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Effects of Surface Treatment on Zirconia-Dentine Microstructure Bonding Interface with Different Particle Sizes of Zirconia Powder

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ABSTRACT

Zirconia-based materials have emerged as promising resources for dental restoration applications owing to their excellent strength and aesthetic appearance. In clinical practices, surface treatment on zirconia is essential to ensure enhanced dental bonding. In this study, a novel method was used to produce zirconia block, and the influence of surface treatment on zirconia-dentine microstructure bonding interface was investigated for different particle sizes of zirconia powder. The particle sizes used in this study were 90 and 30 nm on the basis of manufacturer claims. A zirconia block of both particle sizes underwent surface treatment airborne-particle abrasion (50 μm, 0.4 MPa). Dentine specimens were prepared from extracted premolars stored in 0.1% tymol solution before being randomly cemented with the zirconia block sample by using self-adhesive resin cement (RelyX U2000 Clicker, 3M) and then light polymerised. The results after surface treatment showed higher surface roughness values for 90 and 30 nm at 0.17 and 0.18 μm, respectively, than those of the sample without surface treatment at 0.05 and 0.06 μm, respectively, showing no significant difference between particle sizes. Surface treatment improved the bonding of zirconia because the surface roughness allowed for enhanced interlocking of the cement onto the zirconia surface. However, the unbonded dentine and cement was due to the existence of a smear layer on the dentine surface that prevented the self-adhesive resin cement to work well with the dentine surface. Thus, dentine primer must be used to chemically remove the layer and allow for proper bonding between the resin cement and dentine surface.

Keywords: Zirconia; surface treatment; bonding; dentine; dental restoration

INTRODUCTION

Development in dental restoration has introduced polycrystalline ceramics, which include alumina and zirconia ceramics, as alternative materials besides metal or porcelains fused in metal (Amat et al. 2012; I. Denry & Kelly 2014; Shi et al. 2022). Zirconia ceramic stands out for its exceptional mechanical properties, making it the ideal choice (Isabelle Denry & Kelly 2008). Its mechanical strength, aesthetic appearance and biocompatibility makes zirconia the most suitable material for dental restoration.

One of the factors that contribute to the development of the novel zirconia developed by UKM is the improvisation of mechanical strength by using colloidal processing and slip casting as a fabrication method of the zirconia block. Compared with the commercial zirconia block, this method

had been proven to reduce agglomeration. Colloidal stability can be achieved using nitric acid and a dispersion agent, which allow particle dispersion to be controlled during powder processing and eventually prevent the agglomeration of fine particles (Amat et al. 2012, Chin et al. 2015, 2018). The use of 3 mol% yttria-stabilized zirconia (3YSZ) powder in research produced high-strength and translucent structures required for dental applications (Faeizah et al. 2017).

In dental restoration, specifically dental crowning, surface treatment is used to enhance the surface bonding between zirconia and cement to increase the bonding durability. Air abrasion with chemical formula Al_2O_3 particles is a common surface treatment used to promote a more retentive surface through the creation of rougher topography (De Sousa et al. 2016). A study showed that utilizing air abrasion in conjunction with phosphate ester monomers, specifically 10-methacryloxydecyl dihydrogen phosphate (MDP)-based cements such self-adhesive resin cement, can lead to significantly more robust and long-lasting bonding (Melo et al. 2015). This method is purposedly used to augment the ceramic surface area, generating a textured surface with increased contact points and micro-porosity, or to create optimal chemical bonding conditions to enhance the adhesion between the ceramic and the adhesive (Lin et al. 2021).

Most studied focused on the shear strength bonding of zirconia–cement only, and limited study has been conducted on the direct bonding of zirconia–dentine. Moreover, the performance of the bonding interface between novel zirconia and dentine is still unknown Thus, in this study, a novel method was used to produce a zirconia sample with two different sizes of zirconia powder to evaluate the surface morphology differences that may be affected by the powder sizes. This study aimed to observe the surface morphology after surface treatment and its bond to the dentine by using a self-adhesive resin cement

METHODOLOGY

Figure 1 illustrates the process flow for the sample preparation in this study

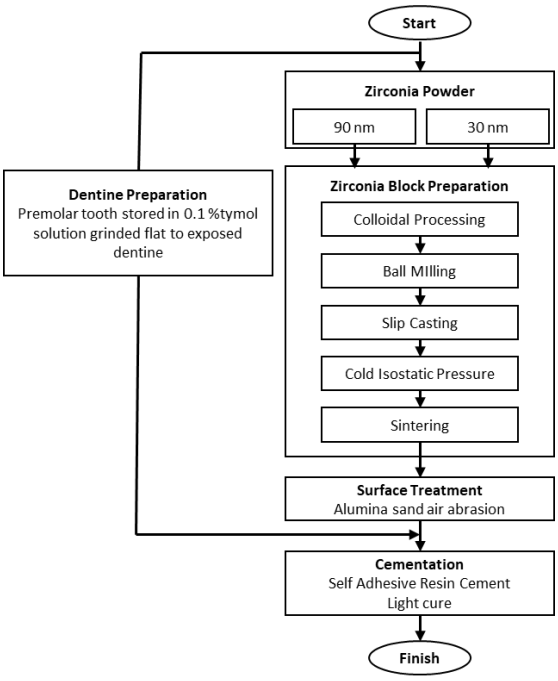


FIGURE 1. Flowchart of sample preparation

MATERIALS

The main material used in this research is zirconia nano powder stabilised with 3YSZ from two different manufacturers that claimed the powder particle size to be 90 and 30 nm, respectively. Each powder was examined to determine the particle size and analyse the average size distribution by using TEM (Talos L120C) and a nano particle-size analyser (NPSA), respectively, for microstructural analysis.

ZIRCONIA BLOCK PREPARATION

The zirconia suspension was prepared from 12% powder loading mixed with distilled water. Initially, 0.5 wt% polyethyleneimine (Sigma–Aldrich) with an average molecular size of 50,000 was added, and the pH was adjusted to 2 by using nitric acid. The suspension was stirred for 45 minutes and ultrasonicated for 10 minutes at 50 Hz to break up soft agglomerates. It was then transferred to a 250 mL zirconia vial and ball milled for 2 hours at 300 rpm with a 10 mm diameter zirconia ball at a ball-to-powder ratio of 10:1. The suspension was slipped cast into a Teflon mould (diameter of 10 mm × height of 21 mm) placed on Plaster of Paris (Multifilla Sdn. Bhd.) and dried at room temperature for 48–60 h to remove residual moisture. The zirconia samples underwent cold isostatic pressing at 250 MPa for 2 minutes and pre-sintering at 1100 °C for 12 hours and 45 minutes, followed by sintering at 1500 ° C for 18 hours and 22 minutes. Figure 2 illustrates the sample preparation process.

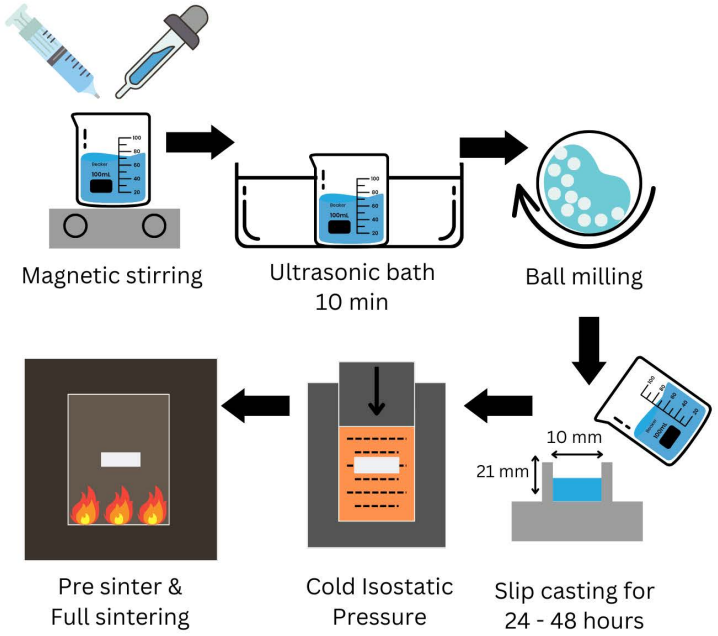


FIGURE 2. Schematic of zirconia block preparation

SURFACE TREATMENT AND BONDING PROCEDURE

Each zirconia disc was rinsed with distilled water and air dried before undergoing surface treatment of air abrasion (Renfert, USA) by using alumina with 50 µm particle size and 0.4 MPa pressure. For the bonding procedure, a self-adhesive resin cement was mixed and applied on the zirconia surface. Then, with hand pressure, the prepared dentine was bonded on top of the cement. A dental LED curing machine was used to irradiate the specimens for 10 s on each side. All specimens were moulded into an acrylic and stored with distilled water for 24 h before grinding for cross-sectional imaging. The surface morphology of all treated specimens and the interface morphology of the bonded specimens were observed by field-emission scanning electron microscopy.

RESULTS AND DISCUSSION

POWDER ANALYSIS

The TEM image of 3YSZ nanoparticles from 2 different manufacturers shown in Figure 3(a) & 3(b), under 100 nm magnification which shows the size of 3YSZ power are accurate as the claimed by the manufacturer where the 30 nm powder shows a uniform spherical shape compared to the 90 nm powder. Observation on the particles size is important because it can infuse the particles to aggregate. The agglomeration occurred between smaller particles size to form bigger and stable particles due to attractive forces (Henry et al. 2013) because the low surface energy from the small specific surface area to attract other particles (Afuza et al. 2020). Since the sample of 30 nm size powder has a smaller average particle size compared to the sample of 90 nm size powder, it has higher possibility to agglomerate.

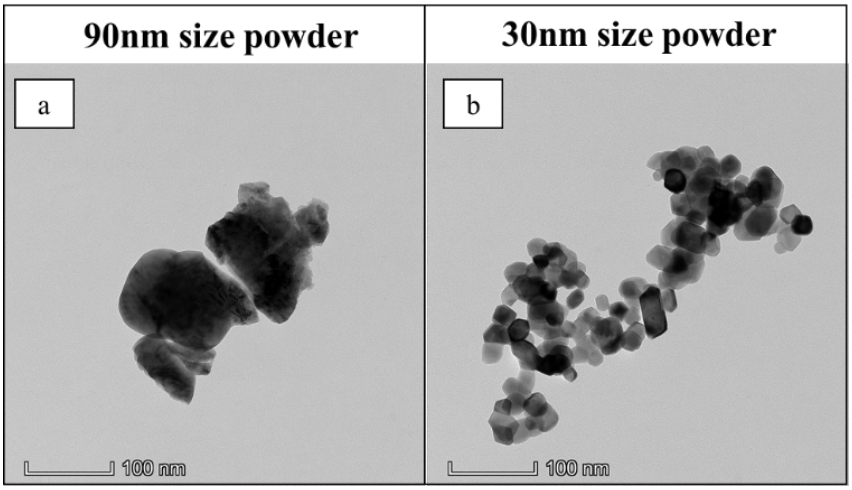


FIGURE 3. TEM image of 3YSZ nanoparticles (a) 90nm (b) 30 nm

The NPSA was used to determine the uniformity level of the overall particle size to determine the particle size distribution. The Z-average obtained from the analysis is referred to the intensity weighted mean hydrodynamic size of the ensembled collection of particles measured by dynamic light scattering, and while the Polydispersity Index (PdI) is the width parameter. The Z-average particle-size distributions of the 90 and 30 nm zirconia powders were 575.8 and 359.2 d.nm, respectively, and the PdI values were 0.36 and 0.40, respectively. A lower PdI indicates a more homogeneous sample with particles that are more uniform in size. Inconsistency in particle size could lead to different rates of energy absorbed by the particles to aggregate to form a uniform grain size. Even though a considerable difference can be observed between the particle sizes, no significant difference can be found in the PdI of both samples.

Figure 4 shows the results of analysis using ImageJ software to determine the grain size by mean standard deviation. The 30 nm zirconia powder had a lower average grain size of 323.38 (±145.10) nm than the 90 nm powder, with an average grain size of 362.11 (116.70) nm measured on the same scale. The homogeneity in the grain size of the 90 nm powder shown in Figure 4 was a result of the bigger surface area of the powder particles and lower PdI, which allowed for consistent energy to be absorbed during sintering. The 30 nm zirconia powder had higher agglomeration than the 90 nm zirconia powder due to its smaller and inconsistent particle size that led to the nonhomogeneous grain size. However, it potentially increases the bonding durability, because a reduction in grain size could enhance the degree of bonding between a composite resin cement and a dental zirconia due to the increase in interfacial free energy (Jeon et al. 2020).

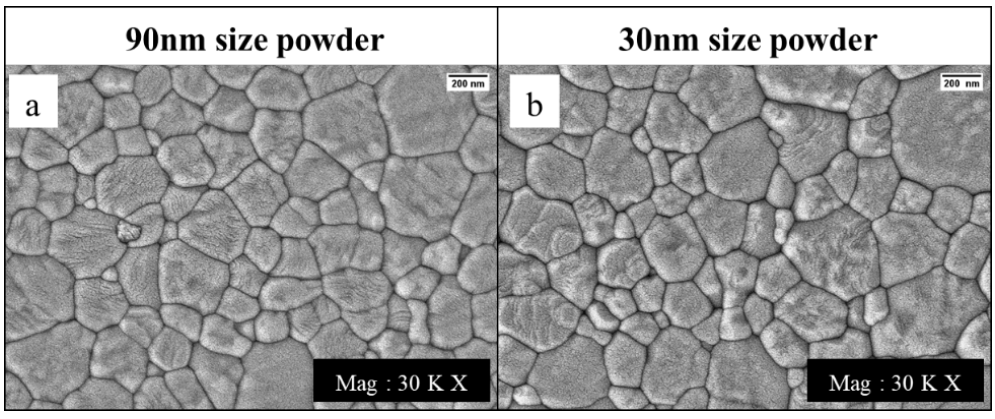


FIGURE 4. SEM of fully sintered zirconia block (a) 90 nm (b) 30 nm powder

SURFACE ANALYSIS

ANALYSISThe tooth is divided into two sections, enamel and dentine. Enamel is the outer layer of the tooth, and is the hardest substance in the human body and comprises rows of hydroxyapatite (calcium and phosphorus salts) embedded in a protein matrix. Dentine makes up the majority of the tooth. It consists of mineralized connective tissue. In dental restoration, removing the enamel is necessary before restoration of zirconia crown or bridge. Figure 5a reveals the exposed dentine surface (red circle), and Figure 5b exhibits the SEM image of the untreated dentine surface. The smear layer entirely covered the exposed dentine surface. The smear layer could create a border between dentine and the resin to bond properly and decrease the bonding strength between these two compounds. In clinical practice, phosphoric acid etching is used to remove the smear layer completely. The acid acts as a demineralizing agent that removes the smear layer and allows the cement to penetrate into the open dentinal cavities Figure 5c shows the dentinal tubule occlusion on the dentine after the smear layer was removed using phosphoric acid.

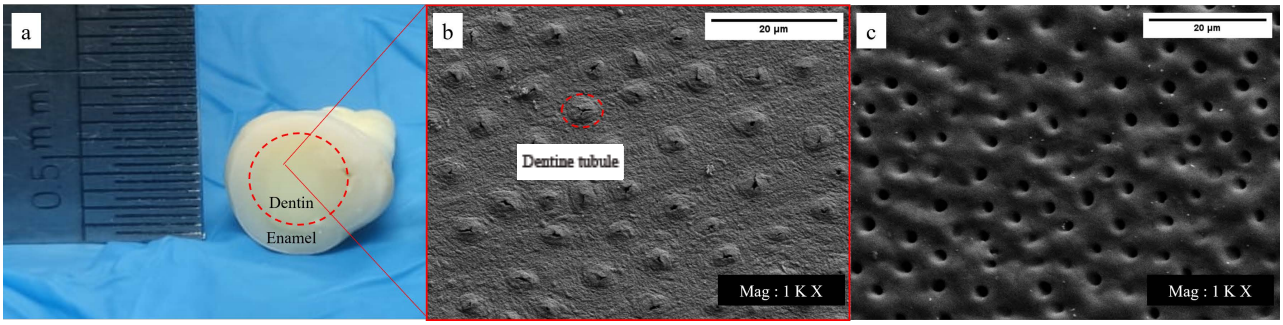


FIGURE 5. (a) Exposed dentine & enamel section (b) SEM of the dentine surface (c) SEM image of exposed dentine surface after remove smear layer (Prabhakar et al. 2013)

Figure 6 displays the SEM images of the effect of surface treatment air abrasion. The SEM image of the 90 nm powder with and without surface treatment showed no significant difference with the SEM image of the 30 nm powder. The pattern on the surface of zirconia block that underwent surface treatment represented the roughness effect after the alumina particles of air abrasion hit the surface, leaving it with a marked and cracked surface. This crack surface can expose the block with higher surface area to increase the bonding durability.

The average surface roughness (Ra) values of zirconia samples with and without surface treatment are shown in Table 1. A significant difference was found between the surface treatment group and the non-surface treatment group. The groups without surface treatment showed low Ra values. However, the group with surface treatment demonstrated a significant improvement because the original surface of the zirconia was altered after air particle abrasion. The rough surface of zirconia can help the resin adhesive to provide contact areas and micropores, thereby increasing the bond strength (Lin et al. 2021). Moreover, the surface roughness properties are important parameters for facilitating surface wettability that could increase the bonding reliability (Wongsue et al. 2023). Thus, surface treatment is essential to establish a reliable resin–zirconia bond strength (Figure 7).

TABLE 1. Average surface roughness of zirconia samples

Powder size	Surface treatment	Average Surface Roughness (μm), Ra
90 nm	No treatment	0.05
	Air abrasion	0.17
30 nm	No treatment	0.06
	Air abrasion	0.18

ZIRCONIA-DENTINE BONDING ANALYSIS

The zirconia block exhibited interlocking between the cement and the zirconia surface when subjected to air abrasion, as shown in Figure 7(b). Figures 7(a) and 7(c) show the flat bond between the zirconia and the cement in the non-surface treatment group. The rough surface of the zirconia with surface treatment allowed the cement to be diluted on the exposed area and increased the bonding strength due to the increased contact area between the cement and zirconia. Figure 7(d) shows the inconsistent thickness of the cement due to human error in sample preparation during the bonding process. The different thicknesses of cement may affect the bond strength.

The SEM image also shows the unbonded dentine-cement. Figures 7(a)–7(c) show the gap between the cement and dentine resulted in the improper adhesion between these two compound. The dentine and cement were separated during the cross-section preparation analysis due to external force. However, this happened due to the existence of a smear layer and an insufficiently rough surface on the dentine. Although self-adhesive resin cements consist of acidic monomer, the use of a primer on the dentine is essential to enhance the bond strength of zirconia to the tooth (Dawood & Ibraheem 2015) because the monomer with phosporus acid group in the cement are not effectively to react on the dentine. Thus, the removal of smear layer is a must to expose the collagen fibres, which can be achieved with the rinse and etch protocol on the dentine surface by using 35% phosphoric acid. The demineralisation of the smear layer will allow the micromechanical interlocking of resin monomers that diffuse into the exposed fibre while the use of primer will form the hybrid layer that resulted in higher bond between the cement and dentine (Elnawawy & Elkaffas 2023; van Meerbeek 2008). Thus, in future studies, the use of dentine primer is essential to ensure the dentine–cement bond. A mechanical bond test must be conducted to clarify the bond strength between the zirconia and dentine.

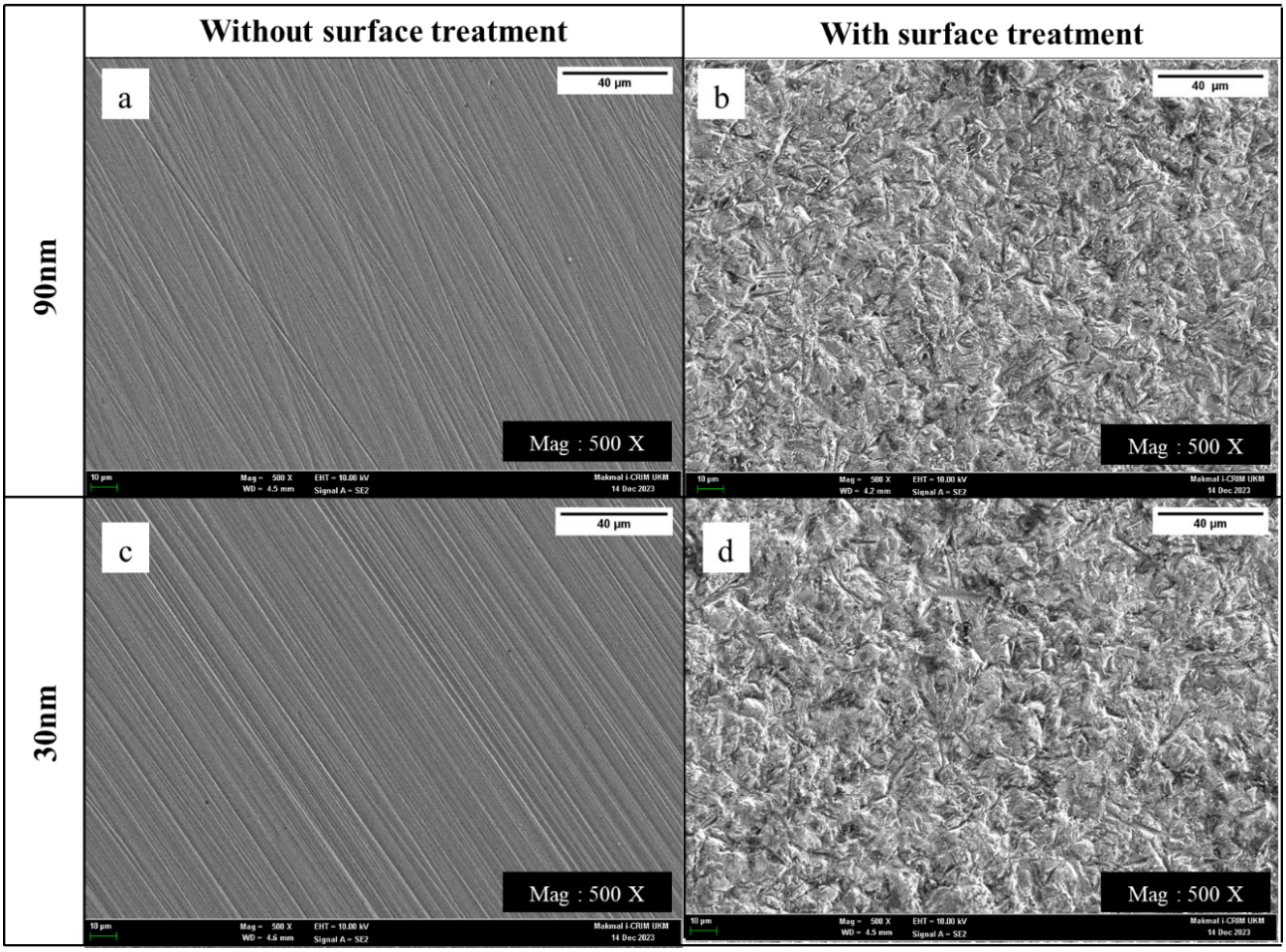
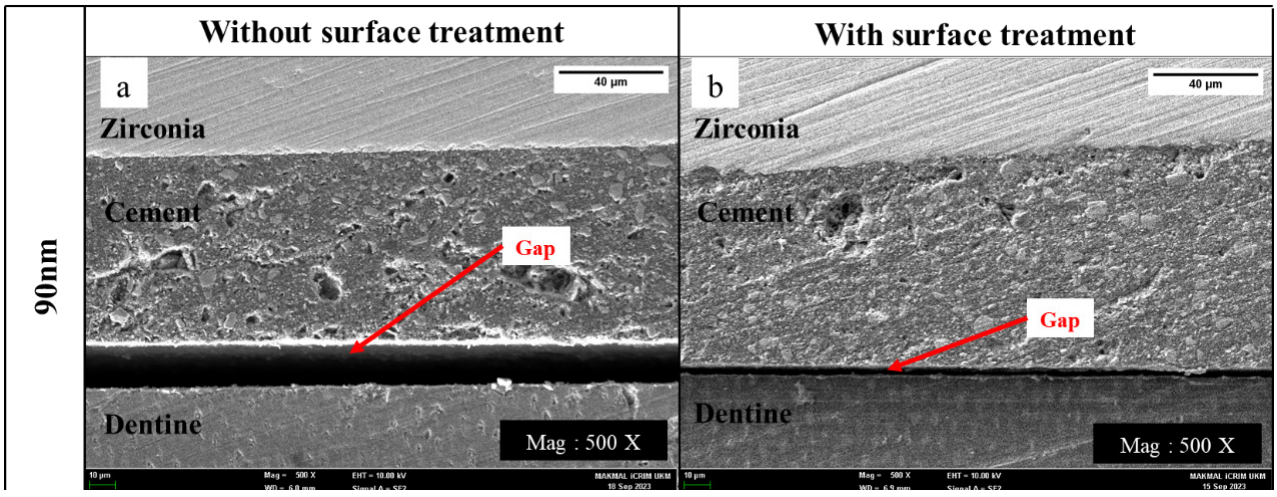


FIGURE 6. SEM image of zirconia surface (a) without surface treatment 90 nm powder, (b) with surface treatment 90 nm powder, (c) without surface treatment 30 nm powder, (d) with surface treatment 30 nm powder



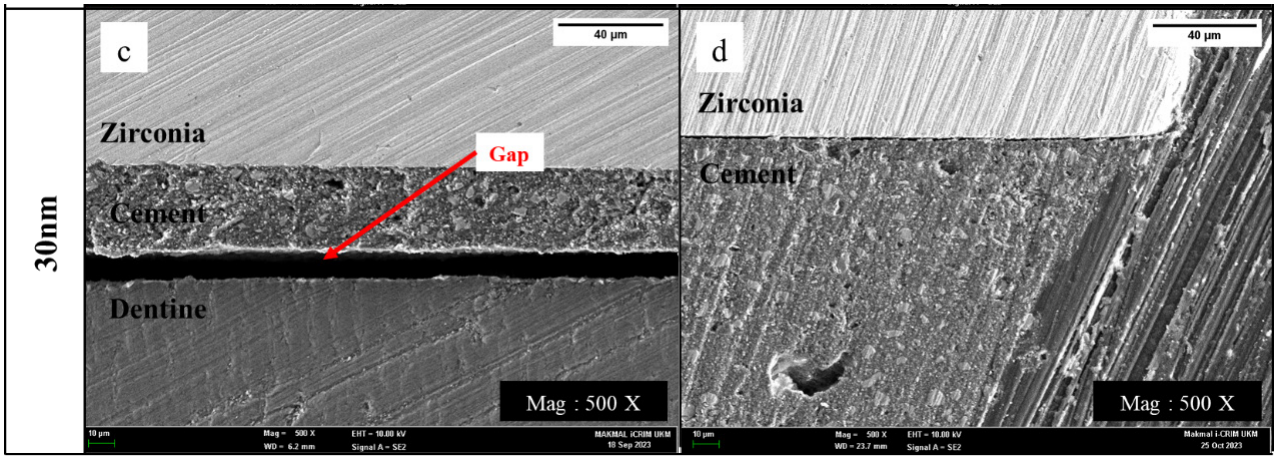


FIGURE 7. SEM image of zirconia-dentine bonding interface (a) without surface treatment 90 nm powder, (b) with surface treatment 90 nm powder, (c) without surface treatment 30 nm powder, (d) with surface treatment 30 nm powder

CONCLUSION

Within the limitations of this study, the following conclusions were drawn:

1. The different particle sizes of powder affect the homogeneity of the grain size. Small and inconsistent particle sizes can cause agglomeration between particles.
2. The different particle sizes of powder do not affect the surface morphology. However, surface treatment can change the surface morphology significantly.
3. Surface treatment creates a retention on the zirconia surface that allows micro-interlocking between cement particles and zirconia.
4. The acidic monomer in the self-adhesive resin is insufficient to remove the smear layer on the dentine surface. It requires pretreatment, such as using an etchant, to enhance the overall bonding durability.

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DECLARATION OF COMPETING INTEREST

The authors declare that they have no known competing finan-cial interests or personal relationships that could have appeared toinfluence the work reported in this paper.

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