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ORIGINAL ARTICLE

Morphological changes of ZnO nanostructures upon addition of Tannic Acid

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ABSTRACT – Due to their potential as a good semiconductor, Zinc Oxide nanostructures (ZnO) have received abundant of interest. In this analysis, ZnO nanostructures are synthesized by a hydrothermal process that uses a green method of synthesis completely aided by Tannic Acid (TA). The mean diameter of ZnO nanostructures are observed to increase with addition of TA due to the aggregation that occurred from the influenced of acidic medium. The morphological properties are discussed based on the TEM images which indicated the average size of 8nm for ZnO NPs and 18-23nm for ZnO-TA nanostructures obtained, respectively.

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Zinc Oxide; Tannic acid; green synthesis; hydrothermal method; TEM

INTRODUCTION

ZnO is a flexible semiconducting material which has received wide attention in the scientific community due to its use in a wide range of technologically relevant areas. It has wide energy from the direct bandgap (about 3.2-3.37 eV) and strong energy from the exciton binding (60 meV). These characteristics allow ZnO an essential material for optoelectronic and nanoelectronic short wavelength applications [1, 2]. The small size of the ZnO nanostructures (average diameter < 100nm) would influence wide band gap character development. Also, few previously reported studies have identified the biocompatible capacity of ZnO NPs that can be applied together to inhibit microbial growth without coating and are indexed as a generally recognised as safe material by the U.S. Food and Drug Administration [3-7].

The most common synthesising nanoparticles method is wet chemical, hydrothermal, sol-gel and green synthesis method [8-12]. Herein, the hydrothermal process using green reduction chemicals, Tannic Acid (TA), was used for the synthesis of ZnO NPs. Due to its cost-effective and ecologically advantageous method of synthesising nanomaterials, the combination of both hydrothermal and green synthesis methods is well known by many studies [13-16]. Essentially, the approach to green synthesis is to manipulate natural resource reagents such as glucose, plant extracts and biodegradable polymers as capping agents[17]. In this research work, TA is used as both the capping agent and second size influencer as it has been proved to influence the synthesis of metal size [13, 18]. This study aimed to synthesise smaller size (average diameter < 20 nm) of ZnO nanostructures with the aid of TA as both the size influencer and capping agent. The smaller the size of ZnO Nanostructures obtained, the better the optical character of the nanostructures.

MATERIALS AND CHARACTERISATION

Materials: All materials were purchased from the commercial market with high purity (95%) and used without further purification. Zinc powder (Zn) as the starting materials, Tannic Acid ($C_{76}H_{52}O_{46}$) as a capping agent and distilled water as dispersing solvent were used to prepare ZnO nanoparticle.

Preparation of ZnO nanoparticles: 0.4g of ZnO powder is diluted in 100ml of distilled water in a beaker. The mixture is then placed on a heated plate and stirred continuously with a heated temperature of 70˚C for 1 hour and 30 minutes. Upon the process, the beaker is then left cooled in the open air, which helps in terminating the regrowth of ZnO NPs. The mixture of the pure ZnO solution is then rinse with distilled water and centrifuged to evacuate other impurities. The process of rinsing and centrifuging are repeated for three times.The mixture is then separated into three sample vials with a volume of 3 ml which one of the samples remains pure and another two samples are then added with 0.1 and 0.4 M tannic acid accordingly before shaken well. Details of each sample are as shown in **Table 1.**

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Table 1: Summary of the parameters used in the preparation of each sample

Sample	Volume of solvent (ml)	Mass of ZnO (g)	Concentration of TA (M)
1	100	0.4	-
2	100	0.4	0.1
3	100	0.4	0.4

Characterisation methods: The morphological structure of the synthesised ZnO and ZnO-TA nanostructures is characterised by the transmission electron microscope (TEM) to monitor the nanostructures' size. The size of the sample are measured by using Gatan Micrsoscopic Suite Software whicha are directly used after monitoring on to the sample at TEM.

RESULT AND DISCUSSION

Morphological Structure

The TEM images of as- synthesised ZnO nanostructure without the addition of TA is shown in **Figure 1**. The size distribution is plotted from the figure, which can be observed that the ZnO nanostructure has a tiny size with an average diameter of 8 nm. From Figures 2 and 3, the TEM image of ZnO nanostructure with TA's addition with 0.1 and 0.4M respectively. The average diameter of the ZnO-TA nanostructure is 18 and 23 nm, respectively. As noticed, the cumulative distribution of the pure ZnO nanostructure showing steeper gradient, which indicates the presence of narrower size distribution of nanostructure. Thus, we can observe that the range size for pure ZnO nanostructure is smaller than ZnO-TA nanostructure. This is believe happened due to aggregation that occurred. The presence of TA generally enhanced the aggregation to occur, resulting in a larger diameter of the ZnO nanostructure.

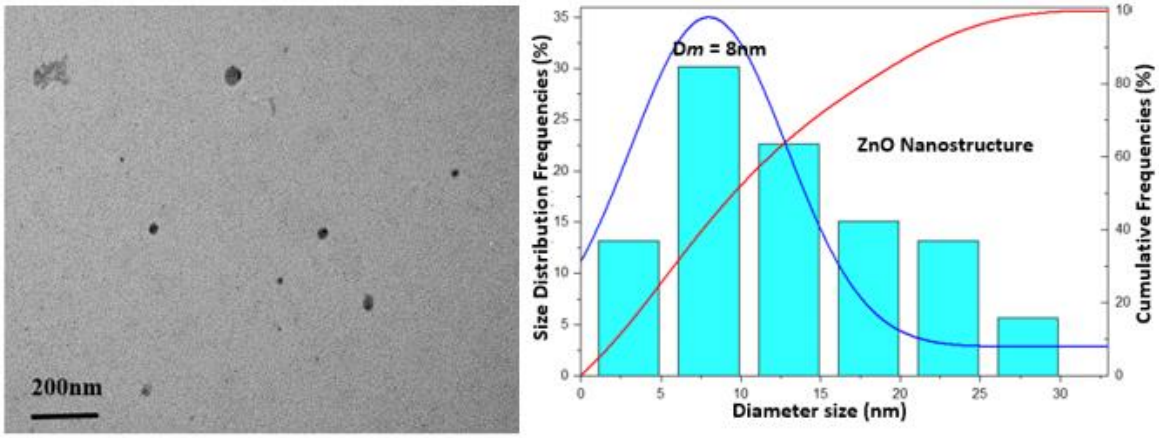

Figure 1: TEM image and size distribution chart of synthesised ZnO nanostructure without the addition of TA.

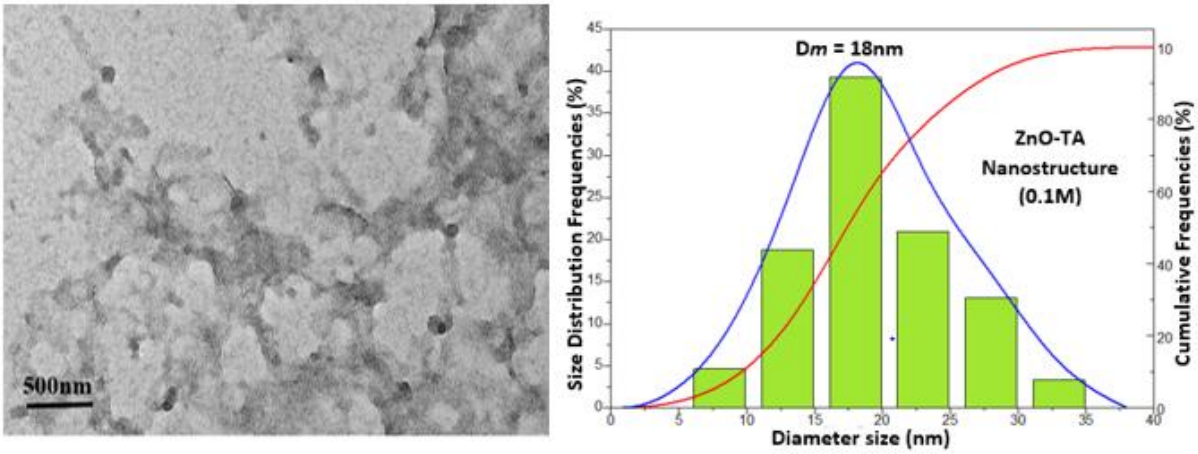

Figure 2: TEM image and size distribution chart of synthesised ZnO nanostructure with the addition of 0.1M TA.

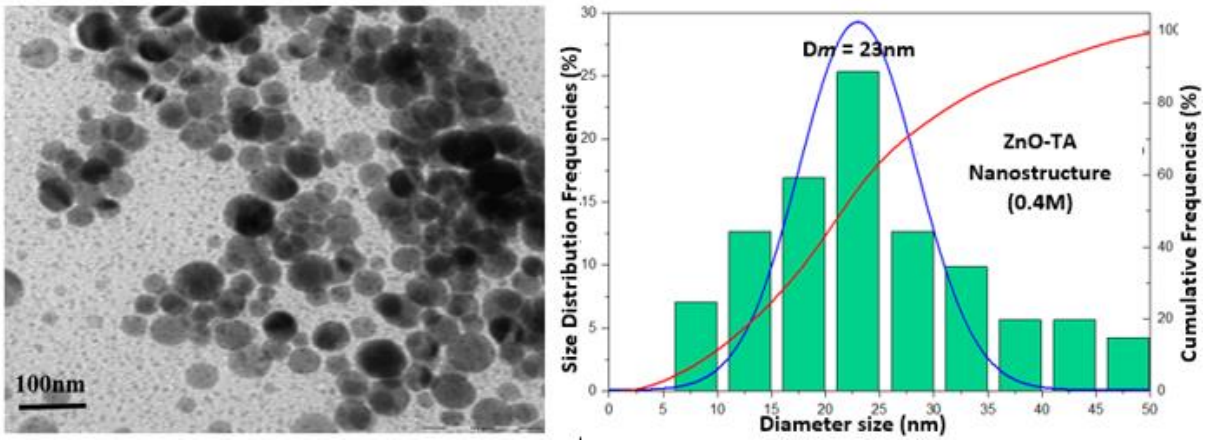

Figure 3: TEM image and size distribution chart of synthesised ZnO nanostructure with the addition of 0.4M TA.

These conditions revealed that the aggregation and agglomeration happened as the TA formed the acidic environment. The aggregation is caused by the coalescence of small particles from the attachment growth, which entirely depends on the samples' projected particles' volume with an increment of TA intensity. [13]. The mixing of the particles is aided by the slight fluctuations caused by a jump to contact action caused by the acidic environment. This condition is also known as oriented aggregations (OA) which is a crucial crystal growth mechanism. This can be considered direct evidence of OA, which reveals the nanocrystals' structure and surface chemistry's fundamental changes. In acidic condition, the presence of proton attack at the surface of ZnO is high. Therefore, the aggregation occurred as the phenolic group's protonation took place in which reduced the accessibility of the functional group to squeeze between the ZnO nanocrystals (jump contact) and created a larger attached particle. Besides, the drying process for TEM sample preparation also promotes non-homogeneous deposition. Thus, the aggregation of ZnO nanostructures is inevitable as the solvent evaporates [19, 20].

CONCLUSION

In conclusion, the reported study presented that ZnO nanostructure can be produced through the simple hydrothermal method and further addition of high concentration of mild acid TA would result in the aggregation of the as -synthesised ZnO nanostructures. Both pure ZnO and ZnO-TA nanostructures showed diameter size smaller that is smaller than 50 nm as compared to other existing literature works. The pure ZnO nanostructures have an average diameter range of 8nm while the ZnO-TA nanostructure has an average diameter of 18 and 23 nm for a sample with a TA concentration of 0.1 and 0.4M respectively. The increment in the size of ZnO nanostructure upon the TA's addition is due to the aggregation due to the acidic medium that enhanced the proton attacked between the particles.

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