



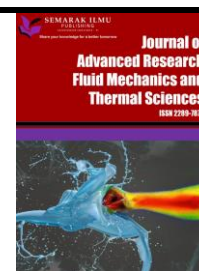
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# Effects of Thermal Annealing on The Morphology and Structural Characteristics of Zinc Oxide Nanopowders for Triboelectric Nanogenerator Applications

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### ABSTRACT

The influence of thermal annealing on the surface morphologies and structural characteristics of zinc oxide (ZnO) nanopowders synthesized via the solution immersion method for triboelectric nanogenerator applications is reported in this paper. The ZnO nanopowders were thermally treated at different temperatures of annealing in the ranges between 300°C to 700°C for 1 hour, and their surface morphologies and structural properties were studied using field emission scanning electron microscopy (FESEM) and X-ray diffraction (XRD) analysis. The ZnO nanopowders have a polycrystalline, hexagonal wurtzite structure and are composed of ZnO nanoparticles and hexagonal nanorods. ZnO based triboelectric nanogenerators were fabricated with these nanopowders and their performance was assessed in terms of the output voltage. It is found that the ZnO based triboelectric nanogenerator fabricated with ZnO nanopowders annealed at 500°C has superior performance compared with the other nanogenerators, with an average output voltage of 1.95 V. This corresponds to a fourfold increase in output voltage relative to that of the ZnO based triboelectric nanogenerator fabricated with as-deposited ZnO nanopowders. In conclusion, thermal annealing significantly influences particle size and

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**Keywords:** crystallinity of ZnO nanopowders, which in turn, influences the output voltage of ZnO based triboelectric nanogenerators.  
Triboelectricity; nanogenerator; zinc oxide; immersion

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## 1. Introduction

With advancements in information technology (Internet of Things, big data) and telecommunications (fifth generation (5G) technology), electronics are becoming smaller and smaller, hence increasing the demand for clean, sustainable, and reliable power sources [1,2]. Metal oxide nanostructures have attracted a great deal of interest owing to their superior and unique properties, which make them appropriate for advanced technological applications, including sensors, solar cells, batteries, and nanogenerators [3-11]. Nanogenerators have gained a significant amount of attention over the past two decades, as this device can generate electricity without the usage of batteries by harvesting mechanical energy from the surrounding environment [12,13]. Associated with the high demand for clean and affordable energy, a low cost, a facile synthesis, and excellent triboelectric nanogenerator (TENG) performance are required. The research community has taken a keen interest in it because of their potential as a clean, highly efficient, and sustainable power source. Typically, zinc oxide (ZnO), lead zirconium titanate (PZT), barium titanate ( $\text{BaTiO}_3$ ), Polyvinylidene Fluoride Polymer Film (PVDF) and polydimethylsiloxane (PDMS) are utilized to fabricate [14-18]. ZnO has excellent potential compared with other materials because it possesses a direct wide band gap (3.37eV), capability to grow various nanostructures, ease of synthesis, environmental friendliness and piezoelectric properties [19-24]. ZnO is one of the metal oxides extensively studied for many applications ranging from electronics, energy conversion or storage, textile, nanofluids, and medical applications which make it appropriate for many applications [25-30].

Several common techniques including chemical vapour deposition (CVD), pulsed laser deposition (PLD), hydrothermal, magnetron sputtering and sol-gel techniques, have been utilized to produce high-quality ZnO nanostructures [31-37]. All these methods depend on the properly controlled synthesis parameters for the high-quality growth of ZnO nanostructures. To fulfill the rising demands for flexible, scalable, and low-temperature device production, a simple solution immersion method through low temperature chemical reaction can be appropriately adopted. However, it has been reported that the growth of ZnO nanostructures at low temperatures ( $<100^\circ\text{C}$ ) produces issues with the stability, dispersion and crystalline structures control of ZnO nanoparticles in aqueous solution bath due to the low kinetics or total suppression of the chemical reactions [38,39]. This could be a significant problem for the output performance of nanogenerators. The synthesis of ZnO nanostructures relies on both the growth and the thermal annealing processes. Thermal annealing is a widely used technique to apply energy to the material and improve the crystalline quality by reducing the defects in the material [40-42]. Some comparative studies on the effects of annealing temperature on the properties of ZnO nanostructures are reported. For example, the work of Malek *et al.*, [43] discovered that thermal annealing in the range  $300^\circ\text{C}$  to  $600^\circ\text{C}$  greatly influenced the surface and structural properties of ZnO nanoparticles prepared by sonicated sol-gel dip coating technique, whereas Sahdan *et al.*, [44] found the surface morphology of ZnO nanostructures deposited using chemical vapor deposition (CVD) could be significantly affected by the annealing temperature ranging between  $100$ - $300^\circ\text{C}$ . Hence, thermal annealing is a crucial component in determining the surface morphologies and structural properties of ZnO nanostructures [45]. Meanwhile, the output performance of ZnO based TENG has significant influences by the structural as well as surface morphology properties of the material. This factor facilitates the charge transfer mechanism by relating to the surface charge density. A larger specific surface area can significantly

enhance the charge transfer density after contact and separation process, resulting in a higher current [46].

Since the output performance of ZnO base TENG varies depending on the surface morphologies of nanostructures, it is essential to explore the surface morphology and structural properties of ZnO utilizing a simple solution immersion method for ZnO based TENG applications. In this work, the surface morphologies and structural characteristics of ZnO nanopowders induced by thermal annealing were studied. The ZnO nanopowders were synthesized by the immersion method. Furthermore, six ZnO based TENG were fabricated with these nanopowders to evaluate the nanogenerator performance in terms of output voltage.

## 2. Methodology

The solution immersion method was used to synthesize the ZnO nanopowders. Zinc nitrate hexahydrate ( $Zn(NO_3)_2 \cdot 6H_2O$ ), hexamethylenetetramine (HMT,  $C_6H_{12}N_4$ ), and deionized (DI) water were used as the starting materials to synthesize the ZnO nanopowders. An ultrasonic water bath was used to sonicate the ZnO solution. Following this, the ZnO solution was poured into Schott bottles, which were then immersed in a  $95^\circ C$  water bath for 4 hours. The synthesized ZnO nanopowders were then filtered and dried.

The ZnO nanopowders were annealed at various temperatures ( $300^\circ C$ ,  $400^\circ C$ ,  $500^\circ C$ ,  $600^\circ C$  and  $700^\circ C$ ), where the samples were placed in a furnace box for 1 hour. Next, the surface morphologies of the prepared ZnO nanopowders were examined using a field emission scanning electron microscope (FESEM, JOEL JSM-6320F) at 5kV accelerating voltage, while the structural properties of ZnO nanopowders were assessed using an X-ray diffraction (XRD, PANalytical X'Pert PRO) with Cu K-alpha radiation corresponding to an X-ray wavelength of  $1.54 \text{ \AA}$ . The X-ray diffractograms were recorded at a diffraction angle ( $2\theta$ ) of  $20^\circ$ – $90^\circ$ .

To fabricate the ZnO based triboelectric nanogenerators, each of the ZnO nanostructured powders was mixed with polystyrene and toluene solution to form the ZnO layer. The bottom triboelectric layer consisted of ZnO layer and silver (Ag) electrode. Using thermal evaporation, the Ag electrode was deposited onto the ZnO layer. The upper triboelectric layer consisted of aluminium (Al) foil as the electrode and Kapton tape as the dielectric layer. A total of six ZnO triboelectric nanogenerators was fabricated — one was prepared using the as-deposited ZnO nanopowder while the others were prepared using ZnO nanopowders annealed at different temperatures. The basic structure of the ZnO triboelectric nanogenerators fabricated in this work is illustrated in Figure 1. The output voltage values of the ZnO triboelectric nanogenerators in response to repetitive finger tapping were recorded using an oscilloscope (Model: RTC 1002, Rohde & Schwarz USA Inc., USA).

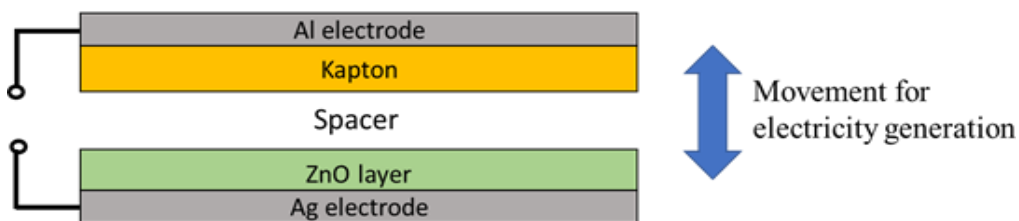


Fig. 1. Basic structure of the ZnO triboelectric nanogenerators fabricated in this work

### 3. Results

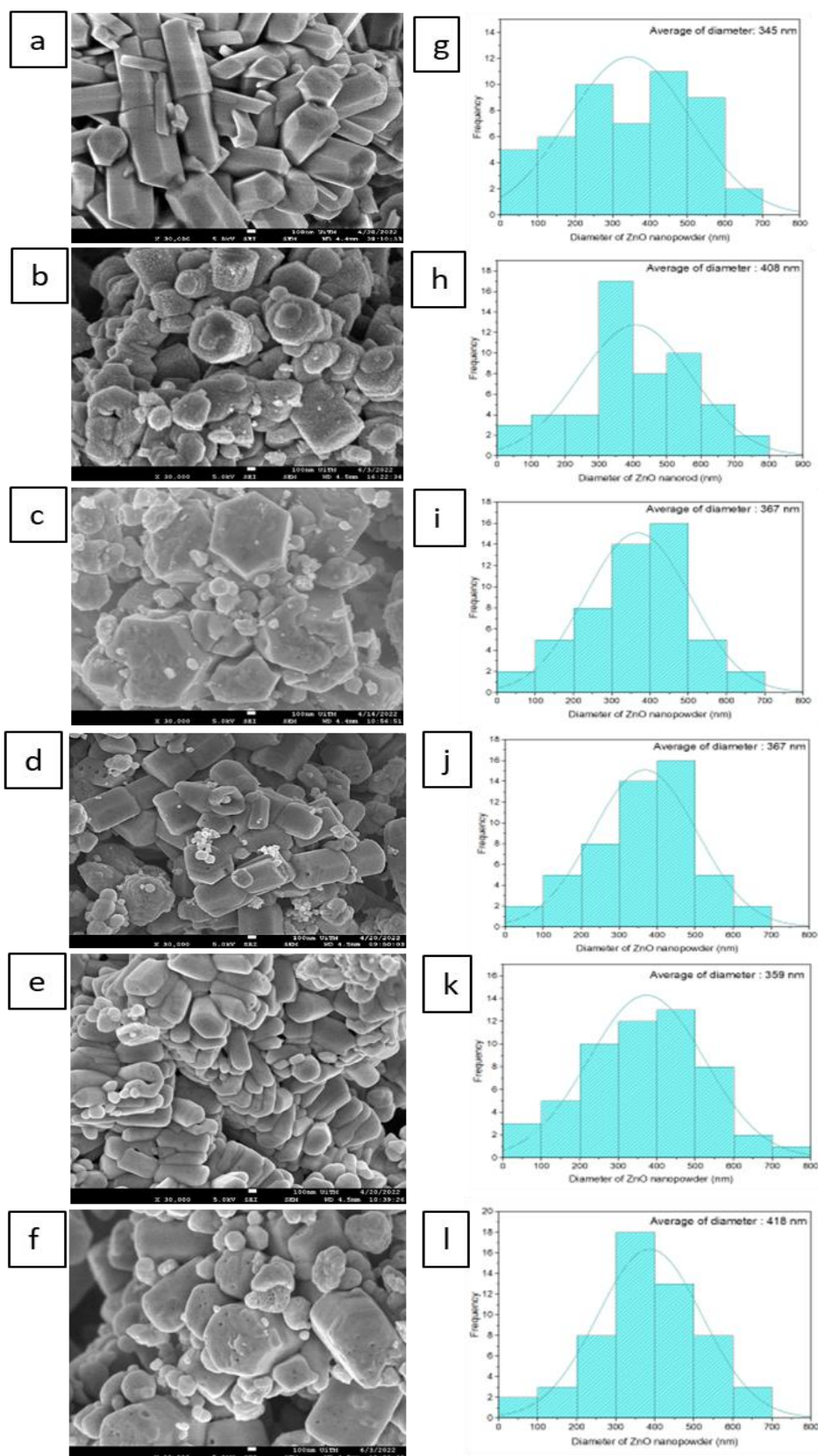
#### 3.1 Surface Morphology

Figure 2(a)-(f) show the surface morphologies of as-deposited ZnO nanopowder and ZnO nanopowders annealed at various temperatures. It can be observed that there is a random growth of ZnO nanoparticles and hexagonal nanorods for all samples. The particle size distributions for all samples were determined using ImageJ software, and the results are shown in Figure 2(g) to Figure 2(l). The average diameters for all ZnO nanopowders are summarized in Table 1. The as-deposited ZnO nanopowder has an average diameter of 345nm. The average diameter of the ZnO nanopowder increases from 408nm to 451nm when the annealing temperature is raised from 300°C to 400°C. This can be due to thermal expansion, in which the kinetic energy of the atoms increases as the annealing temperature rises [47]. But the average diameter of the ZnO nanopowder decreases to 367nm at 500°C and 359nm at 600°C. Atoms with sufficient thermal energy can move, resulting in the rearrangement of the atoms in the crystalline structure. Thus, ZnO nanoparticles notice a decrease in diameter. It is suggested that the crystalline quality enhances as the atoms are free to move to a more favourable position [44]. At 700°C, the atoms move closer together and consequently, the ZnO nanoparticles merge with the adjacent ZnO nanoparticles to form larger nanoparticles which is known as agglomeration. Agglomeration of the ZnO nanoparticles during thermal annealing may be due to the high surface energy of the nanoparticles. Thus, it can be concluded that the annealing temperatures have an impact on the ZnO nanopowder's average diameter.

**Table 1**

Average diameter of the as-deposited ZnO nanopowder and ZnO nanopowders at various annealing temperatures

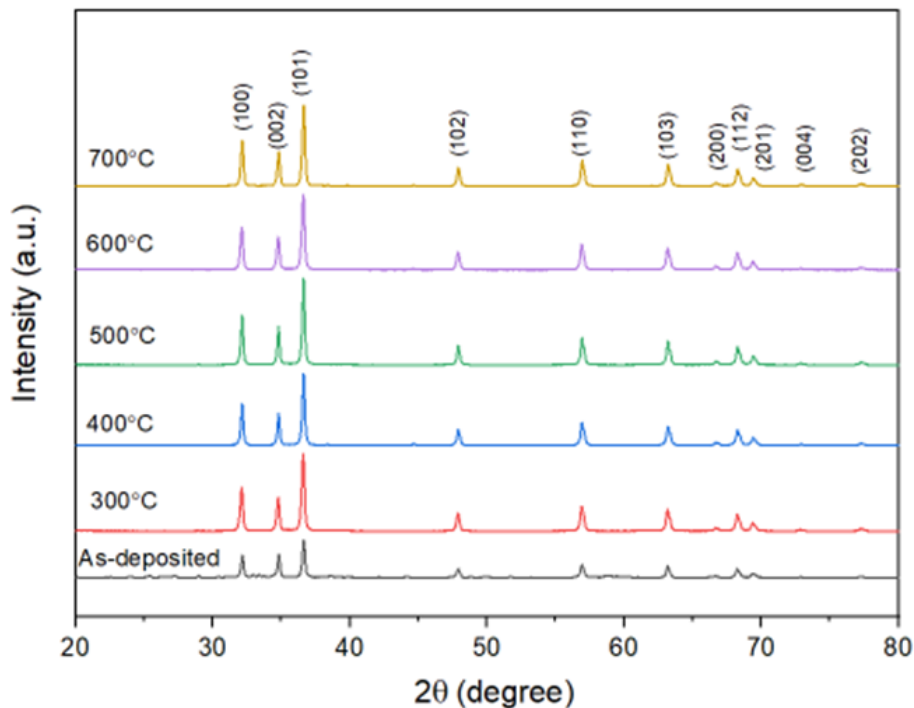
Samples	Average diameter (nm)
As-deposited ZnO nanopowder	345
ZnO nanopowder annealed at 300°C	408
ZnO nanopowder annealed at 400°C	451
ZnO nanopowder annealed at 500°C	367
ZnO nanopowder annealed at 600°C	359
ZnO nanopowder annealed at 700°C	418



**Fig. 1.** FESEM images of the (a) as-deposited ZnO nanopowder and ZnO nanopowders at different annealing temperatures (b) 300°C, (c) 400°C, (d) 500°C, (e) 600°C, and (f) 700°C. Particle size distributions of the (g) as-deposited ZnO nanopowder and ZnO nanopowders at different annealing temperatures (h) 300°C, (i) 40 °C, (j) 500°C, (k) 600°C, and (l) 700°C

### 3.2 Structural Properties

The X-ray diffractograms and crystalline orientation of the as-deposited and thermally annealed ZnO nanopowders are shown in Figure 3. The diffraction peaks of all ZnO nanopowders match well with those of the JCPDS card no. 075-1526, indicating that the ZnO nanopowders have a polycrystalline, hexagonal wurtzite structure. For each sample, three significant ZnO diffraction peaks correspond to the (100), (002), and (101) planes were observed. The (102), (110), (103), (200), (112), (201), (004), and (202) planes correspond to the remaining diffraction peaks. The other diffraction peaks correspond to the (102), (110), (103), (200), (112), (201), (004), and (202) planes. There are no diffraction peaks of noticeable impurities in Figure 3, indicating the high purity of ZnO nanopowders synthesized by the solution immersion method. All the ZnO nanopowders have the same structure, with almost no impurities. The as-deposited ZnO nanopowder has weak diffraction peaks, indicating poor crystalline quality. It is evident that the diffraction peak intensities of the as-deposited ZnO nanopowder increase with the annealing temperature treatment. With an increase in temperature, the high intensity of the diffraction peak suggests an increase in the crystallinity of the ZnO nanopowder [40]. The good crystallinity of the produced ZnO nanopowders is indicated by the strong diffraction peaks.



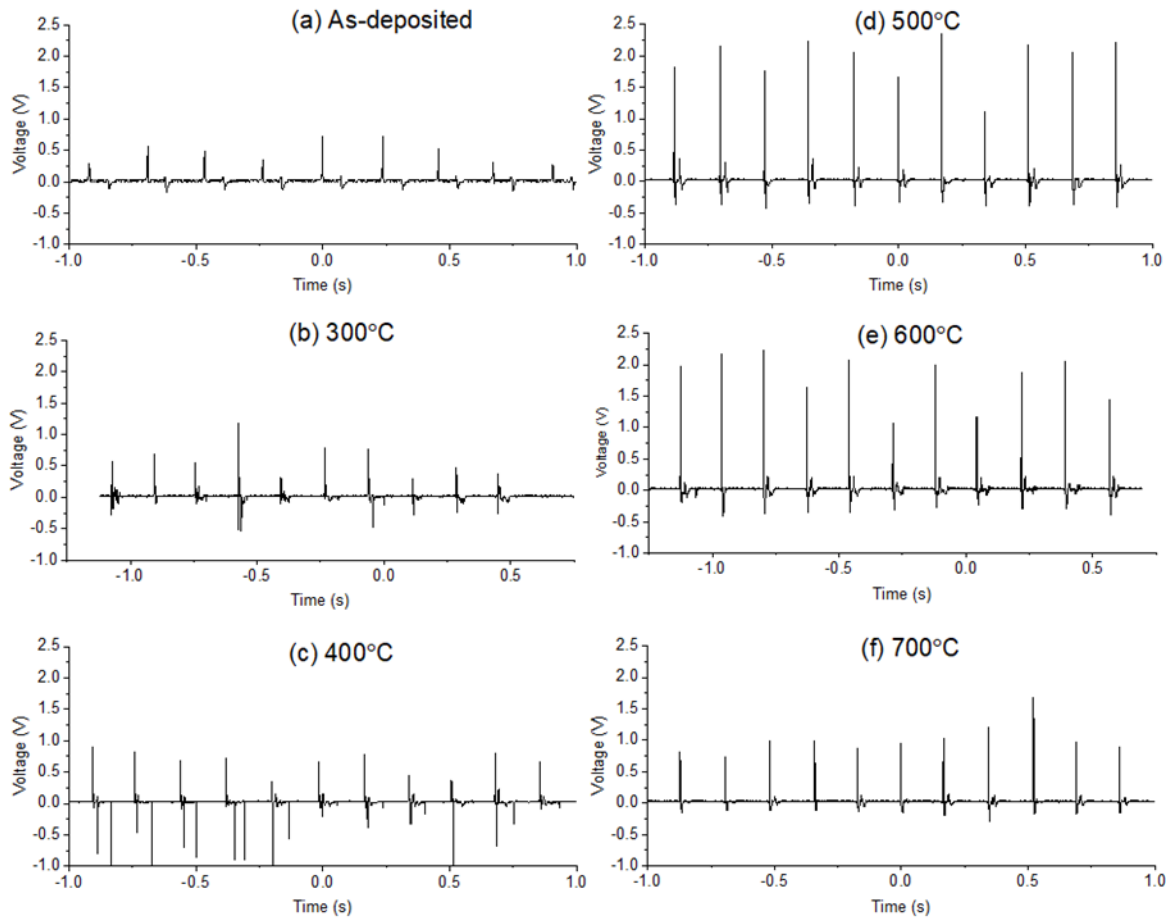
**Fig. 3.** X-ray diffractograms of the synthesized ZnO nanopowders annealed at various temperatures

### 3.3 Performance of the ZnO Based Triboelectric Nanogenerators

After examining the surface morphologies and structural characteristics of both as-deposited ZnO nanopowder and ZnO nanopowders annealed at 300°C, 400°C, 500°C, 600°C, and 700°C, ZnO based triboelectric nanogenerators were fabricated with these nanopowders and their performance was investigated. Figure 4(a)-(e) show the output voltage values of the ZnO based triboelectric nanogenerators. In general, the ZnO based triboelectric nanogenerators fabricated with thermally annealed ZnO nanopowders have a higher average output voltage compared with the ZnO



triboelectric nanogenerator fabricated with as-deposited ZnO nanopowder, as shown in Table 2. It can be indicated that thermal annealing enhances the output voltage of the ZnO based triboelectric nanogenerators, where the ZnO layer was synthesized by the solution immersion method.



**Fig. 4.** Output voltage values of the ZnO based triboelectric nanogenerators fabricated with (a) as-deposited ZnO nanopowder and ZnO nanopowders at different annealing temperatures (b) 300°C, (c) 400°C, (d) 500°C, (e) 600°C, and (f) 700°C

**Table 2**

Average output voltage of the ZnO based triboelectric nanogenerators fabricated with different ZnO nanopowders

Samples	Average output voltage (V)
As-deposited ZnO nanopowder	0.47
ZnO nanopowder annealed at 300°C	0.59
ZnO nanopowder annealed at 400°C	0.61
ZnO nanopowder annealed at 500°C	1.95
ZnO nanopowder annealed at 600°C	1.71
ZnO nanopowder annealed at 700°C	0.99

Figure 4(a) shows that the average output voltage is 0.47 V for the ZnO based triboelectric nanogenerator fabricated with as-deposited ZnO nanopowder. Interestingly, increasing the thermal annealing temperature from 300°C to 500°C increases the average output voltage of the device to 1.95 V, which is roughly a fourfold increase over the average output voltage of the ZnO based

triboelectric nanogenerator fabricated with as-deposited ZnO nanopowder. The ZnO based triboelectric nanogenerator fabricated with ZnO nanopowder annealed at 500°C shows superior performance compared with the other ZnO based triboelectric nanogenerators. This is possibly due to the enhanced surface charge associated with the small particle size and good crystallinity produced by thermal annealing [48]. The reduction in the average output voltage of the device as the thermal annealing temperature is raised to 700°C may be due to agglomeration of the ZnO nanopowder. The results indicate that the ZnO has adequate annealing energy at an annealing temperature of 500°C, resulting in the superior performance of the ZnO based triboelectric nanogenerator.

#### 4. Conclusions

ZnO nanopowders were successfully synthesized by the solution immersion technique, which is an easy, cheap, and effective synthesis route. The surface morphologies and structural characteristics of the ZnO nanopowders were examined in relation to the impact of annealing temperature. The ZnO nanopowders are formed of ZnO nanoparticles and hexagonal nanorods, according to the FESEM images. The annealing temperature has been found to significantly impact the microstructure of ZnO. The X-ray diffractograms confirmed the presence of ZnO nanoparticles. It is found that thermal annealing produces the ZnO nanopowders with high crystallinity. ZnO based triboelectric nanogenerators were fabricated with the synthesized nanopowders, and it is found that the annealed ZnO nanogenerator have a higher output voltage than as-deposited ZnO nanogenerator. The ZnO based triboelectric nanogenerator fabricated with ZnO nanopowder annealed at 500°C shows superior performance compared with the other ZnO based triboelectric nanogenerators, with an average output voltage of 1.95 V. Thus, it can be concluded that the surface morphologies and structural characteristics of ZnO nanopowders are vital factors in determining the performance of ZnO-based triboelectric nanogenerators. The findings of this work provide insight into how annealing temperature affects the synthesized ZnO nanopowders, which in turn affects the performance of ZnO based triboelectric nanogenerators.

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