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Original Research Article

A Zinc Oxide Microparticle (ZnOMP) Characterization Test Using Sumbawa Oil (*Minyak sumbawa*) Base Material

Chiquita Prahasanti[1]*, Syafrizal A Pamungkas[1], Ika Febryana[1], Eka F Augustina[1], Irma J Savitri[1], Veronika P Widiasih[2], Izzul Islam[3]

[1]*Department of Periodontology, Faculty of Dental Medicine, Universitas Airlangga, Surabaya, East Java, Indonesia*
[2]*Faculty of Dental Medicine, Universitas Airlangga, Surabaya, East Java, Indonesia*
[3]*Lifescience and Technology Laboratory, Faculty of Lifescience and Technology, Universitas Teknologi Sumbawa, Sumbawa, West Nusa Tenggara, Indonesia*

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ABSTRACT

Sumbawa oil (*Minyak sumbawa)* has long been used for therapeutic and biomedical purposes. However, its potential use as a capping agent in the synthesis of zinc oxide microparticles (ZnOMPs) has not been thoroughly examined. Therefore, this study aims to characterize the properties of ZnOMPs synthesized using *Minyak Sumbawa* as a capping agent. ZnOMPs were synthesized and characterized in both capped and uncapped forms. The crystallinity of the samples was analyzed using X-ray diffraction (XRD), while particle morphology was examined through scanning electron microscopy (SEM). Fourier-transform infrared (FTIR) spectroscopy was employed to identify functional groups and assess the interactions between *Minyak Sumbawa* and ZnOMPs. Our findings demonstrated that ZnOMPs capped with *Minyak Sumbawa* resulted in smaller particle sizes, more uniform distribution, and better stability compared to uncapped ZnOMPs. FTIR spectra confirmed the presence of carbonyl (C=O) and hydroxyl (O–H) functional groups, which indicates interactions between the bioactive components of *Minyak Sumbawa* and the ZnOMP surface. XRD analysis confirmed that the capped ZnOMPs maintained a pure wurtzite crystal structure with no detectable impurities. In conclusion, the use of *Minyak Sumbawa* as a natural capping agent effectively reduced ZnOMP particle size to approximately 0.3 µm, improved uniformity, and preserved crystal purity while facilitating beneficial bioactive interactions.

Keywords: Biosynthesis, Capping agent, Characterization, *Minyak Sumbawa*, Zinc oxide microparticles.

Introduction

Spherical particles with diameters ranging from 1 and 1000 micrometers are known as microparticles. The development of microparticle-based material has become an important and widely applied area in pharmaceutical technology, as they have proven to be a safe and effective drug delivery systems. Compared to traditional pharmaceutical formulations, recent advances in biomaterials have enabled the design of processable, biocompatible, biodegradable, and nontoxic systems, allowing the development of more efficient and beneficial drug delivery platforms.[1] The application of microparticles and their closely related form, nanoparticles, has also emerged as a key area of innovation in dentistry, with their small size and large surface area conferring advantageous properties that improve material performance. Ongoing studies have explored the use of these materials to design novel therapeutic and regenerative strategies, and to enhance the functional characteristics of existing dental materials.[2]

*Corresponding author. Email: chiquita-p-s@fkg.unair.ac.id
Tel: +62 31 5030255

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Among the various types of microparticles, zinc oxide microparticles (ZnOMPs) are some the most extensively studied and have demonstrated promising applications in wound healing, dental coatings, and as functional components in cosmetic and pharmaceutical formulations.[3] However, a major challenge in their synthesis is the tendency of particles to agglomerate or clump together, which decreases their stability and biological activity. The use of capping agents is therefore crucial, as these stabilizing compounds coat the particle surface, preventing aggregation, and thereby enhancing dispersion and performance.[4] The application of environmentally friendly natural materials as reducing and capping agents in the synthesis of microparticles, commonly referred to as green biosynthesis, has gained increasing attention in recent years.[5] One potential natural source for this process is *Minyak Sumbawa*, a traditional herbal oil derived from extracts of several native plants. The main constituents of *Minyak Sumbawa* include flavonoids, polyphenols, saponins, monoterpenes, fatty acid esters, unsaturated fatty acids, saturated fatty acids, fatty acid esters, and sesquiterpenes. Notably, it contains high concentrations of monoterpenoids and sesquiterpenes including δ-decalactone, 9-octadecanoic acid methyl ester, 1,8-cineole, trans-caryophyllene, and zingiberene.[6] However, despite its rich phytochemical composition, the application of *Minyak Sumbawa* as a capping agent in the synthesis of ZnOMPs has been little investigated.

Hence, the purpose of this study is to characterize biosynthesized ZnOMPs prepared with and without *Minyak Sumbawa* coating and to assess the effects of this natural oil on particle size, morphology, crystallinity, and surface functional groups. The synthesized microparticles were characterized using X-ray diffraction (XRD), Fourier-transform infrared spectroscopy (FTIR), and scanning electron microscopy (SEM).

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Materials and Methods

Plant collection and identification

The herbal ingredients for *Minyak Sumbawa* were collected between June 1 and 13, 2024. Samples of *Minyak Sumbawa* components were taken from Sumbawa Regency, West Nusa Tenggara, Indonesia, (8.7381° S and 118.1171° E). The Biology Service Unit of Airlangga University's Faculty of Science and Technology performed taxonomic analysis, with identification number 20/06/AA/2024.

The study samples consisted of uncapped ZnOMPs and *Minyak Sumbawa*-capped ZnOMPs (CZnOMPs). The biosynthesis and sample preparation were conducted in the Microbiology Laboratory, Faculty of Life Sciences and Technology, Sumbawa University of Technology. Characterization analyses, including XRD, SEM, and FTIR, were performed at the Advanced Minerals and Materials Laboratory, Faculty of Mathematics and Natural Sciences (FMIPA), State University of Malang, in June 2024.

Minyak Sumbawa extract preparation

The extraction of Minyak Sumbawa was achieved through the maceration technique, employing a 1:10 ratio (400 g: 4,000 mL) of 96% ethanol, ensuring complete immersion of the ingredients.[7]

Green synthesized ZnOMP preparation

Uncapped ZnOMPs were synthesized by mixing 400 mL of the *Minyak Sumbawa* extract solution with 0.2 M $Zn(NO_3)_2 \cdot 6H_2O$, stirring at 60°C for one hour, followed by heating at 80°C for another hour. The mixture was dried at 150°C and then calcined by gradually increasing the temperature from 600°C to 1000°C for 15 minutes to yield white ZnOMPs powder.[8]

Capped ZnOMP preparation

For CZnOMPs, a total of 10 grams of biosynthetic ZnOMPs was added to 100 mL of deionized water (DI), then homogenized for two hours using a magnetic stirrer for one hour. Subsequently, the biosynthetic ZnOMPs solution was added to 10 mL of *Minyak Sumbawa* extract that had previously been added to 90 mL of deionized water. Both solutions were subjected to a homogenization process for a duration of two hours to facilitate the nucleation stage. Subsequent to this, the mixtures were agitated with a magnetic stirring device and then exposed to an ultrasonic bath for a total of 2 x 15 minutes. The samples were then put through a spinning procedure at a speed of 1,000 rpm for 15 minutes. The *Minyak Sumbawa*-based ZnOMPs capping was obtained through a drying process that utilized an oven maintained at a temperature of 60°C for a duration of one hour.[9]

XRD examination

XRD analysis was performed to determine particle size and crystal structure using the Debye–Scherrer equation. Approximately 0.5 g of ZnOMP or CZnOMP powder was placed on a sample plate and analyzed with Cu Kα radiation (λ = 1.5406 Å) at 40 kV and 30 mA. Diffractograms were processed using Origin software to calculate the crystallite size.[10]

SEM examination

SEM was used to examine particle morphology and size distribution at magnifications of 10,000× and 50,000×. Approximately 0.5 g of each ZnOMP and CZnOMP sample was transferred into a vial, coated with a thin layer of gold, and mounted on a holder using double-sided carbon tape or conductive paste to enhance conductivity. The samples were analyzed at the Forensic Laboratory Center of the National Police Criminal Investigation Agency in Bogor, Indonesia, providing three-dimensional images of the microparticle surfaces.[11,12]

FTIR examination

FTIR spectroscopy was performed to identify surface functional groups on both capped and uncapped ZnOMPs. Approximately 0.5 g of each powdered sample was mixed with potassium bromide (KBr) and pressed into a pellet under ~7 tons of pressure.[13] Spectra were recorded over the range of 4000–400 cm^{-1}, and infrared absorption was measured to determine the functional groups. The resulting spectra were processed using OriginLab 2018 to analyze the chemical composition.[14,15,16]

Results and Discussion

The purpose of this study was to examine the characteristics of ZnOMPs synthesized with and without *Minyak Sumbawa* extract as a capping agent. The synthesis process produced a white ZnOMP in the uncapped form and a golden-brown ZnOMP powder when capped with *Minyak Sumbawa*. The samples were further characterized using XRD, SEM, and FTIR.

XRD analysis was conducted to determine the crystal structure of the synthesized ZnOMPs. Diffraction patterns for both samples exhibited peaks characteristic of the ZnO wurtzite crystal structure, with no additional phases or contaminants detected, confirming the purity of the synthesized microparticles. For the uncapped ZnOMPs, the peak of highest crystallinity was observed at 2θ = 36.20°, and the average crystallite size, calculated using the Scherrer equation, was consistent with typical ZnO structures. In capped ZnOMPs. The main peak showed a slight shift to 2θ = 36.12°, suggesting interactions between the ZnO surface and bioactive compounds from *Minyak Sumbawa* (Figures 1 and 2).

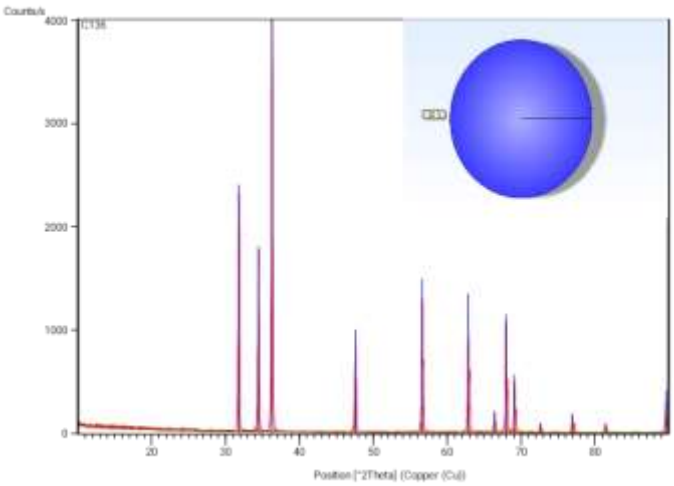

Figure 1: XRD diffraction pattern of ZnOMPs.

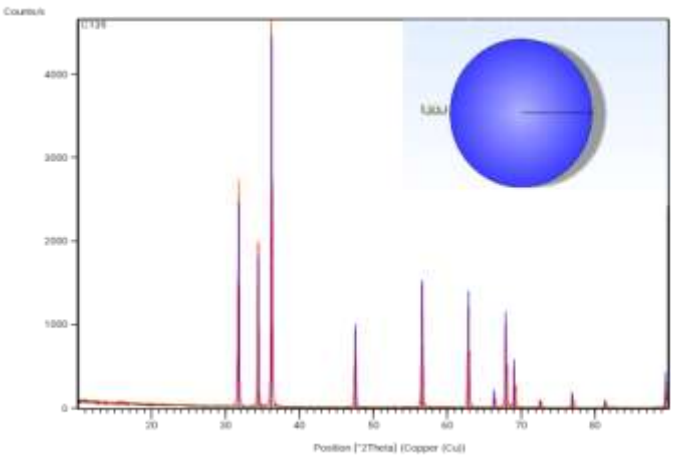

Figure 2: XRD diffraction pattern of CZnOMPs.

SEM analysis revealed differences in surface morphology and particle size distribution between capped and uncapped ZnOMPs. Uncapped ZnOMPs displayed relatively large, irregular particles with an average

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of 0.5-0.7 μm, with heterogeneous morphology and notable particle aggregation. In contrast, capped ZnOMPs demonstrated smaller, more uniform particles averaging approximately 0.3 μm, with smoother surfaces, indicating enhanced stability (Figure 3). Although both samples fall within the microparticle size range, these morphological differences confirm that capping with *Minyak Sumbawa* influenced particle size distribution and surface uniformity.

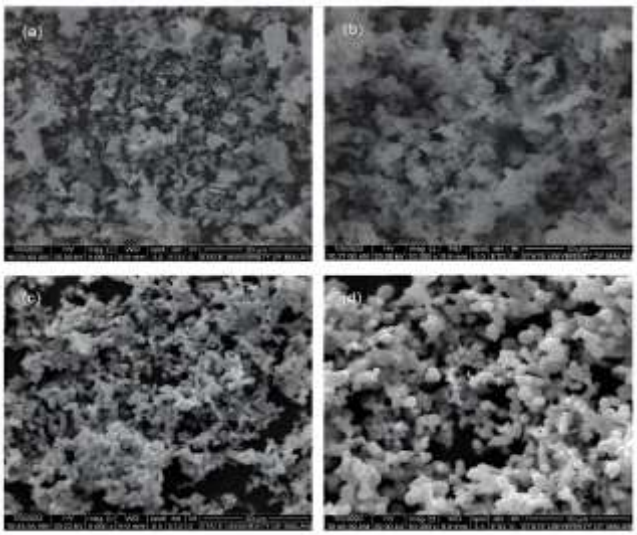

Figure 3: The SEM results for the CZnOMPs are shown in Figures (a) and (b), and the SEM results for the ZnOMPs are shown in Figures (c) and (d)

FTIR analysis was performed to identify the surface functional groups. The FTIR spectra of capped ZnOMPs displayed characteristic absorption peaks corresponding to ethylene C=C, amine (C-N), alkane (C-H), and hydroxyl (O-H), indicating chemical interactions between bioactive compounds in *Minyak Sumbawa* and the ZnOMP surfaces. In contrast, uncapped ZnOMPs showed no significant absorption bands within the 1250-4000 cm^{-1} range, indicating the absence of organic functional groups and confirming that the surfaces were not coated with organic substances (Figure 4). The functional groups identified in capped ZnOMPs are summarized in Table 1.

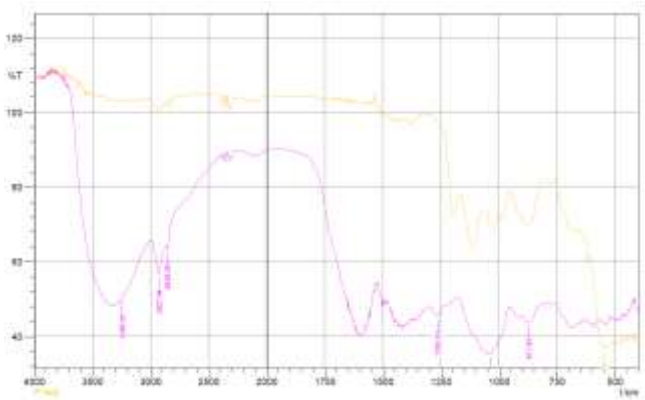

Figure 4: FTIR spectra of (a) CZnOMPs (b) ZnOMPs

Previous studies have shown that capping microparticles with natural materials can improve their stability and effectiveness in medical and pharmaceutical applications.[17] Our findings indicate that ZnOMPs capped with *Minyak Sumbawa* exhibit greater stability than uncapped particles. *Minyak Sumbawa* was extracted to identify bioactive compounds capable of acting as reducing agents and stabilizers during ZnOMP synthesis.[18] The extraction yielded 1,200 mL of a dark green solution, which contributed to the enhanced stability and biological activity of the resulting ZnOMPs.[19,20]

Table 1: FTIR analysis data of CZnOMPs sample.

Peak (cm^{-1})	Functional groups
3246	O-H (alcohol)
2927	C-H stretch (alkane)
2856	C-H stretch (alkane)
1265	C-N (amine)
1041	C-C (ethylene)
871	C-H bending (alkene)
547	M-O (Zn-O)

In the FTIR examination of ZnOMPs. There were no main peaks at wavelengths of 1250 cm^{-1} – 4000 cm^{-1}. This indicates that the synthesis of ZnOMPs does not have C-H, C-C, and C-N groups.
ZnOMPs were synthesized by reacting the precursor $Zn(NO_3)_2 \cdot 6H_2O$ with the *Minyak Sumbawa* extract under controlled heating at 60°C and then 80°C, followed by drying and calcination to produce a white powder. This green synthesis method enables the production of smaller, more stable particles compared to conventional chemical methods.[21,22]
The structural and morphological properties of ZnOMPs were characterized using FTIR, SEM, and XRD, each providing insight into particle structure, morphology, and interactions with *Minyak Sumbawa* bioactive compounds.[23] A discrepancy in size was identified through a comparative analysis of XRD and SEM examinations. Specifically, SEM images revealed the presence of aggregated clusters ranging from 0.3 to 0.7 micrometers, while XRD analysis substantiated the material's fundamental nanoscale nature, yielding a crystallite size of 22-26 nanometers. Consequently, the examination results indicate the occurrence of agglomeration in the SEM examination. Due to their naturally high surface energy and powerful van der Waals forces, nanoparticles with a high surface area, such as those between 22 nm and 26 nm, tend to cluster (agglomerate) on their own when dried into a powder. The material's intrinsic nature is nanoscale, as demonstrated by the XRD, but the SEM reveals big clusters. This indicates that the physical clustering only hides the material's nano-properties, such as its large specific surface area, which is crucial for catalysis or adsorption.[24]
Zinc oxide microparticles, such as ZnOMPs, hold significant potential for biomedical and pharmaceutical applications, although their stability in biological systems remains a challenge.[25] This study demonstrates that *Minyak Sumbawa* effectively enhances both stability and biological performance during green synthesis. Altogether, the study has both theoretical and practical implications. Theoretically, it reinforces the principles of green synthesis, promoting the use of natural materials for functional microparticle production while practically it shows that *Minyak Sumbawa* can support environmentally friendly synthesis while producing ZnOMPs with desirable physicochemical properties suitable for medical and industrial applications.[26]

Conclusion

In conclusion, both capped and uncapped ZnOMPs possess a pure wurtzite crystal structure. However, capping with *Minyak Sumbawa* resulted in smaller, more uniform particles (~0.3 μm) compared with uncapped ZnOMPs (0.5–0.7 μm). FTIR analysis confirmed that the bioactive compounds in *Minyak Sumbawa* interacted with the ZnOMPs, with higher concentrations present in the capped particles, thereby contributing to their enhanced stability and uniformity. These findings highlight the effectiveness of *Minyak Sumbawa* as a natural capping agent to improve the physicochemical properties of ZnOMPs and as a key pre-formulation step for advanced biomedical applications, such as anti-inflammatory and wound-healing capabilities, in further studies.

Conflict of Interest

The authors declare no conflict of interest.

Authors' Declaration

The authors hereby declare that the work presented in this article is original and that any liability for claims relating to the content of this article will be borne by them.

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