Wadi Alshatti University Journal of Pure and Applied Sciences

مجلة جامعة وادي الشاطئ للعلوم البحتة والتطبيقية

Volume 4, No. 1, January-June 2026 Online ISSN: 3006-0877 المجلد 4، الاصدار 1، يناير- يونيو 2026

RESEARCH ARTICLE **NANO-SCIENCE**

Facile Green Synthesis of Zinc Oxide Nanoparticles and its Enhancement in Some Cotton Fabric Properties

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ARTICLE HISTORY
Received 13 April 2026
Revised 27 April 2026
Accepted 04 May 2026
Online 24 May 2026

KEYWORDS
Ficus Carcia;
Green synthesis;
Zinc oxide nanoparticles;
Characterization;
Abrasion resistance.

ABSTRACT

An eco-friendly and facile method has been used for the synthesis of zinc oxide nanoparticles from Ficus carica (Fig) extract. The aqueous extract of (Fig leaves) was used as a reducing and capping agent in the synthesis of zinc oxide nanoparticles. The characterization using XRD, SEM, FTIR confirms the preparation of zinc oxide nanoparticles, ZnO NPs. The average particle size of the prepared ZnO nanoparticle was approximately 36.58 nm calculated from the XRD data. The prepared zinc oxide nanoparticles showed a spherical agglomerated porous nanostructure according to the SEM images. Further, cotton lawn fabrics were coated with zinc oxide nanoparticles via a simple technique using less chemicals, the coated cotton outperforms uncoated cotton in terms of thermal stability and abrasion resistance.

التحضير الأخضر السهل لجزيئات أكسيد الزنك النانوية وتعزيزها في بعض خصائص نسيج القطن

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الكلمات المفتاحية

نبات الكاريكا
التحضير الأخضر
أكسد الزنك النانوي
تشخيص
مقاومة التمزق

الملخص

تم استخدام طريقة صديقة للبيئة وسهلة لتحضير جسيمات أكسيد الزنك النانوية من مستخلص نبات الكاريكا (التين). تم استخدام المستخلص المائي لـ (أوراق التين) كعامل اختزال وتغطية في تحضير جسيمات أكسيد الزنك النانوية. يؤكد التوصيف باستخدام XRD و SEMو FTIR تحضير جسيمات أكسيد الزنك النانوية ZnO NPs . وكان متوسط حجم الجسيمات لجسيمات ZnOالنانوية المحضرة حوالي 36.58 نانومتر محسوبًا من بيانات XRD . أظهرت جسيمات أكسيد الزنك النانوية المحضرة بنية نانوية مسامية متكتلة كروية وفقًا لصور المجهر الإلكتروني الماسح. علاوة على ذلك، تم طلاء أقمشة العشب القطنية بجسيمات أكسيد الزنك النانوية، ويتفوق القطن المطلي على القطن غير المطلي من حيث الثبات الحراري ومقاومة التمزق.

Introduction

As an alternative to chemical and physical processes, green synthesis is a developing bio-nanotechnology technique that has advantages for the environment and economy. Ecofriendly, safe and nontoxic reagents are used in this study. Nanomaterials are at the forefront of nanoscience and nanotechnology, with their distinctive dimensions at the range of 1-100 nanometres (nm). Recently, a wide range of applied sciences, from material science to biology, have shown a special interest in nanomaterials more especially metal nanoparticles [1,2]. Because of their incredibly small size and high surface-to-volume ratio, which increases their contact surface with other objects, nanomaterials are precisely important. Applications for metal and metal oxide nanoparticles have been demonstrated in a wide range of industries, including textile, agriculture, medical, pharmacy, catalysis, consumer products manufacturing, and antimicrobial testing [3].

Nanomaterial substances can be prepared by three different methods: (1) Physical method by which the raw material is grinded to get very fine powder of a particle size less than 100 nm [4]. Physical method which is time and energy consumption, (2) Chemical methods such as gel-sol method, chemical reduction, pyrolysis [5], (3) Biological methods also known as "green" technologies involving naturally occurring sources and their derivatives [6].

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https://doi.org/10.63318/waujpasv4i1_54

According to previous reports [7], plants comprise organic substances such as amino and carboxylic acids, flavonoids, phenols, ketones, and proteins. These substances are essential for the simple, quick, and ecologically safe recovery of mineral salts as well as the synthesis of nanoparticles. Conventional methods of synthesizing nanoparticles such as spray pyrolysis, thermal decomposition and sol-gel, are commonly evaded as they involve critical temperature, pressure and release toxic end products to the environment in addition to their high expense. Hence the plant mediated synthesis of the nanoparticles can serve as a better alternative, more ecofriendly and gains more importance [8].

In this context, seeking cheaper and safer materials for synthesis of ZnO NPs, urged the authors to think for a locally (Libya) available plant, which release its leaves every Autumn (such as the fig plant). Fig (*Ficus carica*) is a tree or shrub inherent to the Mediterranean, Southwest, and Middle East Asia, blooming in warm, dry climates. It belongs to the Moraceae (mulberry) family. Furthermore 100 % cotton fabric was incorporated with zinc oxide nanoparticles to investigate some of the mechanical properties of the cotton fabric.

Zinc oxide nanoparticles (ZnO NPs) have acquired great significance in the textile sector due to their impressive efficiency and multifold utilization, such as antimicrobials, UV protection, photo catalytic activity, and super hydrophobicity behaviour and self-cleaning. [9]

Methodology

Materials

Zinc nitrate was provided from (Carlo Erba)

The fig leaves were collected from the author's house garden in August 2025.

Deionised water was used whenever needed.

Instruments

Scanning Electron Microscope (SEM), **JSM-5610LV JEOL** (Industrial Research Centre); Attenuated total reflectance-Fourier transform infrared (ATR-FTIR), **Cary 630 ATR-FTIR benchtop** (Libyan Advanced Center for Chemical Analysis); Differential Thermal Analyser, TG/DTG, **LD-LDTA-A10, Labodam** (Libyan Polymer Research Centre); X-ray diffractometer **X'Pert Pro PAN analytical Xray** with Cu Kα radiation (1.5406 Å) generated at 45 kV and 35 mA (The Egyptian Mineral Resource Authority-Egypt).

Preparation of Fig leaves extract

The fig leaves were collected in November 2025 from the house garden in Al-Mashtal (Tripoli), cleaned thoroughly first using a continuous flow of tap water to remove any dust or contaminants, then with deionized water and finally air dried (the dried leaves were stored in a glass container and kept at 4°C). The leaves were then dried in a drying oven at 40°C for two hours before being ground to a fine powder. 10 g of the dried substance was then soaked in 100 mL deionized water and heated at 60°C for about 60 minutes. The solution was cooled, decanted, filtered and stored at 4°C for further use as illustrated in figure 1 [10].

Preparation of zinc oxide NPs

The process of manufacturing the nanoparticles was extensively described by Rusli et al. [10], with slight adjustments implemented to the time of calcination 4hrs instead of 2hrs without agitation.

Initially, 50 ml of the plant extract were poured in 200ml ceramic crucible and subjected to heating at 60°C giving a yellow extract.

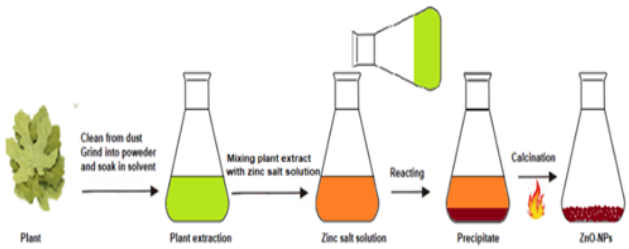

Figure 1: Process of green synthesis of ZnO NPs from Fig Leaves Extract

Furthermore, 5 grams of zinc nitrate hexahydrate was added at 60 °C. The heating was continued until a brown paste-like material was formed. The paste-like product was calcined at 400°C for 2hrs resulting in a white fluffy powder, ZnO NPs. The resulted powder was collected carefully and characterised.

Preparation of ZnO-Coated Cotton Fabrics

A cotton fabric (10*10 cm) was immersed in a solution of 0.1M zinc nitrate ($Zn(NO_3)_2.6H_2O$) solution followed by the addition of the Fig leaves extract dropwise under continuous magnetic stirring at room temperature. The temperature was then increased to 90°C for 120 minutes under continuous stirring. The fabric samples were dried in oven at 90°C for 60 minutes [11].

Results and Discussion

The reaction of Ficus carica (Fig) extract with hexahydrate zinc nitrate resulted in the formation of white nanoparticles of zinc oxide originally observed by change in colour from yellow to dark brown.

Characterisation of ZnO NPs

The ZnO nano material was characterised by infrared (FTIR), thermogravimetric (TGA), X-ray diffraction (XRD) and scanning electronic microscope (SEM).

FTIR Analysis

The FTIR spectrum of the green synthesized zinc oxide nanoparticles using aqueous leaf extract of Ficus carica (Fig) is illustrated in figures 2. This was carried out to identify the functional groups of the possible biomolecules responsible for the capping and stabilization of ZnO NPs.

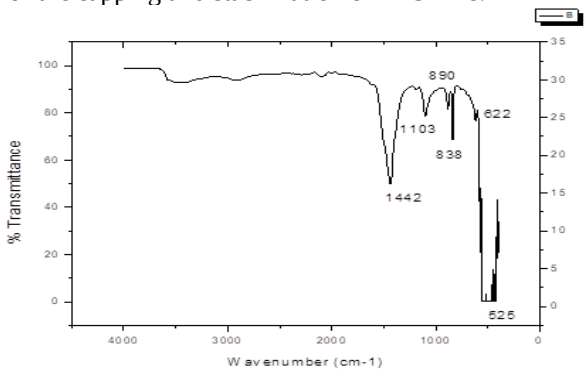

Figure 2: FTIR spectrum of prepared zinc oxide nanoparticles.

According to the literature the spectrum clearly shows ZnO absorption band in the region 400 and 622cm^{-1} corresponding to the ZnO stretching frequency of Zn–O bonds confirming the presence of M–O vibrational bands [12]. The band at 1103 cm^{-1} indicates the presence of C=C stretching in the aromatic ring in polyphenols and aliphatic amines [13-15]. The peak at 2300 cm^{-1} originated from di-substituted alkynes. While the broad band in between 3000-3700cm^{-1} is due to the

O-H stretching vibrations of alcohols, primary and secondary amines, and C-H stretching of alkanes [13].

XRD Analysis

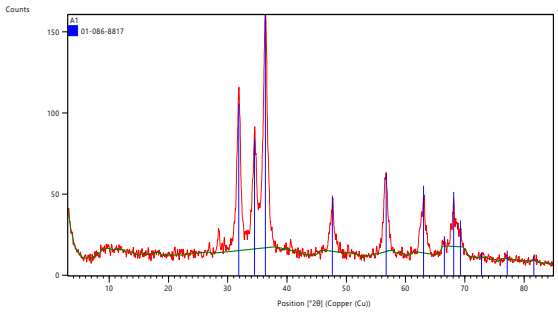

Figure 3: X-Ray Diffraction pattern of ZnONPs.

Table 1: Semi Quantitative of analysis of ZnONPs

Ref. Code	Compound Name	Chemical Formula	Semi Quantitative %
01-086-8817	Zinc Oxide	ZnO_2	100

The white powder ZnO NPs was subjected to XRD analysis as shown in figure 3 and is compared to the standard values found in JCPDS file no. 89-1397. Semi quantitative analysis is shown in Table 1. The crystalline structure of the as-prepared ZnO obtained peaks are indexed as 31.88(100), 34.50(002), 36.33(101), 47.69(102), 56.70(110), 63.03(103), 68.10(112), 69.13(201). The successful production of ZnO NPs is confirmed by the fact that all observable peaks match the pure hexagonal wurtzite phase of ZnO NPs. Previous investigations for the green manufacture of zinc oxide nanoparticles were consistent and attained similar X-Ray diffraction patterns [16-18].

Furthermore, the average crystallite size of the green synthesized ZnO NPs was calculated to be 36.6 nm by applying the widely recognized Debye-Scherrer equation (Eq. 1) to ascertain the nano-sized structure of the generated product, consistent with that reported by [19].

$$D = \frac{K\lambda}{\beta cos\theta} \qquad (1)$$

Where:
D= average crystallite size
K= shape factor (0.9)
λ = Cu kα radiation wavelength (1.54060 Å),
β = the peak width at half maximum (in radians)
θ = Bragg's diffraction angle.

Scanning Electron Microscope (SEM)

The SEM examination, revealed the formation of zinc oxide clusters, with a spherical and bullet like morphology. Topographical view of the SEM images of the as-prepared nano zinc oxide particles at different magnifications shows more or less porous structure. Porous materials are more favourable than non-porous and opens more applications for the use of ZnO NPs due to the higher surface area compared to a dense film, this makes it useful in several applications such as catalysis, photocatalysis [20] and gas sensing [21]. On contrast, several studies reported different morphologies for non-porous zinc oxide nanoparticles using different plants some examples encounter; Rod and cubic shaped using Bacillus megaterium [22], spheroid ZnONPs using Agaricus bisporus [23], Rod-like nano-structured using Anabaena cylindrica [24], Coral-like structure using wasted hops [25] and spherical and hexagonal-shaped using Eucalyptus Globulus extracts. [26]

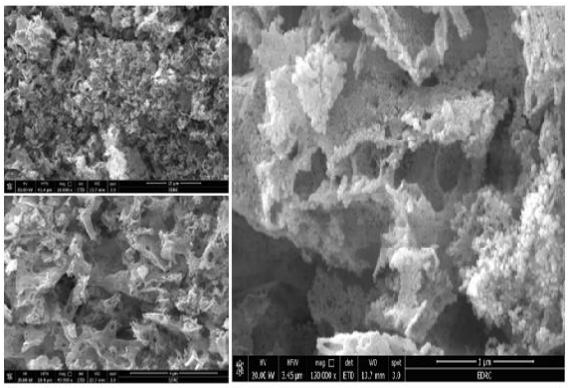

Figure 4: SEM images of green-synthesized ZnO NPs

TGA/ DTG analysis

Thermogravimetric analysis (TGA) combined with Derivative thermogravimetry (DTG) to examine the thermal characteristics of the as-prepared ZnO nanoparticles, under N_2, heated from room temperature to 700 °C at a heating rate of 10 °C/min. The results of the TGA and DTG analyses (Figure 5) show that the percent weight loss of 3.5 % at 103 °C is due to the dehydration (loss of physically adsorbed water) of ZnO NPs, followed by a significant weight loss of 17.86 % at 171°C, which is attributed to the decomposition and volatilization of organic moieties. A final weight loss, 1.21% was observed at an onset of 231°C no any further weight loss was observed beyond 255°C upon further heating to 700°C consistent with reported in the literature. [27]. The DTG curve show clearly the temperatures at which the weight loss occurred.

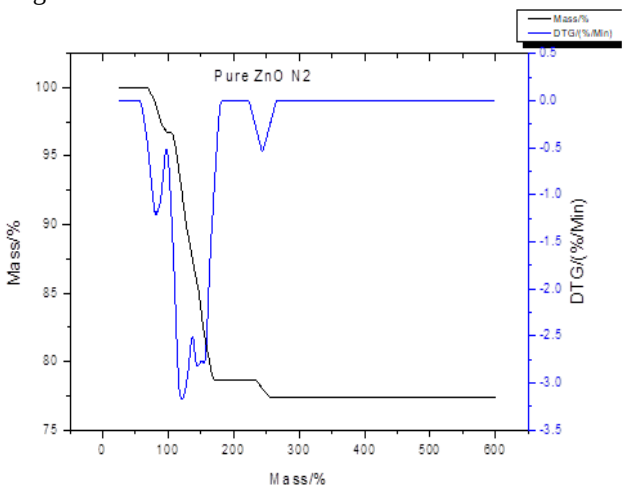

Figure 5: TGA/DTG of as prepared ZnO NPS

Properties of coated cotton Fabric lawn

Due to its exceptional effectiveness and multiple uses, including antimicrobials, UV protection, photocatalytic activity, and self-cleaning, zinc oxide nanoparticles (ZnO NPs) have gained significant importance in the textile industry [11]. Preliminary tests were performed by treating a standard cotton fabric (ISO F09) with the as-prepared zinc oxide nanoparticle material using a one-step facile method as previously described [11].

The coated cotton fabric was tested using different techniques, ATR-FTIR, TGA/DTG, SEM, air permeability and abrasion resistance test in contrast to the uncoated cotton fabric.

ATR-FTIR Analysis

Figure 6 showed ATR-FTIR spectra vibrations of uncoated cotton and cotton coated ZnO.

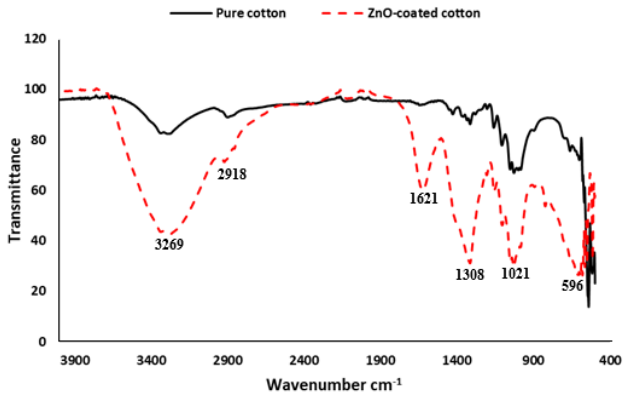

Figure 6: FTIR spectrum of coated and uncoated cotton fabric

The ATR-FTIR spectra of the uncoated Cotton and cotton coated ZnO presents significant changes indicating a successful incorporation of zinc oxide nanoparticles within the cotton fabric. The cotton exhibited bands at 3269 cm^{-1} and 3290 cm^{-1} corresponding to OH stretching, the band at 2918 cm^{-1} was due to CH_2 stretching, 1021 cm^{-1} peak was due to C-O-C bridge. The absorption peaks at around 496 and 596 cm^{-1} was due to Zn-O bonding on coated cotton [28,29].

Furthermore, the surface morphology of the coated and uncoated cotton was analysed using a JEOL JSM.5610LV Scanning Electron Microscope. The SEM images of uncoated cotton (a) and cotton coated ZnO (b) are shown in figure 7.

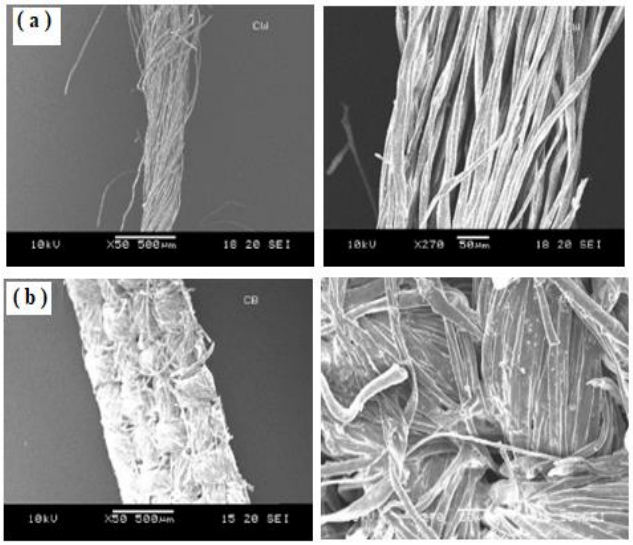

Figure 7: SEM images of (a) uncoated cotton and (b) coated cotton

Figure 7 showed SEM images of (a) uncoated cotton and (b) coated cotton with zinc oxide nanoparticles. The morphology of uncoated cotton is a smooth surface with loosely arranged fibres while that of the cotton coated ZnO the fibres are more compact scattered with ZnO nanoparticles adhering to the surface.

The Thermal behavior of cotton coated ZnO and the uncoated cotton was examined by heating from room temperature to 700°C at a heating rate of 10°C per minute under inert gas N_2. The results of the TGA and DTG analyses (Figure 8) showed that the cotton coated ZnO resulted in reducing the oxidative reactions and combustion during thermal decomposition, this may be due to the formation of a protective barrier that limits the access of oxygen or heat to the fibre. As a result, the final weight loss of the cotton coated ZnO (88.21%) is less than that of the uncoated cotton (96.7%).

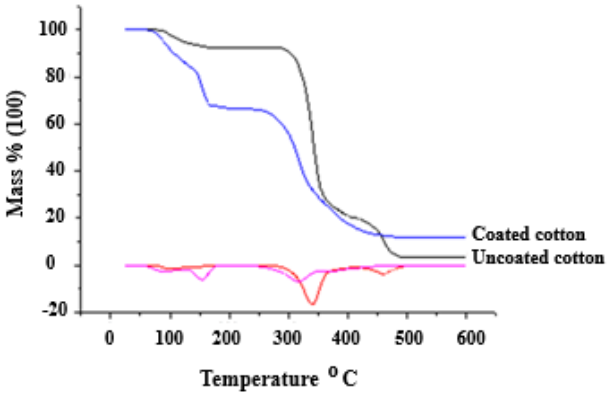

Figure 8: TGA and DTG of coated and uncoated cotton fabric

Table 2 showed air permeability and abrasion resistance of coated and uncoated cotton, there is no air permeability difference between the coated and uncoated fabrics showing a moderate permeability, which allows a reasonable amount of air flow making it beneficial for comfort, especially in clothing.

Table 2: shows the abrasion resistance and air permeability of the coated cotton lawn fabric.

Samples	Air permeability (ISO 9237)	Abrasion resistance (ISO 12947-2)
Uncoated cotton	>2500 (ml/min)	11500 (cycle)
Coated cotton	>2500 (ml/min)	20000 (cycle)

Whereas, a significant improvement was observed in the durability of cotton coated ZnO indicating a substantial enhancement in its ability to withstand wear and tear. This may be due to penetration of the nanoparticles of ZnO into the cotton fabric creating a protective layer on the fabric, reducing friction between fibres and preventing fibre breakage during abrasion leading to a longer lifespan for the fabric. This may expedite new applications for the coated cotton making it suitable for items that experience more wear, such as active wear, outdoor clothing or durable home textiles.

Conclusion and Recommendations

Green synthesis of ZnO nanoparticles using Ficus carica (Fig) aqueous extract was successful, offering a novel approach due to its eco-friendly, simple and less expensive route. The resulted zinc oxide nanoparticles exhibited a distinctive hexagonal wurtzite structure with an average crystallite size of 36.6 nm and a spherical bullet-like porous morphology. The FTIR spectral analysis reveals the characteristic peaks for Zn-O stretching. It was demonstrated that treatment of cotton lawn fabric with zinc oxide nanoparticles could improve thermal stability and abrasion properties. ZnO NPs have effectively enhanced the abrasion resistance of the fabric, demonstrating the potential benefits of nanotechnology in textile applications making it suitable for use as outdoor clothing or durable home textiles.

This technique for creating ZnO nanoparticles had the bnefits of being easy to use, inexpensive, and suitable for text ile applications.

Authors recommend applying incorporation of zinc oxide

nanoparticle to other types of fabrics and investigations should be performed to study the possibility of utilizing the treated fabrics for medical uses or any other applications elsewhere.

Author Contributions: "**Hana:** supervision on practical work, writing the whole draft manuscript; **Amna:** All practical work; **Ragiab:** revised manuscript, interpretation of XRD & TGA; **Fatma:** interpretation of SEM results; **Eeman:** Textile tests; **Aich:** participated in final revision of manuscript. All authors have read and agreed to the published version of the manuscript."

Funding: "This research received no external funding."

Data Availability Statement: "No data were used to support this study."

Conflicts of Interest: "The authors declare that they have no conflict of interest."

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