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Research Article

EFFECT OF CALCINATION TEMPERATURE ON ANTIMICROBIAL PROPERTIES OF ZnO/Ag NANOCOMPOSITE INCORPORATED IN ARCHITECTURAL PAINTS

***Hamed Asadi**
Department of Polymer Engineering, College of technical, South Tehran Branch, Islamic Azad University, Tehran, Iran
**Author for Correspondence*

ABSTRACT
In this work, ZnO/Ag nanocomposite were successfully synthesized using sol-gel process in different calcinations temperature and characterized by X-ray diffraction and scanning electron microscopy. These nanocomposites can be applied as the effective antibacterial or antifungal additives for architectural paints.
The antibacterial activity was evaluated by inhibition zone test for Escherichia coli. Based on the presented results it can be stated that these materials are very effective biocides and The small crystal size and large surface area of the hetero structure nanoparticles contribute to antibacterial enhancement.

Keywords: *Antibacterial, ZnO/Ag Nanocomposite, Sol-Gel*

INTRODUCTION
Nanoparticles have attracted considerable interest in recent years because of their excellent physical and chemical properties. As particles are reduced from micrometer to a nanometer size, the resultant properties can change dramatically. Since ancient times, the world's smallest life forms proved to be the most deadly. Improving hygienic standards in many parts of the world allow infectious diseases to be increasingly better controlled. Aside from contagion through contaminated air and direct contact with infected people or animals, contaminated objects play an important role in the spread of infectious diseases. The hygienic properties of material surfaces are therefore important (Shah *et al.*, 2013; Manikandan *et al.*, 2010).
The antimicrobial activity of silver is well established, especially when metals are applied in the form of nanoparticles (NPs). Silver NPs have strong inhibitory and antimicrobial effect as well as broad spectrum of biocides activities (Pal *et al.*, 2007; Panacek *et al.*, 2006; Sawai *et al.*, 2004) However, the mechanism of antibacterial effect of silver is still not fully understood. Nanoparticles of these metals are a new group of antimicrobial materials due to the different physicochemical properties as compared to the bulk materials of the same composition. The antimicrobial efficiency of these metals increase with decreasing their particle size due to their larger specific area.
Compared to all other metal-semiconductor materials, ZnO/Ag has received considerable attention, not only because ZnO with various nano-sized structures can be simply made via a series of simple processes (Karunakaran *et al.*, 2010) but also because Ag nanoparticles have good chemical and physical properties. Nano-sized ZnO is a bactericide and inhibits both Gram-positive and Gram-negative bacteria (Karunakaran *et al.*, 2011). Furthermore, doped Ag reduces the ionization energy of acceptors in ZnO and consequently enhances the emission (Gogoi *et al*., 2006). Therefore, Ag ions can enhance the antimicrobial ability of ZnO (Li *et al.*, 2008; Aymonier *et al.*, 2002).
In the present article, we report on the synthesis of Ag/ZnO nanocomposite with different calcination temperature through facile chemical sol-gel method. Effects of incorporation of Ag into ZnO crystal on their structural and antibacterial behavior of the ZnO nanocomposite incorporated in architectural paints are studied in details.
The present contribution provides an overview of currently used methods for improvement of hygienic properties of architectural paints materials and outlines potential novel strategies and topics of additional research in this area.

Experimental

High purity zinc acetate $[Zn(CH_3CO_2)_2]$, silver nitrate $Ag(NO_3)_2$, sodium hydroxide (NaOH) were purchased from Sigma-Aldrich and poly vinyl pyrrolidone (PVP) as capping agent, were used for chemical processing. for the synthesis of pure ZnO, 0.05 M zinc acetate $Zn(Ac)_2$ was dissolved in 100 ml of de-ionized water and 0.1 M NaOH was dissolved in 100 ml deionized water. NaOH solution was added drop wise to $Zn(Ac)_2$ solution. Then, a white colored gel is produced and this gel was kept for ageing overnight. Similarly for the synthesis of Ag doped ZnO nanorods, 0.1 M $Zn(Ac)_2$ solution with milimolar silver nitrate and aqueous ammonia (1:1) was added drop wise to reach a pH = 7, and the stirring was continued for another 30 minutes. A few drops of PVP were also added during stirring for controlling growth. The formed glassy like white gel was allowed to age overnight. It was filtered, washed, dried at 100°C for 12 hrs and annealed at 500°, 600°, 700°and 800°C for 2 hrs in a muffle furnace.

Acrylic emulsion paint containing ZnO/Ag nanocomposite was prepared using laboratory mixer. The Paint was modified with 0.1 wt% of ZnO/Ag nanocomposite.

RESULTS AND DISCUSSION

Characterization

The morphology and structure of the Ag/ZnO nanocomposite were further investigated by transmission electron microscope (SEM), SEM (Philips EM208) were operated at 100 kV. The Powder X-ray diffraction (XRD) pattern was recorded on a Seisert Argon 3003 PTC using nickel-filtered XD-3a Cu Kα radiations (λ=0.1542 nm). The average primary particle size was also estimated from the XRD pattern using the X-ray line broadening analysis.

Antibacterial Test

The Ag/ZnO nanocomposite with different calcination temperature incorporated in architectural paints were used to find the optimum temperature for calination of nanocomposite mixed in the sterilized nutrient broth and it was sonicated for 15 minutes in an ultrasonic bath. 100 µl of 24 hrs E.coli culture was added to the sonicated broth and allowed to photo-catalysis. Photo-catalysis was performed in circular beam light setup equipped with 250 watts halogen lamp for 40 minutes. After 40 minutes the sample containing nutrient broth was taken out and serially diluted using sterile water. From the above diluted sample 50 µl was inoculated in nutrient agar by spread plate method. These plates were incubated at 37°C for 24 hours. Then the plates were visually observed and bacterial colonies were counted by colony counter. Analyses were in duplicates and control runs were carried out each time under the same illumination conditions but without any photo catalytic materials. Similar tests were also performed in the dark in presence of the photo catalyst for comparison.

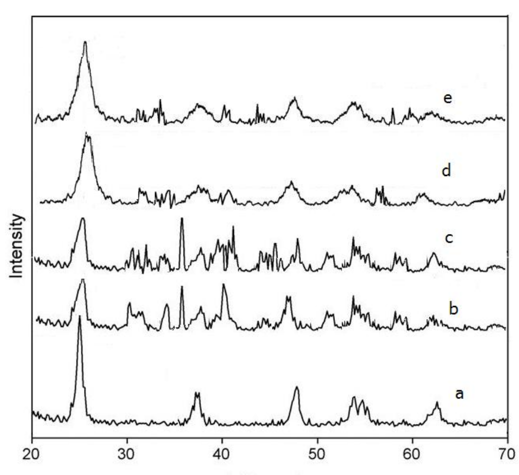

Figure 1: X-ray diffraction patterns of: a) ZnO and ZnO/Ag samples with different preparing temperature; b) 500C; c) 600°C; d) 700°C; e) 800 °C

XRD patterns of selected Ag/ZnO nanocomposite are shown in Figure 2. All peaks can be assigned to the standard hexagonal structure zinc oxide (JCPDS no. 01-079-0207) and (111), (200), (220), and (311) crystallographic planes of face centered cubic (fcc) silver nanoparticles, respectively [JCPDS no.01-087-0597]. No other crystalline phase was found.

The average grain sizes (D) of the products prepared in different temperatures are shown in Table 1, which is calculated from the width of the lines in the XRD spectrum with the aid of the Scherrer formula: $D = k\lambda / (\beta\cos\theta)$, where λ is the wavelength of X-ray used (Cu Kα radiation λ = 0.1541 nm), β the width of the line at the half maximum intensity and $K\alpha$ constant, 0.89. From Table 1, it can be seen that the average particle size increase with increasing preparing temperature, which is in a good agreement with the result of TEM analysis

Table 1: The average grain size of products obtained in different temperatures

Preparing temperature(◦C)	Preparing time (h)	D (nm)
500	2	26.35
600	2	28.53
700	2	32.78
800	2	33.65

Figure 2 a-d shows the SEM images of the ZnO/Ag nanocomposite. The SEM images of the ZnO/Ag composites are shown in Figure 2. It can be seen that ZnO/Ag nanocomposite has hexagonal structure and as the calcination temperature increased the size of the ZnO/Ag nano-particles increased too.

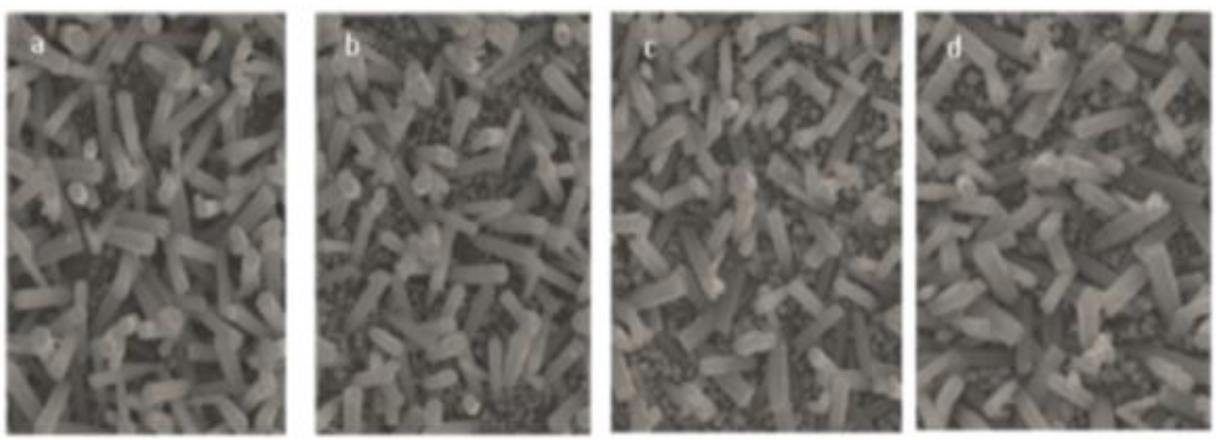

Figure 2: SEM micrographs of samples of ZnO/Ag nanocomposite prepared in different Calcinated temperatures: a) 500°C, b) 600°C, c) 700°C and d) 800°C

Antibacterial Activity of Ag/ZnO Nanocomposite Incorporated in Architectural Paints

The silver nanoparticles were doped in the ZnO nanoparticles (Ag doped ZnO nanoparticles) in different calcination temperature incorporated in architectural paints were tested for Antibacterial activity.

After the specified contact time, antimicrobial activities of raw and the treated samples against E. coli bacteria were calculated. Antibacterial activity of samples was determined in terms of inhibition zone formed on agar medium. The results of antimicrobial activity of Ag/ZnO nanocomposite incorporated in architectural paints are presented on Figure 3.

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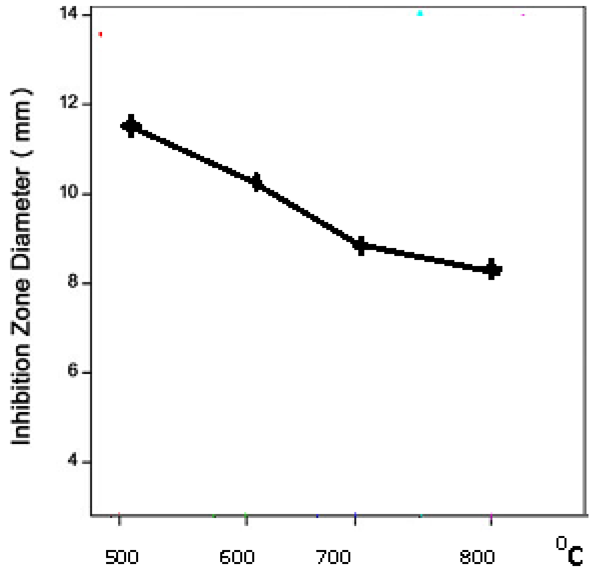

Figure 3: The results of antimicrobial activity of ZnO/Ag nanocomposite prepared in different Calcinated temperatures incorporated in architectural paints: a) 500°C, b) 600°C, c) 700°C and d) 800°C

Based on the presented results it can be stated that these materials are very effective biocides in malnutrition conditions against E. coli. The small crystal size and large surface area of the hetero structure nanoparticles may contribute to antibacterial enhancement.

Conclusion

Ag/ZnO nanocomposite were synthesized by the sol-gel method and then incorporated in architectural paints. The grain size was controlled by using poly vinyl pyridine as capping agent. The enhanced bioactivity was demonstrated by studying the antibacterial activity of paints with Ag/ZnO nanocomposite. These improved bioactivities of smaller particles were attributed to the higher surface to volume ratio.

The smaller particles need more particles to cover a bacterial colony, which results in the generation of active oxygen species, which will kill bacteria more effectively.

The application of the developed additives allows achieve higher hygienic level with the significant decrease of the applied amount of aggressive biocides which are harmful to human health.

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