnO's wide direct bandgap (3.37 eV) and excellent UV-shielding properties make it ideal for cosmetic applications as a UV-shielding material. However, the high photocatalytic activity of ZnO is undesirable for cosmetic applications, as it promotes the generation of reactive hydroxyl radicals that are harmful to human skin. In addition, direct contact of the photoactive particles with the skin can further aggravate these catalytic effects, thereby necessitating the use of encapsulation or surface modification strategies. Driven by this gap, this study explores the synthesis and surface modification of ZnO nanoparticles using biocompatible agents, polyethylene glycol (PEG) and oleic acid (OA) and assess its effect on the photoactivity of ZnO. ZnO and modified ZnO particles were synthesized via a simple chemical precipitation method, using $Zn(CH_3COO)_2$ and NaOH as precursors. XRD analysis confirmed the high purity of the hexagonal wurtzite ZnO phase. FTIR and TGA analyses indicated successful surface modification, showing distinct functional groups and weight loss profiles attributed to PEG and OA. SEM images revealed that the modification increased nanoparticle size, attributed to surface coating by the polymer modifiers. UV—Vis spectra showed a slight shift in absorption and bandgap values, with OA-modified ZnO exhibiting the highest bandgap (3.62 eV), making it suitable for potential skin UV-protection applications.

Keywords: ZnO, Precipitation method, PEG-ZnO, OA-ZnO, UV-protection

1. Introduction

Zinc oxide (ZnO) is a widely studied metal oxide due to its diverse range of applications, spanning from photonic devices such as sensors and LEDs [1] to biomedical products like sunscreens and antibacterial materials [2—4]. It possesses a wide direct bandgap of 3.37 eV at room temperature, excellent photocatalytic properties [5], and exceptional UV-shielding activity [6]. However, in sunscreen and cosmetic applications, while effective UV absorption is critical, high photocatalytic activity is undesirable. This property can initiate light-assisted photocatalytic reactions, leading to the formation of hydroxyl radicals ($OH\cdot$) or superoxide radical anions ($O_2\cdot^-$), which are highly reactive species that pose threats to human skin [7—9]. Moreover, the photoactivity of these semiconducting UV filters can degrade other organic components in the sunscreen, rendering the product unstable [8,9].

The photocatalytic activity of ZnO is significantly influenced by its morphology and surface properties. Recent efforts to address this issue include coating

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ZnO with metal-phenolic networks [10], two-dimensional morphological engineering [8], and compositing with other metal oxides such as CeO_2 [11] and Fe_3O_4 [12]. These strategies have successfully reduced the photocatalytic activity of semiconducting UV-blockers while maintaining or even enhancing their UV absorption capabilities, thereby making them more suitable for sunscreen applications. However, the global sunscreen market still heavily relies on existing inorganic metal oxide UV blockers like ZnO due to current regulatory standards [9]. Hence, it is crucial to continue developing cost-effective and efficient enhancements for these materials.

One promising approach for improving the properties of ZnO in sunscreen and related applications is surface modification. Coating or encapsulating UV-blocking nanoparticles effectively isolates the material, preventing direct interaction between free radicals generated during UV exposure and skin cells, including those in deeper layers [9]. This strategy not only mitigates photoactivity issues but also preserves the functionality of ZnO as a UV filter. For example, silane coatings have been employed to improve the compatibility and stability of ZnO in organic matrices [13,14]. Additionally, the use of additives such as polymers and surfactants has been shown to enhance the dispersibility of ZnO nanoparticles. Oleic acid (OA), an anionic surfactant, stabilizes nanoparticles by forming strong chemical bonds between its carboxylic acid groups and the surface of amorphous metal oxide particles [15,16]. On the other hand, polyethylene glycol (PEG), widely used in cosmetics and pharmaceuticals, acts as a capping agent [7]. Both OA and PEG are therefore excellent candidates that enhance the performance of ZnO nanoparticles in sunscreen formulations.

Various methods can be used to synthesize ZnO nanoparticles, including spray pyrolysis [17], thermal degradation [18], hydrothermal synthesis [19], and chemical precipitation [20,21]. Among these, chemical precipitation is most advantageous for its simplicity and ability to produce well-dispersed ZnO nanoparticles with high yields desirable for large scale production. In this study, ZnO and surface modified-ZnO nanoparticles were synthesized via a facile chemical precipitation method using biocompatible agents-polyethylene glycol (PEG) and oleic acid (OA)- and assessed its effects on the photoactivity of ZnO for potential skin UV-protection applications.

2. Material and methods

For the synthesis of ZnO, a chemical precipitation method was adapted from previous reports [21,22], with slight modifications. In a typical experiment, a 50 mL solution of 0.2 M zinc acetate [$Zn(CH_3COO)_2$, Techno Pharmchem] was prepared. Sodium hydroxide (1.0 M NaOH, Macron Fine Chemicals) was then slowly added dropwise to this solution until the pH reached 12. The mixture was heated at 80 °C for 2 h, resulting in the formation of a white slurry [23]. The precipitate was collected via centrifugation at 2500 rpm for 10 min, washed with deionized water, and subsequently air-dried for 3 h before annealing at 300 °C for 1 h [21,24]. For surface-modified ZnO samples, 5.0 mL of polyethylene glycol (PEG 400 $H(OCH_2CH_2)_nOH$, Alfa Aesar) or oleic acid (OA, $C_{18}H_{34}O_2$, UNILAB) was introduced into the $Zn(CH_3COO)_2$ solution prior to pH adjustment under constant stirring. In these cases, the precipitates were dried at 60 °C for 2 h to remove the solvent.

The phase composition and crystal structure of the annealed product were determined by X-ray diffraction (XRD, Shimadzu XRD-7000) to determine crystalline phases formed. Surface morphology was examined using scanning electron microscopy (SEM, Hitachi S-3400 N) to observe particle size and morphology. The incorporation of organic functional groups from surface modification was verified through Fourier Transform Infrared Spectroscopy (FTIR, Shimadzu Prestige). Thermogravimetric analysis (TGA, Q500 V20.13) was conducted to assess the thermal stability and decomposition behavior of the samples at a heating rate of 5 °C/min, as well as to quantify the amount of polymer incorporated into the synthesized material. Finally, the optical properties of the synthesized samples were analyzed using UV—Vis spectroscopy to assess its performance in UV-protection applications [24,25].

3. Results and siscussion

Fig. 1 presents the XRD pattern of the annealed ZnO nanoparticles synthesized via the chemical

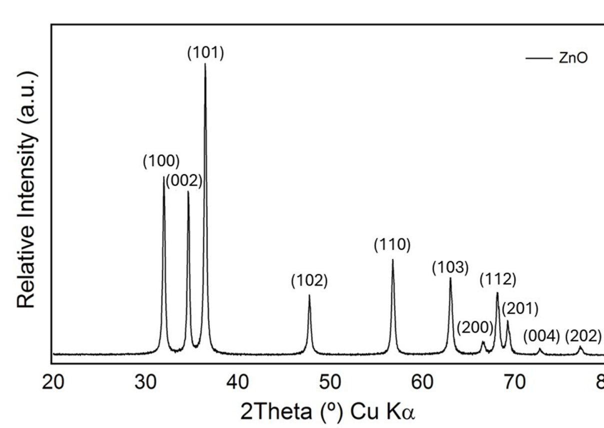

Fig. 1. XRD pattern of the annealed ZnO.

precipitation method. Peaks at $2\theta = 31.76°$, $34.42°$, $36.26°$, $47.54°$, $56.60°$, $62.86°$, $66.36°$, $67.94°$, $69.08°$, $72.54°$, and $76.98°$ correspond to the (100), (002), (101), (102), (110), (103), (200), (112), (201), (004), and (202) planes of hexagonal wurtzite ZnO (JCPDS 36-1451) [26,27]. No other peaks were observed in the XRD pattern, indicating the high purity of the product. Furthermore, Fig. 2(a—c) shows the morphologies of the as-synthesized ZnO and polymer-coated ZnO. The ZnO consists mainly of nearly spherical particles with an average diameter of 65 nm, while PEG- and OA-modified ZnO have average diameters of 82 nm and 75 nm, respectively. Modification of the ZnO nanoparticles resulted in a slight increase in particle diameter. This slight increase in size can be attributed to the possible coating of the modifiers on the surface of ZnO, as also observed in previous works [28,29]. Surface modification of nanosized ZnO has been shown to mitigate agglomeration and enhance its dispersibility [28,30].

The production of the synthesized nanoparticles may be described through the following sequential reactions. The Zn^{2+} ions from the $Zn(CH_3COO)_2$ precursors react with OH^- ions, forming the $Zn(OH)_2$ phase (Equation (1)) [22]. $Zn(OH)_2$ particles are then transformed into ZnO (Equation (2)). In the presence of modifiers such as OA or PEG, $Zn(OH)_2$ and any co-existing ZnO particles react to form complexes with the long alkyl chains of the modifiers (Equation (3)). These coordinated complexes, formed by OA or PEG, are likely located on the top and bottom surfaces of the ZnO particles, interacting through Van der Waals forces. Stabilizers then cap the surfaces, restricting anisotropic growth.

$$Zn(CH_3COO)_2 + 2NaOH \rightarrow Zn(OH)_2 + 2CH_3COONa \tag{1}$$

$$Zn(OH)_2 \rightarrow ZnO + H_2O \tag{2}$$

$$\begin{aligned} &2Zn(OH)_2 + HOOC(CH_2)_7CH = CH(CH_2)_7CH_3 \\ &\rightarrow Zn(OH)_2 - ZnO\left[OOC(CH_2)_7CH \right. \\ &\left. = CH(CH_2)_7CH_3\right]^- + H_3O^+ \end{aligned} \tag{3}$$

Pertinent functional groups on the surface of the samples were studied and are shown in the FTIR spectra of ZnO, surface-modified ZnO-PEG, and ZnO-OA in Fig. 3. A distinct peak in the 500—600 cm^{-1} range, characteristic of ZnO [29,30], is evident in all samples. In Fig. 2(a), the broad peak observed at 2800—3000 cm^{-1} corresponds to the alkyl stretching vibrations of C-H bonds in PEG [23,31], while the peak at 900—1000 cm^{-1} is attributed to the C-O stretching of phenol groups, confirming the presence of PEG on the ZnO nanoparticle surface [23,31]. A prominent peak at approximately 1600 cm^{-1} is indexed to the vibration of the C=O group of acetate, suggesting that acetate ions may have been adsorbed on the ZnO surface [32,33].

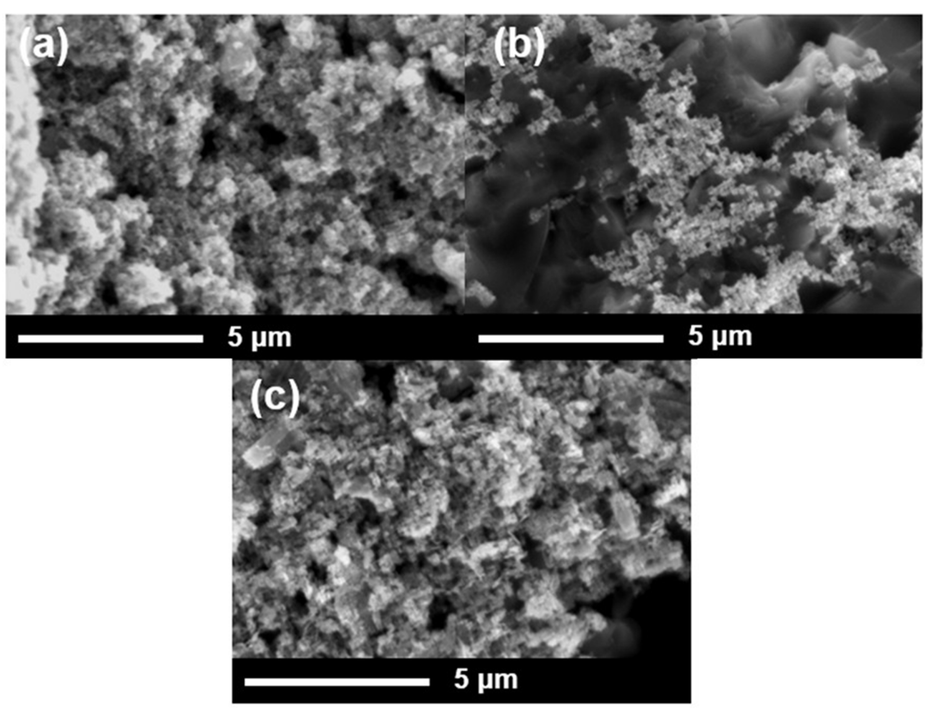

Fig. 2. SEM images of a) as synthesized-ZnO, b) ZnO-PEG and c) ZnO-OA nanoparticles.

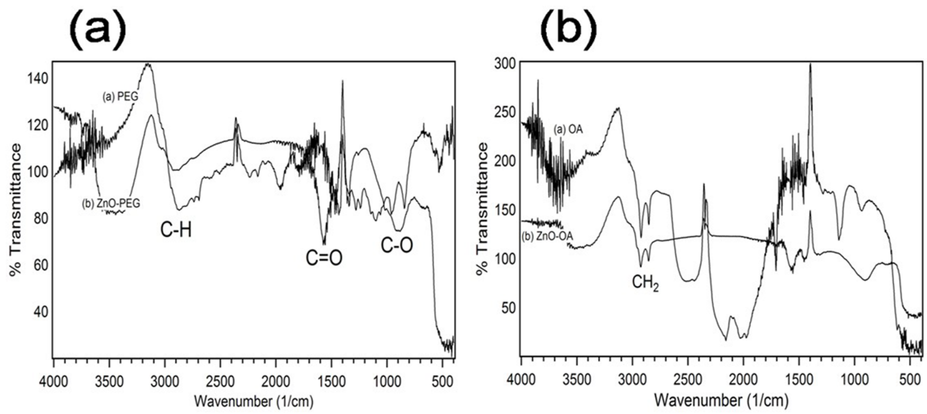

Fig. 3. FTIR spectra of modified ZnO nanoparticles with a) PEG and b) OA.

In Fig. 3(b), two distinct peaks at 2800–2900 cm^{-1} correspond to the asymmetric and symmetric stretching vibrations of -CH_2 groups present in oleic acid [30,32]. Additionally, a peak near 1700 cm^{-1} is assigned to the oleate ion, indicating its immobilization on the ZnO surface. These observations confirm the successful surface modification of ZnO using the biocamptible agents [34].

To determine the extent of coating and surface modification of the ZnO particles, the thermogravimetric graphs of the modified and ummodified ZnO products are shown in Fig. 4(a). In all curves, the initial decrease in weight at the onset of the analysis is attributed to the evaporation of moisture still present in the samples. For the unmodified sample, the degradation of the material is shown to commence at around 200 °C. From the stoichiometry of Equation (2), a decrease of around 18 wt % is expected for the degradation of the hydroxide to its oxide form. However, the thermogram only showed a total decrease of 1.5 weight % before stabilizing at around 600 °C. This strongly suggests that ZnO may have already formed even before calcination or that ZnO has also already formed during precipitation.

For the ZnO-OA sample, the portion at the 100–310 °C temperature range may be attributed to the decomposition of residual hydroxide followed by a two-step characteristic degradation of OA on the surface of ZnO at around 210 °C and 308 °C, as reported in recent work [35]. The onset degradation at 210 °C was attributed to the loss of physisorbed secondary layer of OA around a primary layer of OA, while the second weight loss at around 308 °C was due to the degradation of chemisorbed primary layer around the ZnO nanoparticles [35]. The weight loss then further increased, and the remaining OA finally degrades around its boiling point reported at 360 °C [36]. For the ZnO-PEG sample, slow decomposition of the hydroxide is apparent up to 180 °C followed by the gradual removal of the PEG component. Likewise, both calcination and evaporation processes may have simultaneously occurred. The trend strongly reflects the boiling point for PEG-400 at around 250 °C. With a total weight decrease of around 4.5%, this suggests

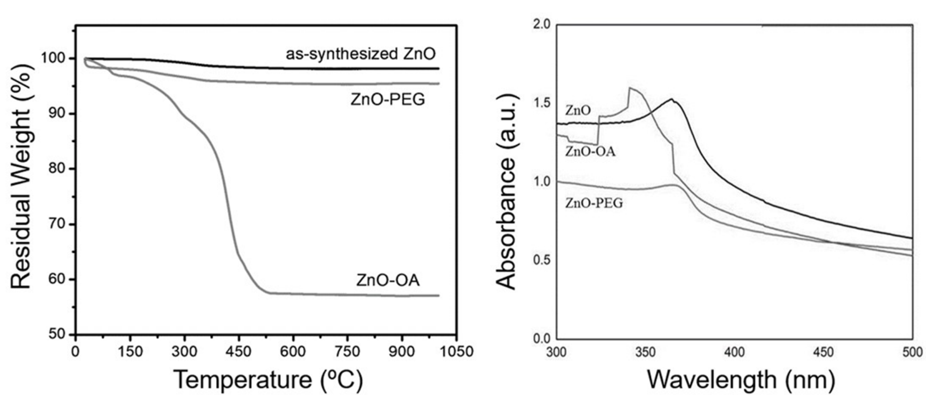

Fig. 4. (a) Thermogravimetric curves and (b) absorbance spectra of unannealed ZnO nanoparticles and ZnO modified with OA and PEG.

that more OA attaches to the ZnO nanoparticles as compared to PEG in terms of weight fractions.

To assess the UV-blocking properties of the synthesized samples, Fig. 4(b) presents the optical absorption spectra of the nanoparticles. The position of the absorbance peaks confirm its shielding capabilities in the UV-region. The maximum absorption wavelengths were observed at 365 nm, 368 nm, and 343 nm for unmodified ZnO, ZnO-PEG, and ZnO-OA, respectively, which lies well within the UVA and UVB regions. Corresponding band gaps were calculated to be around 3.40 eV, 3.32 eV, and 3.62 eV for ZnO, ZnO-PEG, and ZnO-OA, respectively. The increase in the bandgap for the ZnO-OA sample suggests attenuation of the photoactivity of ZnO which makes it a viable UV blocking material in topical sunscreen applications. Previous work also observed a blue-shift of the optical band gap after treating ZnO with OA which was attributed to energy level shifts due to the interaction between the nanoparticle and the capping agent [37]. Past research has shown that the presence of modifiers or capping agents on the surface of the nanoparticles affect the atomistic arrangement on the surface of the particle depending on the predominant crystallographic facet present on the surface, which ultimately affects optical band gap energies [38]. Further experimental and theoretical studies are needed to establish the effect of surface modifiers on the optoelectonic properties of ZnO.

4. Conclusion(s)

This study demonstrated the synthesis of ZnO and surface-modified ZnO nanoparticles via a facile chemical precipitation method. The resulting ZnO and surface-modified ZnO-PEG and ZnO-OA nanoparticles exhibited nearly spherical morphologies, with particle diameters ranging from 65 to 82 nm. Thermal analysis revealed weight reductions of 4.5% and 42.5% for PEG- and OA-modified ZnO, respectively. Optical characterization estimated energy band gaps of 3.32 eV and 3.62 eV for PEG- and OA-modified ZnO, respectively. The increase in the optical band gap of ZnO with the OA modification underscores its potential for cosmetic applications, particularly in UV-blocking formulations where reduced photocatalytic activity is desired. Further work is highly encouraged to validate these promising results in *in vitro* and *in vivo* studies.

Despite these encouraging results, several challenges remain in this field. There is still a need to better understand the effects of surface modifiers on the photoactivity and optoelectronic properties of ZnO. In addition, standardized safety testing for nanoparticle-based UV blockers is lacking, particularly with respect to their long-term effects on skin and their environmental impact. Future studies should therefore focus on clarifying the interactions between surface modifiers, UV-blocking materials, and human tissue, as well as on developing modification strategies that enhance photostability while ensuring environmental safety.

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Conflicts of interest

The authors have no relevant financial or non-financial interests to disclose.

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