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Synthesis and Characterization of ZnO Nanoparticles and Their Application on Cotton Fabrics to Obtain Superhydrophobic Surfaces

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Abstract

Zinc oxide nanoparticles were synthesized in a wet chemical manner at two different reaction temperatures 30, 90 ℃ utilizing zinc chloride as precursors in an aqueous medium. Zinc oxide nanoparticles were characterized by scanning electron microscopy (SEM), X-ray analysis (XRD), and infrared spectroscopy (FTIR). X-ray patterns pointed to a Wurtzite structure with an average crystalline size of 19.5 nm and 23 nm for the ZnO NPs synthesis at 30℃ and 90℃, respectively. SEM showed that with increasing temperature, the morphology of Zinc oxide nanoparticles changed from nanowire to spherical structures.To fabricate super hydrophobic cotton fabric, the fabric was immersed in a colloidal solution of ZnO NPs with a concentration of 1% and then modified later with stearic acid. The samples were characterized by scanning electron microscopy (SEM) and contact angle (CA) test. The SEM results showed that the ZnO NPs synthesis at 90℃ created a rough structure on the cotton surface better than that of ZnO synthesis at 30℃. The CA test showed super hydrophobic properties for ZnO@90˚C samples, while ZnO@30˚C samples showed hydrophobic properties only after modified stearic acid.
Different stearic acid concentrations were used for the hydrophobization of the ZnO surface. (CA) results revealed that the fabric treated with ZnO NPs prepared at 90°C and modified with 1% by weight of stearic acid showed a superhydrophobic surface at a contact angle (CA = 154°).

Keywords: Cotton fabric, Reaction temperature, Stearic acid, Superhydrophobic, ZnO NPs.

Introduction

Since the discovery of the self-cleaning property in the lotus leaf, extensive research efforts have been made to imitate this phenomenon known as the "lotus effect" by attempting to fabricate surfaces with contact angles higher than 150˚ and slide angles less than 10˚, which are known as superhydrophobic surfaces[1].In the last two decades, scientists developed various methods to manufacture superhydrophobic surface structures. Most of these methods are based on creating an appropriate roughness on the surface and then reducing the surface energy [2,3]. The surface of the textiles has a micro-roughness caused by the knitting or weaving process and the threads, which are composed of many microfibers, but it is not enough to obtain superhydrophobic[4]. The most familiar techniques to create roughness on fabric are the deposition of nanomaterials such as TiO_2, SiO_2, and ZnO by dip-coating methods, spray-coating, chemical vapor deposition (CVD), sol-gel, and plasma processing techniques[5-9]. Zinc oxide nanoparticles have acquired attention in textile

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finishing processes because of their unique chemical and physical properties, biosafety properties, good stability, and their simple preparation methods with low cost[10-12], further, their multiple functional properties: UV protection, antibacterial, and photolytic self-cleaning properties[13]. Recently several studies used ZnO NPS to develop superhydrophobic properties by creating roughness on the surface of the fabric. Soran et al. offered a method for the fabrication of superhydrophobic cotton textiles using ZnO nano flower/PDMS polymer nanocomposites[14]. Gajanan et al. fabricated superhydrophobic cotton fabric with a contact angle of 152˚ by coating the surface with ZnO nanoparticles and subsequent modification with hexadecyltrimethoxysilane (HDMS) [15]. Superhydrophobic cotton fabric with a contact angle of 155˚ was fabricated by Meng et al. via a two-step: creating roughness on the surface through hydrothermal growth of ZnO nanoparticles and using stearic acid to reduce surface energy the superhydrophobic cotton fabric showed the significant possibility for oil/water separation [16].
This study aimed to produce superhydrophobic cotton fabrics using a simple and low-cost method that can be easily applied in conventional textile finishing processes utilizing a low concentration of ZnO with subsequent modification with stearic acid. The effect of the morphology and size of ZnO NPs and changed stearic acid concentration on the superhydrophobic property of cotton fabrics were studied.

Materials and Methods

$ZnCl_2$, ethanol, H_2O_2, and NaOH (99%) were purchased from (Merck), stearic acid was obtained from Sigma-Aldrich, and 100% cotton fabric twill 2/1 with mass per unit surface 240g/m^2 was purchased from Lattaki Weaving company.

Synthesis of Nano Zinc Oxide

ZnO nanoparticles were prepared at two different temperatures (30, 90)℃ according to the following method: 100ml of 1 M NaOH, and 100 ml of (0.5 M) $ZnCl_2$ were prepared then two solutions were heated under constant stirring until the required temperature was reached, after that the solution was added drop-wise to the NaOH aqueous solution with continuous stirring (the color of the solution $ZnCl_2$ changed from transparent to white). After completing dripping, the prepared solution was aged for 2 hours. The oxide was left to precipitate, then washed five times with distilled water and dried at a temperature of 150˚C for 4 hours. The reactions mechanism is shown in the following: [17]

$$Zn^{+2} + 2OH^- \rightarrow Zn(OH)_2$$
$$Zn(OH)_2 + 2OH^- \rightarrow Zn(OH)_4^{2-}$$
$$Zn(OH)_4^{2-} \rightarrow Zno + H_2O + 2OH^-$$

The prepared zinc oxide samples were named as follows: ZnO90 for ZnO NPs synthesis at 90℃, ZnO30 for ZnO NPs synthesis at 30℃

Cotton Fabric Pretreatment

The purpose of the pretreatment is to remove the added and natural impurities from the cotton fabric to make the fabric suitable for the subsequent finishing process. Therefore, the fabric was boiled in a solution containing 4% NaOH and 8% H_2O_2 for an hour at 100˚C. Then rinsed fabric with water and adjusted with acetic acid, and the fabric was rinsed again with water. Finally, the fabric was left to dry at room temperature.

Deposition Zinc Oxide onto Fabric

A pad-dry-cure technique is used to deposit ZnO NPs onto fabric according to the following steps: First, a colloidal suspension of each ZnO NPs was prepared at a concentration of (1%) by dispersing ZnO powder into ethanol by ultrasound for 30 minutes. Second, the cotton fabric was immersed in the colloidal suspension for 30 min in the ultrasonic bath, then the fabric was squeezed . Finally, the fabric was dried at 80˚C for 10min, then cured at 110˚C for 10min.

Surface Modification by Stearic Acid

Different concentrations of stearic acid solution (0.5,1,3,5,7%) were prepared by dissolving stearic acid in ethanol at 70˚C, then immersing the (ZnO NPs cotton fabric) and pristine cotton samples in the stearic acid solutions for 10 minutes, then squeezing the samples and finally drying samples at room temperature.

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Characterization

The crystalline structure of the prepared ZnO nanoparticles powder was investigated by the X-ray diffraction patterns obtained using a PHILIPS XRD-PW 1840 Diffractometer using Co-Kα_1 radiation at a wavelength of λ =1.78901 Å. Fourier transforms the infrared spectrometer (Jasco FTIR/4100) at the wavenumber region of 4000–400 cm^{-1} to determine the functional groups of the synthesized ZnO NPs. The morphology of the ZnO NPs was investigated by using Scanning Electron Microscopic (VEGA II XMU). To evaluate the wettability of samples, the water contact angle (CA) was measured using a sessile drop method[18]. A drop (5 µl) of deionized water was set on the surface. The droplet image is taken using a smartphone and processed using the Image J program to measure the CA. Three measurements at different positions on the surface of the sample were made.

Results and Discussion

X-ray Diffraction (XRD)

Fig. 1. shows the X-ray diffraction spectra of ZnO NPs prepared at two different temperatures (30˚-90˚ C) in the range (2Θ= 35-89). It is noted that all the diffraction peaks in the XRD pattern of the ZnO NPs are identical and correspond to the crystal planes found in the reference card (JCPDS 01-079-0205) which indicates ZnO NPs have a hexagonal structure (wurtzite hexagonal). The sharp and narrow peaks indicate that ZnO NPs samples had good crystallinity. ZnO NPs powder was high purity because there are no peaks indicating impurities. The crystal size of the zinc oxide particles was calculated using Debye-Scherer Equation Eq. 1:

$$D = \frac{0.9\,\lambda}{\beta \cos\theta} \ldots\ldots\ldots 1$$

Where λ: wavelength of the X-ray (1.78901 Å), β: full width at half maximum peak (radians), and θ: Bragg angle (degree). The average crystalline size of the ZnO Nanopowder was around 19.5 nm at 30℃, which increased to 23 nm when the temperature reaction was raised to 90℃.

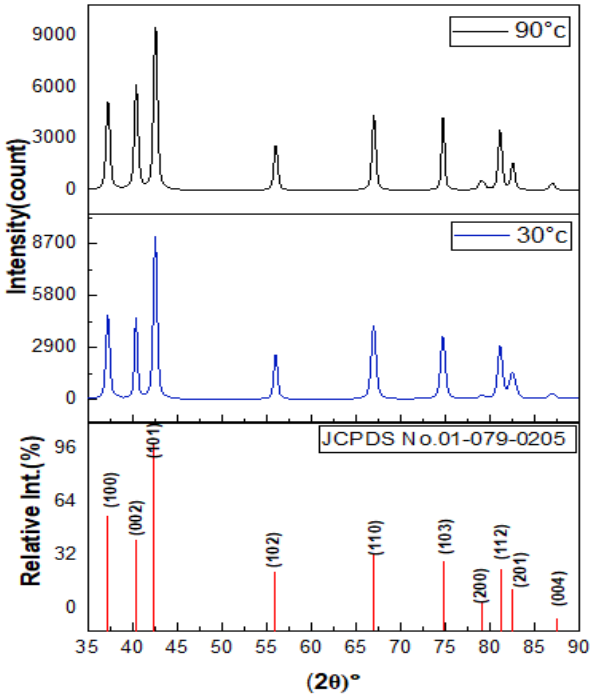

Figure 1. XRD patterns of ZnO synthesis under different temperatures

Fourier Transform Infrared Spectroscopy (FT-IR)

FT-IR was used to find out the functional groups in the prepared ZnO. Fig. 2 shows the infrared transmittance spectrum of zinc oxide prepared at two different temperatures (30°-90°C). The peaks at 3433 cm^{-1} and 1633 cm^{-1} indicate the stretching-bond vibration of the O-H, and the bending vibration of the O-H, respectively. The absorption peaks located in the range between 417-517 cm^{-1} indicate the stretching vibration of the (Zn–O) bond. It is also noted that the Zn-O absorption peaks became sharper with smooth edges, with increasing

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temperature indicating an increase in the crystallization of nanoparticles[19]. These results demonstrate the success of the synthesis of ZnO NP. The change in the shape and position of the IR peaks can be attributed to the structure and size of the particles[20, 21].

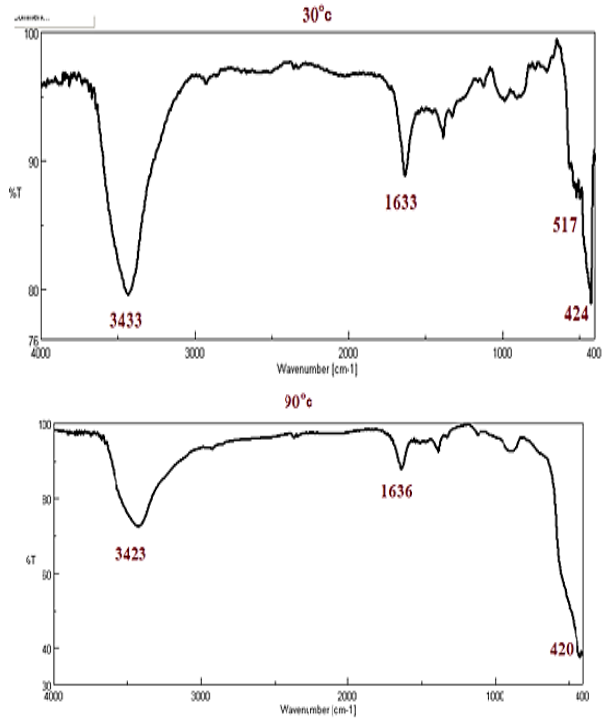

Figure 2. FTIR spectrum of the synthesis ZnO NPs

Scanning Electron Microscope (SEM)

Scanning electron microscopy was used to characterize the morphological structure of zinc oxide nanoparticles prepared at two different temperatures (30 and 90℃) and to descry the difference in the surface morphology before and after treatment with both types of zinc oxide. The SEM images indicate two different types of morphology according to the reaction temperature during the ZnO NPs synthesis. Fig. 3a shows a ZnO nanowire with an average diameter of 30 nm and an average length of 4µm. With the increase in temperature up to 90℃, Fig. 3b, the particles had a spherical shape with an average diameter (41 nm). The microstructure of the cotton fabric before and after treatment with ZnO NPs also is shown in Fig. 3`. The untreated cotton fabric had smooth surfaces, Fig. 3c, with no solid particles observed. On the other hand, the treated cotton fabrics showed solid white precipitation distributed on the surface, and it was noticed that the spherical shape of the Zinc oxide nanoparticles, Fig. 3(d, d1), provides a roughness on the surface better than ZnO nanowires Fig. 3(e, e1).

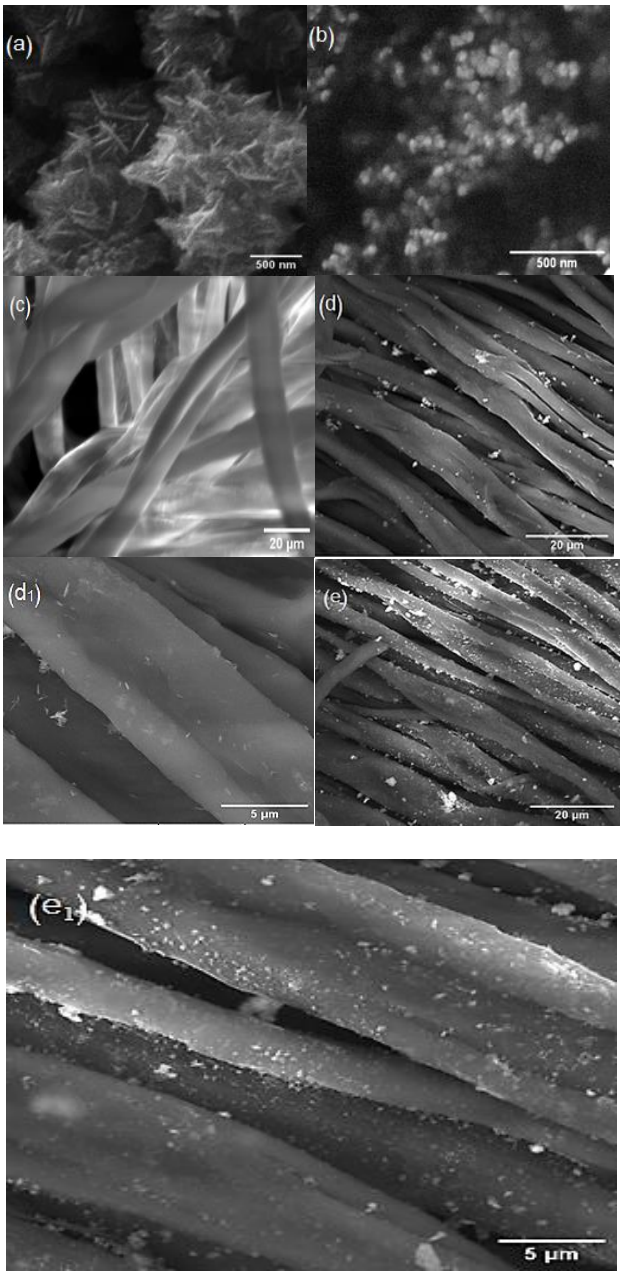

Figure 3. SEM of the ZnO NPs synthesis. (a) 30˚C, (b) 90˚C, (c) untreated cotton, (d, d1) ZnO30 treated cotton fabric, (e, e1) ZnO90 treated cotton fabric

Contact Angle Measurement

Contact angle measurement is a qualitative method of evaluating if a surface is hydrophilic or hydrophobic. For comparison, the contact angle was

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measured for cotton fabric as follows: untreated, treated with ZnO NPs, modified by stearic acid, and finally, fabric treated with zinc oxide modified by stearic acid later.

The untreated cotton fabric showed super hydrophilic behavior (CA=0˚) as the water droplet spread directly on the surface and was rapidly absorbed. Also, cotton fabric treated with zinc oxide showed hydrophilic behavior (CA=43˚). This indicates that the deposition of nanoparticles on cotton fabric does not change its hydrophilic property to hydrophobic this is due to the presence of hydroxyl (OH) on the ZnO NPs surface; on the other hand, the cotton fabric turned hydrophobic by modification by stearic acid with(CA=124˚), but this treatment was not sufficient to obtain a super hydrophobic property. The contact angle increased significantly (WCA> 140˚) after the cotton fabric treatment with ZNPs and modified by stearic acid. This increase in CA is to the combination of roughness formation on the surface of the fabric by ZnO NPs and reducing surface energy by stearic acid. These steps are essential for obtaining superhydrophobic surfaces. The following reaction can express the mechanism of modified zinc oxide nanoparticles by stearic acid [22]:

$$HO-ZnO-OH+2CH_3(CH_2)_{16}COOH \rightarrow CH_3(CH_2)_{16}COO-Zno-OOC(CH_2)_{16}CH_3+2H_2O$$

The effects of changing the content of stearic acid on the hydrophobic properties of ZnO NPs coated cotton fabric with different morphologies were studied. Therefore, different concentrations of stearic acid (0.5, 1, 3, 5, and 7 wt%) were used. Figure 4 shows the water contact angle results. As Can see from Fig. 4, the quantity of stearic acid markedly influenced the hydrophobic properties of cotton fabric; the contact angle increased at first, then decreased with the increase of stearic acid content. When the concentration of stearic acid was 1wt%, ZnO NPs-coated cotton fabric showed superhydrophobic properties. It is due to the full coverage of the ZnO NPs with the mono-layer of stearate molecules with their hydrophobic tails. On the other hand, any increase in the stearic acid concentration after 1wt% decreases the CA. This is due to the formation of the thick stearic-acid layer on the ZnO surface, which reduces the roughness effect of the ZnO NPs; In addition, the excess carboxylic groups of stearic acid are deposited on the surface, leading to a decrease in CA [23].

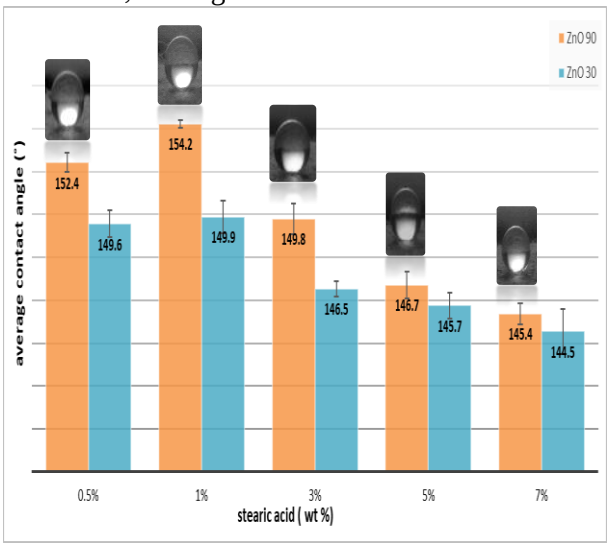

Figure 4. Effect of stearic acid on contact angle

The effect of the changing morphology of ZnO NPs on the superhydrophobic property of cotton fabrics was studied by comparing the contact angle results of cotton fabric treated with the two different morphology of ZnO NPs and modified with stearic acid. It was established that the cotton samples treated with ZnO90 showed superhydrophobic properties, with CA > 150. while cotton Samples treated with ZnO30 showed hydrophobic properties with CA <150 because ZnO90 with spherical morphology created a rough structure on the cotton surface better than that of ZnO30.

Conclusion

In summary, ZnO NPs were prepared at different reaction temperatures by a simple wet chemical method. The synthesized ZnO NPs were characterized by (XRD), (FTIR), and (SEM). The SEM results confirmed that reaction temperature influences the morphologic structure of ZnO NPs. FTIR results confirmed forming of Zn–O bond. XRD analysis confirmed that ZnO NPs samples had good crystallinity and a hexagonal structure (wurtzite). To fabricate superhydrophobic cotton surfaces, the cotton fabric was coated with 1% wt of synthesis zinc oxide nanoparticles and modified by different concentrations of stearic acid. Water contact angle (CA)and scanning electron

microscopy (SEM) characterized the treated cotton fabric. According to the results, samples treated with ZnO90 showed superhydrophobic, while samples treated with ZnO30 showed only hydrophobic properties after stearic acid modification.

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Authors' Declaration

- Conflicts of Interest: None.
- We hereby confirm that all the Figures and Tables in the manuscript are ours. Furthermore, any Figures and images, that are not ours, have been included with the necessary permission for re-publication, which is attached to the manuscript.
- Ethical Clearance: The project was approved by the local ethical committee at Al-Baath University.

Authors' Contribution Statement

M. I. contributed to the design and implementation of the research, analysis of the results, interpretation, and writing the manuscript. Z.S. contributed to the analysis of the results and proofreading of the manuscript.

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تحضير وتوصيف جسيمات أوكسيد الزنك النانوي وتطبيقها على الأقمشة القطنية للحصول على سطوح فائقة الكره للماء

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الخلاصة

حضرت جسيمات أوكسيد الزنك النانوية ZnO (NPs) بطريقة كيميائية رطبة عند درجتي حرارة مختلفتين ℃(30-90) باستخدام كلوريد الزنك كبادئ في وسط مائي. تم توصيف جسيمات أوكسيد الزنك النانوي المحضر باستخدام المجهر الإلكتروني الماسح (SEM), حيود الأشعة السينية (XRD) ، وطيف الأشعة تحت الحمراء (FTIR) ، أشارت أنماط الأشعة السينية الى بنية سداسية (wurtizte) بمتوسط حجم بلوري (19.5-23nm) لجسيمات أوكسيد الزنك المحضرة عند درجتي الحرارة (30-90℃)على التوالي. أظهرت صور المجهر الالكتروني الماسح أنه مع زيادة درجة الحرارة ، تغير شكل جسيمات أوكسيد الزنك النانوي من الأسلاك النانوية إلى االشكل الكروي . لتصنيع قماش قطني فائق المقاومة للماء. تم غمر القماش في محلول غروواني لأوكسيد الزنك بتركيز (1٪) ,ثم تعديله لاحقًا بحمض الشمع . تم توصيف القماش القطني المعالج باستخدام المجهر الإلكتروني الماسح (SEM) واختبار زاوية التماس (CA) .أظهرت نتائج المجهر الالكتروني الماسح أن جسيمات أوكسيد الزنك النانوي المحضر عند(90℃) خلق خشونة على سطح القطن أفضل من جسيمات أكسيد الزنك النانوي المحضرعند (30℃). أظهرت نتائج اختبار زاوية التماس CA خصائص فائقة الكره للماء لعينات أوكسيد الزنك النانوي المحضر عند(90℃) ، بينما أظهرت عينات أوكسيد الزنك النانوي المحضر عند(30℃) خصائص كارهة للماء فقط بعد التعديل بحمض الشمع. تم استخدام تراكيز مختلفة من حمض الشمع لجعل سطح أوكسيد الزنك كارهاً للماء. أظهرت نتائج زاوية التماس (CA) أن القماش القطني المعالج بأوكسيد الزنك النانوي المحضر عند الدرجة (90℃) المعدل ب 1٪ وزناً من حمض الشمع أظهر سطحاً فائق الكره للماء بزاوية تماس (CA=154°).

الكلمات المفتاحية: قماش قطني، درجة حرارة التفاعل, حمض الشمع ، فائق الكره للماء ، أوكسيد الزنك النانوي.