J Nanopart Res (2013) 15:1925
DOI 10.1007/s11051-013-1925-6

RESEARCH PAPER

Microleakage and antibacterial properties of ZnO and ZnO:Ag nanopowders prepared via a sol–gel method for endodontic sealer application

M. Shayani Rad · A. Kompany · A. Khorsand Zak · M. Javidi · S. M. Mortazavi

Received: 1 December 2012 / Accepted: 10 August 2013

Abstract One of the most important problems in dentistry is the microleakage, whether apical or coronal, which may cause failure of root canal therapy. The aim of this study is to prepare suitable sealer to decrease the microleakage of the root canals as well as having good antibacterial property. Pure ZnO and ZnO:Ag nanopowders were synthesized via sol gel method using gelatin as polymerization agent calcined at different temperatures of 500, 600, and 700 °C for 8 h. The prepared samples were characterized using X-ray diffraction and transition electron microscopy. The microleakage and antibacterial properties of the prepared samples were investigated and compared with zinc oxide eugenol (ZOE) and epoxy resin sealer (AH26), which are commonly used in dentistry as sealers. The results showed that the synthesized pure ZnO and ZnO:Ag nanopowders exhibit better microleakage and antibacterial properties in comparison with ZOE and AH26 sealers, and therefore are more suitable filling materials to be used as sealer in root canal treatment.

Keywords ZnO:Ag nanopowders · Sol–gel · Sealer · Microleakage · Antibacterial · Zinc oxide eugenol · AH26

M. Shayani Rad · A. Kompany
Materials and Electroceramics Laboratory, Department of Physics, Faculty of Sciences, Ferdowsi University of Mashhad, Mashhad, Iran

A. Khorsand Zak (✉)
Nanotechnology Laboratory, Esfarayen University, North Khorasan, Iran
e-mail: alikhorsandzak@gmail.com

M. Javidi · S. M. Mortazavi
Dental Material Research Centre, Department of Endodontics, Faculty of Dentistry, Mashhad University of Medical Sciences, Mashhad, Iran

Introduction

ZnO is a safe material which has been used widely in biomedical applications such as cancer treatment (Cory et al. 2008; Nair et al. 2009) and DNA detection (Nitin et al. 2006). Although, ZnO has interesting antibacterial properties (Applerot et al. 2009; Li et al. 2007; Zhang et al. 2008) which can be enhanced by doping some elements such as Ni and Co in ZnO matrix (Moribe et al. 2007; Nair et al. 2011); ZnO nanostructures show better antibacterial properties due to their surface enhancement. Different methods have been used for the synthesis of nanomaterials including solvothermal and hydrothermal (Balucani et al. 2011; Hao et al. 2012; Razali et al. 2011), chemical vapor deposition (CVD), laser ablation, oxidation process, precipitation, gel-combustion, and sol–gel (Chandrappa et al. 2010; Gui et al. 2006; Khorsand Zak et al. 2011; Yousefi et al. 2010, 2011a, b; Zak et al. 2011a, b; Zamiri et al. 2012).

Published online: 24 August 2013

As mentioned earlier, in comparison with ZnO, ZnO:Ag shows better antibacterial activity (Karunakaran et al. 2010; Matei et al. 2008) which can be used as sealer for dental applications. In dentistry, root canal microleakage is a big clinical problem, whether apical or coronal, which may cause failure of root canal therapy (Dow and Ingle 1955; Madison and Wilcox 1988). Therefore, an endodontic sealer must have good sealing ability (Branstetter and von Fraunhofer 1982). It has been reported that using zinc oxide can decrease the root canal leakage (Camps et al. 2004). Later, Takatsuka et al. (2005) found that the toothpaste with zinc is statistically significant, 49 % greater inhibitory efficacy on dentin demineralization over the control. This is important in both static and dynamic situations, because a sealer must eliminate any space which allows penetration of fluid between the filling materials and walls of the root canals (ØRstavik et al. 1983). Wong et al. (2011) used casein phosphopeptide–amorphous calcium phosphate (CPP–ACP) to determine its effect on the physical properties of two commercially available zinc oxide non-eugenol temporary luting cements. They found that the compressive and diametral tensile strengths progressively decrease with increasing the concentrations of CPP–ACP up to 8.0 % (w/w). The leakage along the root canal fillings may increase or decrease with time. Dissolution of the sealer can increase the leakage (Wu et al. 2000a), whereas swelling of gutta-percha may decrease it in the root canal (Wu et al. 1994). Physical and chemical properties of the sealers such as the thickness, microleakage, and antibacterial properties can also play an important role in sealing of the root canals (Wu et al. 2000b).

In this work, ZnO nanopowders were synthesized and characterized to be used as sealer in the root canal treatments. Nanoparticles are able to diffuse in the root bone which results in the decrease of the root canal leakage. In order to increase the antibacterial properties of ZnO nanopowders, silver was added into the ZnO matrix. ZnO and ZnO:Ag nanopowders were prepared by a modified sol–gel method. The antibacterial activity and the apical leakage of the roots obturated with gutta-percha filled with synthesized nano-sized zinc oxide eugenol (ZOE) sealer, epoxy resin sealer (AH26), and micro-sized ZOE sealer were investigated and compared.

Materials and method

Synthesis of ZnO and ZnO:Ag nanoparticles

To synthesize 5 g of the final product, first a solution of gelatin (type B from bovine skin, Sigma Aldrich) was prepared by dissolving 10 g gelatin in 150 ml deionized water at 60 °C. Then, appropriate amounts of zinc nitrate ($Zn(NO_3)_2{\cdot}6H_2O$, Merck 99%) and silver nitrate ($AgNO_3$, Merck %99) were dissolved in a minimum volume of deionized water at room temperature. The amounts of zinc and silver nitrates were chosen according to $Zn_{1-x}Ag_xO$ ($x = 0$, 0.01, and 0.03) formula considering 5 g of the final product for each compound. The two prepared solutions were then mixed and stirred for 8 h while the temperature was kept at 80 °C. This process was performed for $x = 0, 0.01$, and 0.03 to achieve the brown resins. The role of gelatin is to terminate the nanoparticles growth, due to the cation mobility limitation in the presence of gelatin (Khorsand Zak et al. 2012). Ag^+ needs one electron to be reduced to Ag in which the electron is obtained from the gelatin. Therefore, gelatin plays as a reducing agent for Ag^+ (Darroudi et al. 2012). The reactions are as follows:

$$Zn^{2+} + O^{2-}_{gelatin} \xrightarrow{\text{Calcination}} ZnO + CO_2 + NO_2 + \cdots$$

$$Ag^+ + e^-_{gelatin} \xrightarrow{80\ ^\circ C} Ag$$

Finally, the prepared resin of the pure sample was calcined at different temperatures of 500, 600, and 700 °C in which the pure ZnO nanopowders were obtained. Also, ZnO:Ag nanopowders with 1 and 3 % Ag were produced at 700 °C calcination temperature.

Sample preparation for microleakage measuring

In this study, 56 single-rooted anterior teeth were selected. All the roots were cross-sectioned at the cemento-enamel junction (CEJ) by a carborundum disk (Brasseler USA, Savannah, GA), then a #10 K-file (Dentsply Maillefer, Ballaigues, Switzerland) was placed in the canal until visible at the apex and pulled back 1 mm to determine the working length. Instrumentation of all teeth was performed using stainless steel K-files. The root canal system was instrumented by a step back technique using hand files (Densply, Maillefer, Switzerland). All the samples were prepared to an ISO size 35. Irrigation was performed using 1 ml of 5.25 %

NaOCl between each file. After that, a #15 file was used to maintain a patent apex. On completion of instrumentation, the specimens were randomly divided into seven groups consisting eight teeth in each group. In all groups, the smear layer was removed using 1 ml 17 % EDTA (Ariadent, Asia Chemi Teb, Tehran, Iran) for 1 min, followed by 3 ml of 5.25 % NaOCl and the canals were finally flushed with 5 ml normal saline. The root canals were dried with paper points before obturation. The first group was filled with gutta-percha using AH26 (Dentsply, DeTrey, Konstanz, Germany) as a sealer cement with the lateral condensation technique, according to the manufacturer's instruction. Five groups were filled with the prepared pure ZnO and ZnO:Ag nanopowders and the last group was filled with ZOE sealer (zinc oxide eugenol micro-powder, Pulp Dent, Watertown's, MA, USA). To allow the material to be set, all the roots were stored at 100 % humidity and 37 °C environment for 3, 45, and 90 days in an incubator. Then, each tooth was placed in a device for measuring its microleakage using fluid transport process, designed by Derkson et al. (1986). The method of Wu and Wesselink (Wu et al. 1995) was used for restorative material leakage and later adapted for endodontic studies.

Measurement of microleakage

The leakage measuring system used in this study is based on the evaluation of fluid transport in the specimen (Mahera et al. 2009), calculated from the bubble movement, shown in Fig. 1. An oxygen tank equipped with a monometer was used to apply pressure on the fluid to make it pass through the specimen and hence move the bubble. All the connections of the system were smeared with cyanoacrylate glue (Inter Lock, Japan) and covered by multiple layers of parafilm strips (Parafilm "M"; Laboratory Film, Chicago, IL, USA). This strip seals the connections of the tubes which ensure having impervious connections. This system had two parts; the first part contains the tubes, micropipette, pipes, and the tooth sample that transfer pressure to the specimen and the second part includes a recorder for recording the fluid transport, Fig. 1. The camera was adjusted in micrograph to take a precise picture from a short distance. The control faucet was opened to the tooth and the syringe was removed from the pass. Now, only the tooth and the fluid filtration system were connected. The main faucet of the oxygen tank was then opened. The pressure had previously been adjusted, since it must be kept constant during all steps of the experiment. We waited 30 s to attain a balance in the system, and then the first picture of the bubble position in the micropipette was taken. Four subsequent pictures were taken at 2 min intervals (2, 4, 6, and 8 min), after taking the first one. The same steps were repeated for the other samples.

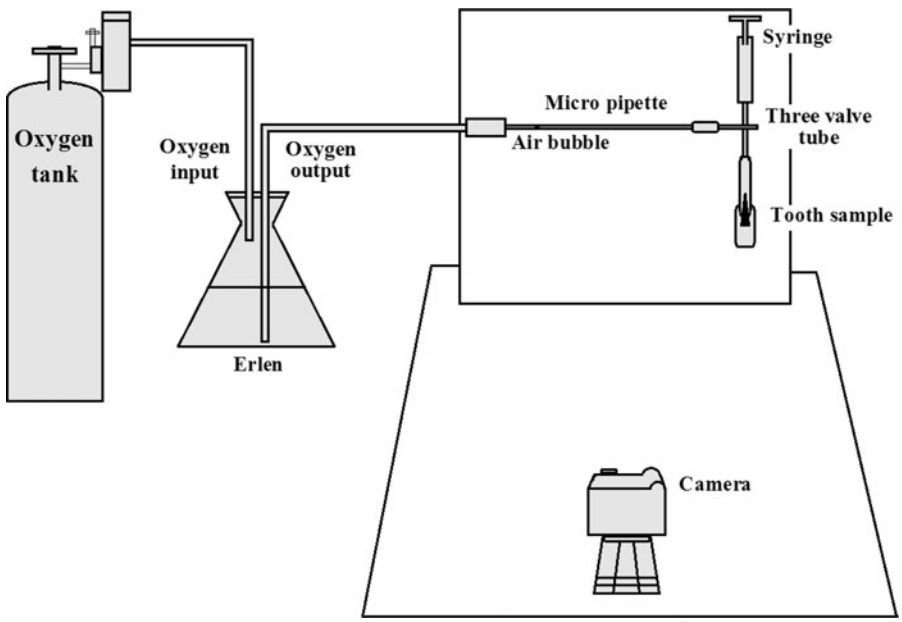

Fig. 1 Schematic view of the designed fluid filtration system for measuring microleakage

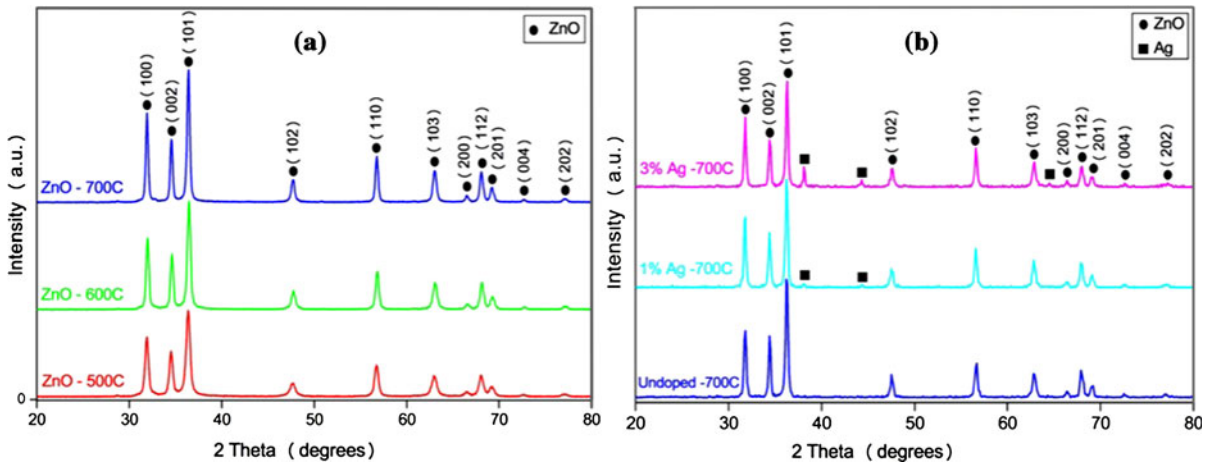

Fig. 2 X-ray diffraction (XRD) patterns of: **a** pure ZnO nanoparticles prepared at three different calcination temperatures and **b** pure ZnO and ZnO:Ag nanoparticles prepared at 700 °C

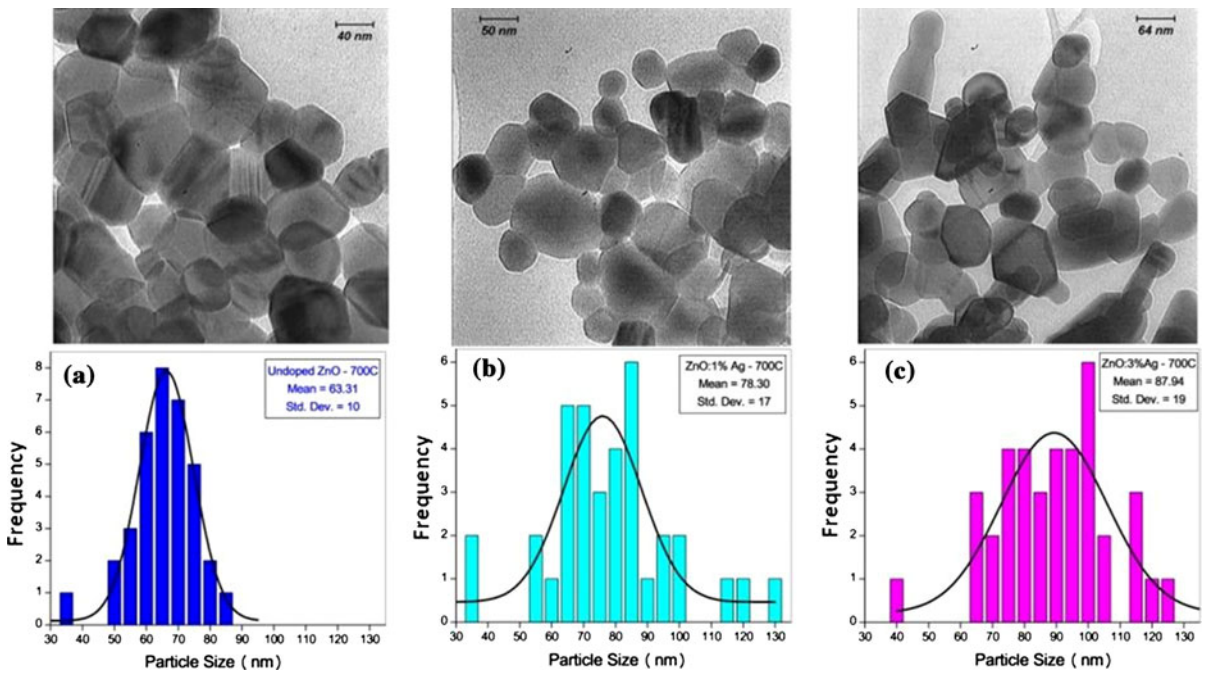

Fig. 3 TEM images of: **a** pure ZnO, **b** ZnO:1 % Ag, and **c** doped ZnO:3 % Ag nanoparticles calcined at 700 °C with average particle sizes of 63, 78, and 88 nm, respectively

Then, the samples were placed in the incubator for 3 days, 1.5 and 3 months, after which the same steps were carried out for all of them. The mean displacement of the bubble per minute was calculated and the longitudinal displacement of the bubble was converted into the volume of fluid passing through the samples, showing it in terms of µl/min/cm H_2O.

Measurement of antibacterial activity

The blood agar culture medium was used for *Enterococcus Faceless* bacteria susceptibility test. First, the medium was made and sterilized according to the manufacturer's instruction and then divided in petri dishes. After that, four holes with 6 mm diameter were made in each petri dish. A swap was inserted in

microbial located and moved to the medium surface along two perpendicular directions. Certain amounts of pure ZnO nanoparticles calcined at 500, 600, and 700 °C, ZnO:1 % Ag and ZnO:3 % Ag calcined at 700 °C, and also, ZnO micropowders were poured into each petri's hole. Each petri was held at room temperature for 2 h, and then incubated at 35 °C. The petri dishes were removed from the incubator after 24, 48, and 72 h and the diameter of inhibition growth zones was measured by a special ruler. The mean zone diameters between all groups were compared using Kruskal–Wallis and the subsequent Mann–Whitney tests.

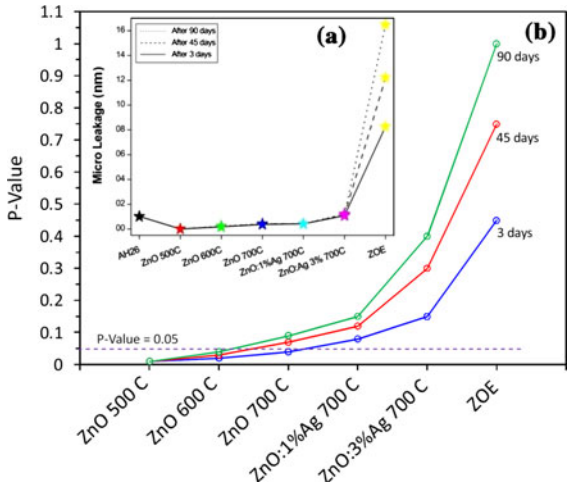

Fig. 4 **a** The mean microleakage and **b** microleakage of pure and ZnO:Ag nanoparticles and ZOE in comparison with AH26 ($p < 0.05$)

Results and discussion

X-ray diffraction and TEM analysis

Figure 2a shows X-ray diffraction (XRD) patterns of pure ZnO nanopowders synthesized at 500, 600, and 700 °C calcination temperatures. All the diffraction peaks are related to the hexagonal structure of ZnO. There is no extra diffraction peak detected corresponding to impurities or other compounds. In Fig. 2b the peaks corresponding to the metallic phase of silver confirm the existence of the silver element in the ZnO matrix. As mentioned earlier, Ag^{+} obtains one electron from the gelatin and reduces to Ag. Since, it is not easy for Ag atoms to diffuse into the ZnO matrix, during the calculation process, ZnO:Ag composite is formed. Transition electron microscopy (TEM) images and the corresponding particle size histograms of pure ZnO and ZnO:Ag nanopowders calcined at 700 °C are shown in Fig. 3. The average size of the nanoparticles is found to be about 63, 78, and 88 nm for pure ZnO and for 1 % and 3 % ZnO:Ag nanopowders, respectively. The crystallite size of the samples was calculated using Scherrer equation ($D = \frac{K\lambda}{\beta \cos\theta}$), where K is the shape factor equal to 0.98 and β is the FWHM of the diffraction peak used in our calculation. The crystallite size of the pure ZnO and for 1 and 3 % ZnO:Ag nanopowders were obtained to be 31, 38, and 45 nm, respectively.

Microleakage and antibacterial results

The microleakage measurement of the synthesized nanopowders and also the conventional ZOE and

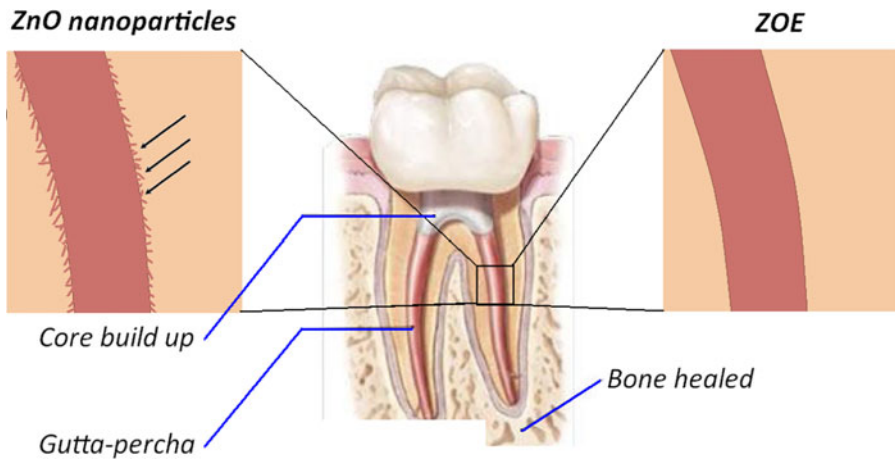

Fig. 5 The ZnO nanoparticles are able to diffuse in the bone, as shown by *arrows*, and therefore can fill the holes and calk of the pores better than ZOE

Table 1 The *p*-value results of the synthesized samples of this work in comparison with some other works

Materials and methods	*p*-value	Time (day)	References
Thermafil	0.1	–	Rajeswari et al. (2007)
Obtura	0.21		
AH26	0.75	60	Shantiaee et al. (2011)
AH26 and ZOE	0.45	3	Present work
	0.75	45	
	1.0	90	
ZnO—500 °C	0.01	3	
	0.01	45	
	0.01	90	
ZnO—600 °C	0.02	3	
	0.03	45	
	0.04	90	
ZnO—700 °C	0.04	3	
	0.07	45	
	0.09	90	
ZnO:1 % Ag—700 °C	0.08	3	
	0.12	45	
	0.15	90	
ZnO:3 % Ag—700 °C	0.15	3	
	0.30	45	
	0.40	90	

AH26 sealers were performed after 3, 45, and 90 days to check the stability of their sealing property. The results that are shown in Fig. 4a indicate that the ZnO and ZnO:Ag nanopowders prepared at different calcination temperatures exhibit less microleakage than ZOE and AH26. The maximum microleakage belongs to ZnO:3 % Ag nanopowders calcined at 700 °C; whereas, the minimum is attributed to the pure ZnO calcined at 500 °C. Therefore, the microleakage of the ZnO nanopowder has increased by adding Ag, because of the bigger size of ZnO:Ag nanoparticles. One can conclude that the root canal can be sealed better using smaller nanopowders. As mentioned earlier, the ZnO nanoparticles are able to diffuse into the root bone due to their small size leading to better calk of the pores, Fig. 5. Figure 4b shows the value of the microleakage differences between AH26 and our synthesized nanopowders, revealing that all groups have remarkable leakage much different from ZOE common sealer. The significant of p value should be: $p < 0.05$ (Ann Ximenez-Fyvie et al. 1996). It was observed that the

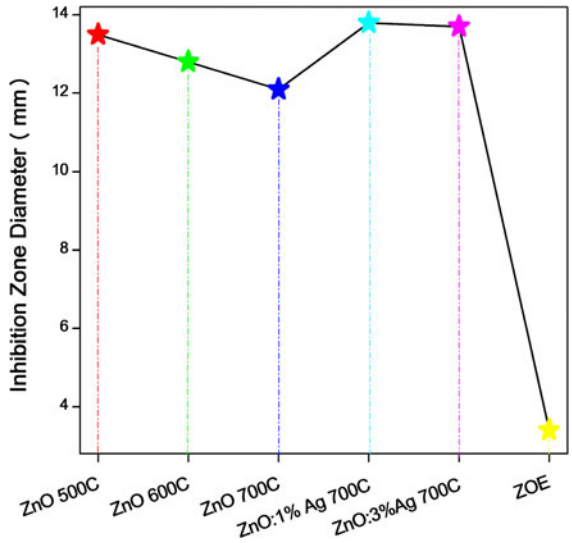

Fig. 6 The antibacterial activity of the synthesized pure and ZnO:Ag nanoparticles compared with ZOE

samples calcined at 500 and 600 °C show significant *p*-value results. The obtained results are presented and compared with the others in Table 1.

As shown in Fig. 6 the antibacterial activity of pure ZnO nanopowders decreases with increasing the calcination temperature. This can be related to the increase of the particle size of ZnO nanopowders at higher calcination temperature, since the effective surface area of ZnO nanopowders decreases with the particle size growth (Amornpitoksuk et al. 2011). As expected, the presence of Ag has slightly enhanced the antibacterial effect of ZnO nanopowders, due to the antibacterial property of Ag. The results show that the synthesized pure ZnO and ZnO:Ag samples in this research clearly have better antibacterial properties, in comparison with ZOE conventional sealer.

Conclusions

The pure ZnO and ZnO:Ag nanopowders were synthesized via a sol–gel method in order to be used it as sealer for dental applications. The structure, particle size, microleakage, and antibacterial activity of the prepared samples were investigated and compared. It can be concluded that the prepared ZnO and ZnO:Ag nanopowders have a leakage less than the standard gutta-percha (AH26) and ZOE which are commonly used as root filling materials. Considering

the better antibacterial properties of the ZnO and ZnO:Ag, the use of these materials might be more efficient in endodontic treatments. However, further ex vivo and in vivo studies are needed to assess the other properties of these new materials.

Acknowledgments This work was supported by the Ferdowsi University of Mashhad (Vice President for Research and Technology), under code number 1/20960. We also acknowledge the assistance of the Endodontics Department, Mashhad University of Medical Sciences.

References

Amornpitoksuk P, Suwanboon S, Sangkanu S, Sukhoom A, Wudtipan J, Srijan K, Kaewtaro S (2011) Synthesis, photocatalytic and antibacterial activities of ZnO particles modified by diblock copolymer. Powder Technol 212(3):432–438. doi:10.1016/j.powtec.2011.06.028

Ann Ximenez-Fyvie L, Ximenez-Garcia C, Manuel Carter-Bartlett P, Javier Collado-Webber F (1996) Accuracy of endodontic microleakage results: autoradiographic vs. volumetric measurements. J Endod 22(6):295–297

Applerot G, Lipovsky A, Dror R, Perkas N, Nitzan Y, Lubart R, Gedanken A (2009) Enhanced antibacterial activity of nanocrystalline ZnO due to increased ROS-mediated cell injury. Adv Funct Mater 19(6):842–852. doi:10.1002/adfm.200801081

Balucani M, Nenzi P, Chubenko E, Klyshko A, Bondarenko V (2011) Electrochemical and hydrothermal deposition of ZnO on silicon: from continuous films to nanocrystals. J Nanopart Res 13(11):5985–5997. doi:10.1007/s11051-011-0346-7

Branstetter J, von Fraunhofer JA (1982) The physical properties and sealing action of endodontic sealer cements: a review of the literature. J Endod 8(7):312–316

Camps J, Pommel L, Bukiet F, About I (2004) Influence of the powder/liquid ratio on the properties of zinc oxide–eugenol-based root canal sealers. Dent Mater 20(10):915–923. doi:10.1016/j.dental.2004.02.002

Chandrappa K, Venkatesha T, Vathsala K, Shivakumara C (2010) A hybrid electrochemical–thermal method for the preparation of large ZnO nanoparticles. J Nanopart Res 12(7):2667–2678. doi:10.1007/s11051-009-9846-0

Cory H, Janet L, Alex P, Reddy KM, Isaac C, Andrew C, Kevin F, Denise W (2008) Preferential killing of cancer cells and activated human T cells using ZnO nanoparticles. Nanotechnology 19(29):295103

Darroudi M, Khorsand Zak A, Muhamad MR, Huang NM, Hakimi M (2012) Green synthesis of colloidal silver nanoparticles by sonochemical method. Mater Lett 66(1):117–120. doi:http://dx.doi.org/10.1016/j.matlet.2011.08.016

Derkson GD, Pashley DH, Derkson ME (1986) Microleakage measurement of selected restorative materials: a new in vitro method. J Prosthet Dent 56(4):435–440

Dow PR, Ingle JI (1955) Isotope determination of root canal failure. Oral Surg Oral Med Oral Pathol 8(10):1100–1104. doi:10.1016/0030-4220(55)90062-1

Gui Y, Xie C, Zhang Q, Hu M, Yu J, Weng Z (2006) Synthesis and characterization of ZnO nanostructures by two-step oxidation of Zn nano- and microparticles. J Cryst Growth 289(2):663–669. doi:10.1016/j.jcrysgro.2005.11.114

Hao Y, Lou S, Zhou S, Wang Y, Chen X, Zhu G, Yuan R, Li N (2012) Novel magnetic behavior of Mn-doped ZnO hierarchical hollow spheres. J Nanopart Res 14(1):1–9. doi:10.1007/s11051-011-0659-6

Karunakaran C, Rajeswari V, Gomathisankar P (2010) Antibacterial and photocatalytic activities of sonochemically prepared ZnO and Ag–ZnO. J Alloy Compd 508(2):587–591. doi:10.1016/j.jallcom.2010.08.128

Khorsand Zak A, Razali R, Abd. Majid WH, Darroudi M (2011) Synthesis and characterization of a narrow size distribution of zinc oxide nanoparticles. Int J Nanomed 6:1399–1403

Khorsand Zak A, Yousefi R, Majid WHA, Muhamad MR (2012) Facile synthesis and X-ray peak broadening studies of Zn1–xMgxO nanoparticles. Ceram Int 38(3):2059–2064. doi:http://dx.doi.org/10.1016/j.ceramint.2011.10.042

Li Q, Chen S-L, Jiang W-C (2007) Durability of nano ZnO antibacterial cotton fabric to sweat. J Appl Polym Sci 103(1):412–416. doi:10.1002/app.24866

Madison S, Wilcox LR (1988) An evaluation of coronal microleakage in endodontically treated teeth. Part III. In vivo study. J Endod 14(9):455–458

Mahera F, Economides N, Gogos C, Beltes P (2009) Fluid-transport evaluation of lateral condensation, ProTaper gutta-percha and warm vertical condensation obturation techniques. Aust Endod J 35(3):169–173. doi:10.1111/j.1747-4477.2009.00158.x

Matei A, Cernica I, Cadar O, Roman C, Schiopu V (2008) Synthesis and characterization of ZnO–polymer nanocomposites. Int J Mater Form 1:767–770. doi:10.1007/s12289-008-0288-5

Moribe S, Ikoma T, Akiyama K, Zhang Q, Saito F, Tero-Kubota S (2007) EPR study on paramagnetic species in nitrogen-doped ZnO powders prepared by a mechanochemical method. Chem Phys Lett 436(4–6):373–377. doi:10.1016/j.cplett.2007.01.067

Nair S, Sasidharan A, Divya Rani V, Menon D, Nair S, Manzoor K, Raina S (2009) Role of size scale of ZnO nanoparticles and microparticles on toxicity toward bacteria and osteoblast cancer cells. J Mater Sci: Mater Med 20:235–241. doi:10.1007/s10856-008-3548-5

Nair MG, Nirmala M, Rekha K, Anukaliani A (2011) Structural, optical, photo catalytic and antibacterial activity of ZnO and Co doped ZnO nanoparticles. Mater Lett 65(12):1797–1800. doi:10.1016/j.matlet.2011.03.079

Nitin K, Adam D, Jong-in H (2006) Ultrasensitive DNA sequence detection using nanoscale ZnO sensor arrays. Nanotechnology 17(12):2875

ØRstavik D, Eriksen HM, Beyer-Olsen EM (1983) Adhesive properties and leakage of root canal sealers in vitro. Int Endod J 16(2):59–63. doi:10.1111/j.1365-2591.1983.tb01297.x

Rajeswari P, Gopikrishna V, Parameswaran A, Gupta T, Kandaswamy D (2007) In-vitro evaluation of apical micro leakage of thermafil and obtura II heated gutta percha in comparison with cold lateral condensation using fluid filtration system. Paper presented at the Endodontology, Indian Dental Association, Pune Branch

Razali R, Zak AK, Majid WHA, Darroudi M (2011) Solvothermal synthesis of microsphere ZnO nanostructures in DEA media. Ceram Int 37(8):3657–3663. doi:10.1016/j.ceramint.2011.06.026

Shantiaee Y, Maziar F, Dianat O, Mahjour F (2011) Comparing microleakage in root canals obturated with nanosilver coated gutta-percha to standard gutta-percha by two different methods. Iran Endod J 6(4):140–145

Takatsuka T, Tanaka K, Iijima Y (2005) Inhibition of dentine demineralization by zinc oxide: In vitro and in situ studies. Dent Mater 21(12):1170–1177. doi:10.1016/j.dental.2005.02.006

Wong RH, Palamara JE, Wilson PR, Reynolds EC, Burrow MF (2011) Effect of CPP–ACP addition on physical properties of zinc oxide non-eugenol temporary cements. Dent Mater 27(4):329–338. doi:10.1016/j.dental.2010.11.011

Wu MK, De Gee AJ, Wesselink PR (1994) Leakage of four root canal sealers at different thicknesses. Int Endod J 27(6):304–308. doi:10.1111/j.1365-2591.1994.tb00273.x

Wu MK, Wesselink PR, Boersma J (1995) A 1-year follow-up study on leakage of four root canal sealers at different thicknesses. Int Endod J 28(4):185–189. doi:10.1111/j.1365-2591.1995.tb00297.x

Wu MK, Fan B, Wesselink PR (2000a) Diminished leakage along root canals filled with gutta-percha without sealer over time: a laboratory study. Int Endod J 33(2):121–125. doi:10.1046/j.1365-2591.2000.00274.x

Wu MK, Özok AR, Wesselink PR (2000b) Sealer distribution in root canals obturated by three techniques. Int Endod J 33(4):340–345. doi:10.1046/j.1365-2591.2000.00309.x

Yousefi R, Muhamad MR, Zak AK (2010) Investigation of indium oxide as a self-catalyst in ZnO/ZnInO heterostructure nanowires growth. Thin Solid Films 518(21):5971–5977. doi:10.1016/j.tsf.2010.05.111

Yousefi R, Muhamad MR, Zak AK (2011a) The effect of source temperature on morphological and optical properties of ZnO nanowires grown using a modified thermal evaporation set-up. Curr Appl Phys 11(3):767–770. doi:10.1016/j.cap.2010.11.061

Yousefi R, Zak AK, Mahmoudian MR (2011b) Growth and characterization of Cl-doped ZnO hexagonal nanodisks. J Solid State Chem 184(10):2678–2682. doi:http://dx.doi.org/10.1016/j.jssc.2011.08.001

Zak AK, Abrishami ME, Majid WHA, Yousefi R, Hosseini SM (2011a) Effects of annealing temperature on some structural and optical properties of ZnO nanoparticles prepared by a modified sol–gel combustion method. Ceram Int 37(1):393–398. doi:10.1016/j.ceramint.2010.08.017

Zak AK, Majid WHA, Darroudi M, Yousefi R (2011b) Synthesis and characterization of ZnO nanoparticles prepared in gelatin media. Mater Lett 65(1):70–73. doi:10.1016/j.matlet.2010.09.029

Zamiri R, Zakaria A, Ahangar HA, Darroudi M, Zak AK, Drummen GPC (2012) Aqueous starch as a stabilizer in zinc oxide nanoparticle synthesis via laser ablation. J Alloy Compd 516:41–48. doi:10.1016/j.jallcom.2011.11.118

Zhang L, Ding Y, Povey M, York D (2008) ZnO nanofluids—a potential antibacterial agent. Prog Nat Sci 18(8):939–944. doi:10.1016/j.pnsc.2008.01.026