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The Effect of the Pre-treatment by Hydrofluoric and Nitric acid in combined and sandblasting on the retentive force of monolithic zirconia with different yttria content

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ABSTRACT

Background: Zirconia is chemically stable biocompatible material used in dentistry. Current study aimed to evaluate the effect of yttria content (5Y, 7Y & 9Y) in combination with (Hydrofluoric and Nitric Acid) and silica sandblasting on the surface structure of crown to achieve mechanical retention. *Methodology:* From April 2021 to November 2021 study was conducted on 54 maxillary premolars, and zirconia crowns with planned and constructed proximal extensions. Based on yttria content samples were divided in 3 groups (n=16) ceramics Copran Zri (5Y), Copra Supreme (7Y) and Copra Smile (9Y). Each group is subdivided in to three pretreatment subgroups (n=6), followed by specimen characterization via scanning electron microscopy (SEM). Targeted zirconia crown pre-treated before cementing to the associated tooth. The test of crown retention was recorded in Newtons and performed to displace the crown. The mechanical strength (MPa) was determined. *Result:* In Yttria concentration group the retentive strength of various zirconia crowns was found to be significant $p<0.05$. The Post Hoc test revealed that group 1 having the greatest mean retention strength value (5YS1). There is significant difference ($p<0.0001$) in retention strength and surface pretreatment observed among group 3 and 2. *Conclusions:* With the combination of air abrasion and hot acids, the 5Y group demonstrated the greatest retentive force. The study discovered an inverse relationship between the quantity of Yttria content and the retentive force, whereas pre-treatment with a hot acid air abrasion increased the retention of zirconia crown.

Keywords: Restorative material, Prosthetic material, monolithic crown, zirconia surface treatment.

DISCOVERY SCIENTIFIC SOCIETY

1. INTRODUCTION

The optimal physical, mechanical and biocompatible properties of zirconium oxide, combined with advances in computer aided design and computer aided manufacture technologies, have contributed to its aggregate the use as a dental restorative material. The phase change toughness at room temperature (meta) stabilizes the zirconia at room temperature, which increases strength, depending on the type and amount of stabilizer used (Zhang et al., 2019). The highest strength dental zirconia contains approximately 3% yttria. Despite recent developments, 3YTZP restorations are opaque and often need to be veneered with a glass ceramic (Zhang et al., 2019; Zhang & Lawn, 2018). To prevent the veneering ceramic from chipping in two-layer restorative systems before it becomes a problem and to allow less intrusive preparation of dental hard tissue through adhesive cementation with different types of cement light-curing composite (Azeem & Sureshbabu, 2018). The demand for monolithic ceramic restorations is increasing Zirconium oxide ceramic with high transparency. The most reliable method of increasing the translucency of yttrium oxide-containing zirconia ceramics is to incorporate optically isotropic zirconia in a cubic phase and less birefringent tetragonal zirconia and minimize light scattering in secondary phases using aluminum oxide particles (Saran et al., 2017). Nowadays, the most used zirconia is the higher yttria content 4-5 mol% (Yu Zhang, 2014).

Zirconia is classified according to yttria content, three moles of YTZP (mainly tetragonal), four moles of YTZP, and five moles of YTZP with impaired mechanical properties. The yttrium oxide content reduces the grain size of zirconium oxide and increases the coefficient of thermal expansion (Yu Zhang, 2014). Zirconia materials (4YTZP and 5YTZP) were synthesized with a higher yttria concentration (6 Y2O3 10% by weight) and increased translucency than 3YTZP. As a result, 5YTZP is gaining popularity as a monolithic restoration that does not require ceramic veneers. due to thinner preparation, higher patient satisfaction, and reduced production time and costs (Kim, 2020). Therefore, the influence of these modifications on the bonding of the adhesive cement and the maintenance of the tooth structure has not been thoroughly investigated. Another problem is that the chemical composition and mechanical properties of traditional zirconia and highly translucent zirconia are very different (Kontonasaki et al., 2020), which affects the relative effect of particle abrasion in air.

The strength of traditional zirconia increases with the abrasion of airborne particles as the phase change near the surface increases. However, it is assumed that highly transparent zirconia does not undergo phase transformation and has lower strength Zirconium ceramics cannot be scratched with conventional acids because they do not contain silicates (Sulaiman et al., 2017; Inokoshi et al., 2018). Consequently, hot acid etching and piranha solution techniques are used to condition the zirconia (Guazzato et al., 2005; Uhrenbacher et al., 2014). Different variable affect the retention of the prosthesis such as the tooth precreation design (Guazzato et al., 2005), fitting of the prosthesis itself (Yenisey et al., 2016) and the cement used (Grasel et al., 2018).

Dental cements must have high tensile strength to adhere firmly to the fitting surface and the prosthesis. The mechanism of retention based either on rubbing, micromechanical interactions, chemical bonds, or a mixture of all (Hill, 2007). Self-adhesive resin cements are becoming popular due to strong adhesion, ease of use, and saving time (Yu Zhang et al., 2004; Han et al., 2007). Earlier researches have shown that the use of resin cements containing MDP gives acceptable results (Quigley et al., 2021; Sakrana et al., 2020). Retention tests have been used in many studies, where cemented reconstructions are subjected to axial displacement forces prior to failure (Ali et al., 2012). This in vitro study aimed to determine the influence of the yttrium content (5Y, 7Y and 9Y) of the ceramic material and the combined pretreatment of sandblasting and hot acid on the preservation of monolithic zirconium oxide restorations. The null hypothesis demonstrates that different yttria content does not affect the retention. The other null hypothesis demonstrates that the combination of sandblasting and hot acids could improve the retention of the zirconia crown.

2. METHODOLOGY

Selection of dental tooth

The current study involved 54 maxillary first premolars that had been extracted for orthodontic reasons. Each impacted tooth is free from cavities, fissures or fractures. Faculty of Dentistry Al-Azhar University, Egypt, the study starts from April 2021 to November 2021 and has obtained approval for the use of human teeth from the Research Ethics Committee (No. AUAREC20210315-11). All teeth were measured to ensure that only teeth of comparable size (buccolingual and mesiodistal) were included (Fernández-Estevan et al., 2017). Each tooth was debrided and cleaned before being stored at room temperature in standard saline solution. All teeth (n = 18) were divided into groups based on the type of ceramic used (table 1). To eliminate tooth movement during the retention test, each tooth root was quickly brushed with a carbide bur to facilitate assign of orthodontic bracket. Each tooth is placed in the center of the acrylic resin block (table 2).

Table 1 According to data published by manufacturers and literature sources, materials, codes and configurations of zirconia ceramics used.

#	The name of Zirconia	Material code	composition
1	CopranZri HT	CZI, group 1	ZrO_2, 4.95–5.35% Y_2O_3, 0.15–0.35% Al_2O_3, Fe_2O_3 < 0.01%, Other <0.06%
2	CopraSupreme	CSP, group 2	ZrO_2, 6.93–6.97% Y_2O_3, 0.05% Al_2O_3, Fe_2O_3 < 0.01%, Other <0.02%
3	CopraSmile	CSM, group 3	ZrO_2, 9.92% Y_2O_3, 0.15–0.35% Al_2O_3, Fe_2O_3 < 0.02%, Other <0.02%

Table 2 A schematic representation of the study grouping

	Group (n=18)	Study code	Subgroup (n=6)	Measurements and characterization
Specimens' numbers = 48	CopranZri HT (5Y)	5YS1	Crowns milled from Copran Zriultra translucent Zirconia abrasively blasted with aluminium oxide particles.	Retentive force Scanning electron microscopy
		5YS2	The crowns etched using the hot acid (49% hydrofluoric acid and 69% nitric acid) (1:1)	
		5YS3	Crowns machined from highly translucent CopranZri Zirconia were abraded with aluminium oxide particles and then etched with hot acid (49 percent hydrofluoric acid and 69 percent nitric acid)	
	CopraSupreme (7Y)	7YS1	Crowns milled from CopraSupreme zirconia were subjected to air abrasion with aluminum oxide particles.	
		7YS2	The crowns etched using the hot acid (49% hydrofluoric acid and 69% nitric acid) (1:1)	
		7YS3	The crowns milled from Copra Supreme	
			Translucent zirconia was air abraded using aluminium oxide particles then etched using the hot acid (49% hydrofluoric acid and 69% nitric acid).	

	CopraSmile (9Y)	9YS1	The crowns milled from Copra Smile zirconia were air abraded using aluminium oxide particles	
		9YS2	The crowns etched using the hot acid (49% hydrofluoric acid and 69% nitric acid) (1:1)	
		9YS3	crowns milled from Copra Supreme translucent zirconia were etched with hot acid after being air abraded with aluminum oxide particles (49 percent hydrofluoric acid and 69 percent nitric acid)	

Designing of tooth model

The standardize dental preparation was achieved via CAD CAM technology (Bergamo et al., 2019) with the help of a dental professional, preparation of a single tooth. The preparation procedures include a centered cusp reduction of 1.5mm, a non-centric cusp reduction of 1 mm, a chamfer of 0.5mm and a taper of 6 degrees. After scanning the teeth with an optical scanner, the scanned data was edited using Ceramill Amann Girrbach software. As a result, the designed model was completed before being replicated. Each prepared tooth was scanned and a gap of 90mm of cement was chosen. In order to remove the restoration after cementation during the retention test, the crowns made a protrusion on the mesial and distal aspect.

Preparation of the specimen

Current study utilized three commercially available (72*90*14mm) disc-shaped pre-sintered YTZ blocks (Copran Zri, Copra Supreme, Copra Smile; Whitepeaks Dental Solutions Gmb H & Co KG) listed in Table 2. The crowns were manufactured using a CAD CAM milling machine (Ceramill Motion 2, Amann Girrbach). According to the manufacturer's manual, the crwons sintered then glazed (high-speed sintering; Ceramill Therm RS, Amann Girrbach). The STL files of each prepared tooth were edited using professional 3D computer graphics software (MeshLab software, Institute of Science and Information Technologies, Rome, Italy) to define the surface area for the prepared tooth. All crowns were scraped with 250ɱ aluminum oxide at one bar for ten seconds from particles in air. Then the crowns cleaned and dried. The second subgroup zirconia crowns were freshly scraped with hot acid, each zirconia crown in third group was air scrubbed before being scratched with a one to one mixture of 49% hydrofluoric acid and 69% nitric acid (Liu et al., 2015).

The acid mixture then injected into the internal surface of the crown and burned for 25 minutes at 100 °C. The crown was then cleaned and dried. Panavia SA (Kuraray Noritake Dental Inc., Okayama, Japan) was used for cementation according to the manufacturer's guidelines. Each restoration was under a continuous pressure of 10 N. Finally, each surface is illuminated for 20 seconds (Bergamo et al., 2019). To mimic the intraoral environment, thermocycling was used on semen samples (SD Mechatronics, Westerham, Germany). The thermocycle is carried out for 7,000 cycles between 5 and 55 degrees Celsius (25 seconds residence time). After that each, prosthesis was exposed to axial displacement stresses using a universal testing machine (Instron, UK) with a crosshead speed of 0.5mm/min till it fail. The sample is fixed to the bottom fixed part of the device by means of screws. The prosthesis adjourned from the top of the moving device using a specially designed tool to adjust the projection of the crown. The breaking load expressed in Newton (N). Each prosthesis examined at 20X magnification after retention test by a single operator (OH) using an Olympus SZ 61 (Tokyo, Japan), Adhesive (interface with cement), cohesive (in restoration or tooth substructure) or mixture.

Characterization of the surface

After gold powder coating, the surface morphology of the sintered samples was investigated by using ESEM microscope (FESEM; JEOL, Japan). Statistical analyses were done by using SPSS Statistics for Windows v26.0 program SPSS Inc., Chicago, IL, USA. The Shapiro Wilk test revealed that the data distributed normally (df = 10, $P > 0.05$ for each group) in terms of retention power.

3. RESULTS

The most retentive mean was observed in Group 1 (5Y) followed by group 2 (7Y), then group 3 (9Y) (Table 3). In terms of the retention strength of different zirconia crowns relative to their Yttria concentration, a statistical difference ($p < 0.05$) was found among the groups using the Post Hoc test, with group 1 being the mean value of highest retention resistance (5YS1) had. In addition, a significant difference between groups 2 and 3 was identified ($p < 0.0001$), with group 2 having a stronger retention force. Regarding the surface pretreatment, there was a statistical difference within the groups ($p < 0.05$), with the highest mean value of retention resistance for S3 (air wear followed by hot acid etch), followed by S1 (air wear) by S2 (hot acid attack).

The two-way ANOVA test showed a statistically difference influence ($p < 0.05$) of the variation in the Yttria content on the mean of the holding force. In addition, a statically difference ($p < 0.05$) was observed for the nature of pretreatment surface. In addition, the relationship between the surface treatment and the Yttria content is significant ($p < 0.05$) (Fig.1; Table 4).

Table 3 The means and standard deviations (SDs) of retention strength (MPa) of all groups

Groups	Air abrasion with AL2O3(S1)	Etching with hot acids (S2)	Air abrasion followed by hot acids applications (S3)	P value
Group 1 (5Y)	3.44±0.25	3.02± 0.43	3.97± 0.23	0.05 SN
Group 2 (7Y)	2.98±0.44	2.90±0.67	3.00±0.31	0.23 NS
Group 3 (9Y)	2.22±0.12	2.10±0.11	2.56±0.43	0.06 NS

Figure 1 The milled zirconia crown with two wings.

Table 4 Two-way ANOVA test concerning the yttria content and surface pretreatment on the retention strength of zirconia crown

Tests of Between-Subjects Effects

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
yttria content	12.676	2	6.338	45.67	0.004
surface pretreatment	2.337	2	1.168	8.54	0.05
yttria content * surface pretreatment	1.105	4	.276	2	0.036
Error	6.145	45	.138		
Total	473.744	54			

Table 5 The failure mode of all tested groups

GROUP CODE	adhesive	cohesive	mixed
5Y (S1, S2, S3)	2	3	13
7Y (S1, S2, S3)	4	4	9
9Y (S1, S2, S3)	6	5	7

Through multiple comparison tests between subgroups, the mean 5Y group differed significantly in the different surface preparations. Group 7Y showed no statically difference among the different pretreatment surface, while in group 9Y the significant difference is related to air abrasion and hot acid treatment combined as compared to each alone Fig 2. The primary failure mode for group 5Y with different surface treatment is mixed failure mode followed by lower group Y7. No stickier and cohesive break with the 9Y group, failure modes of all tested samples are shown in (Table 5).

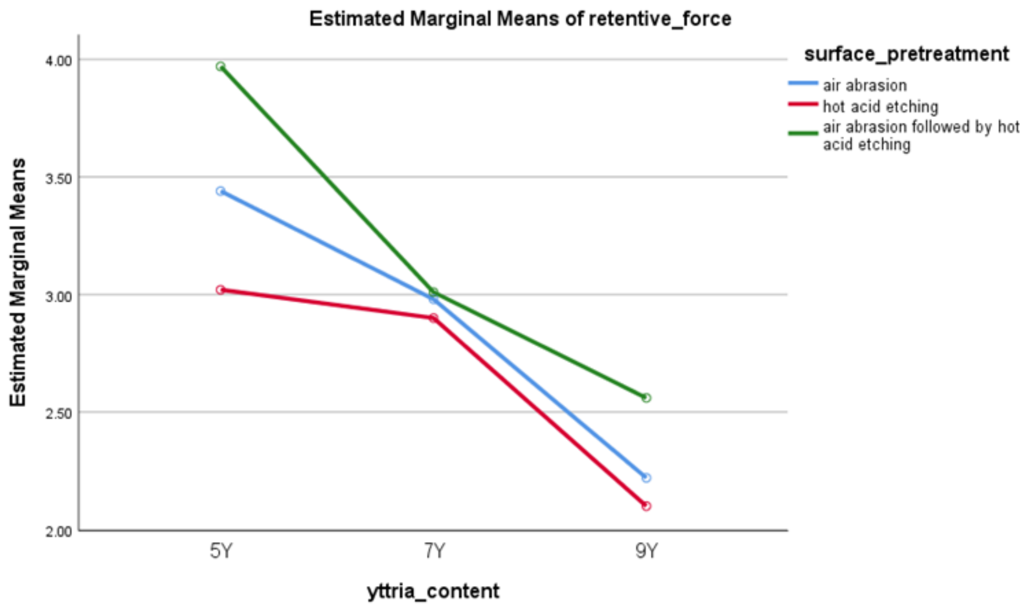

Figure 1 The graphical representation showing difference among the three yttria content groups combined with the surface treatments methods (Blue, red and green lines).

Scanning electron microscope images of samples showed rough surfaces with multiple pores increased by 5Y and more gaps in 9Y groups due to air abrasion, for hot acid treatment only (Fig. 3). Pores and spaces increased from 5Y and 7Y and appeared with cracks in the 9Y samples. The combined surface treatment by air wear and hot acid etching produced the highest effect, pores and cracks in 5Y samples, multiple fiber networks and depths in 7Y, but the in 9Y samples revealed islands of particles separated from huge pores and caves (Fig. 3, 4, 5).

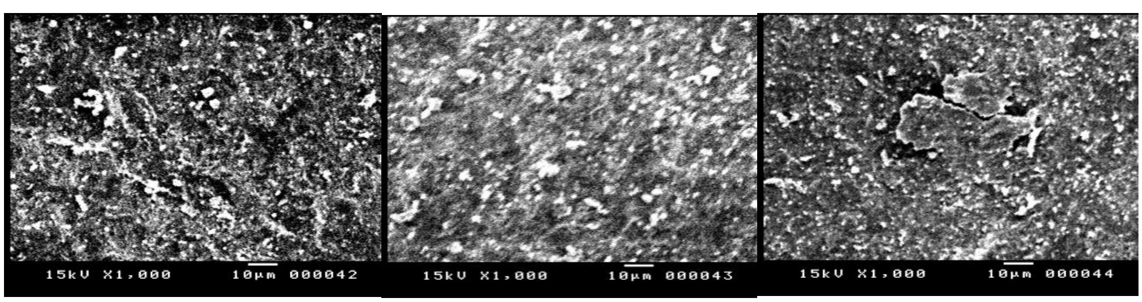

Figure 2 The representative samples of three groups of Zirconia containing 5Y, 7Y, and 9Y under the scanning electron microscopy at 1000x magnification after air abrasion by alumina particles size 110 µm.

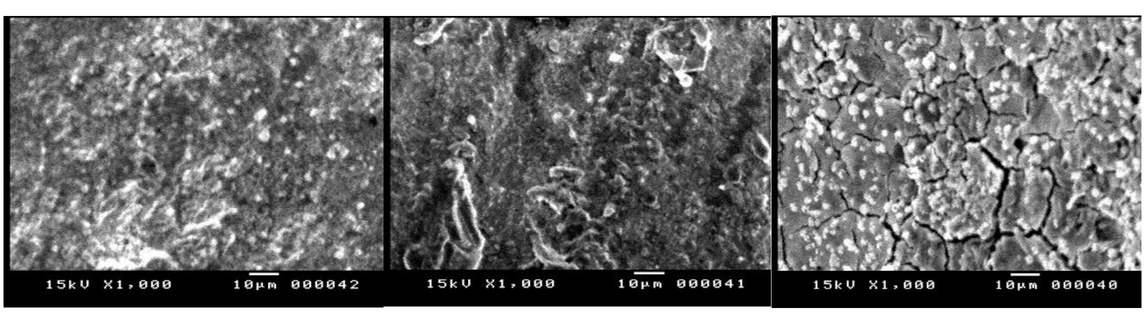

Figure 3 The scanning electron microscopy images of three groups of Zirconia containing 5Y, 7Y, and 9Y at 1000x magnification after treated by hot air acids [49% hydrofluoric acid and nitric acid (1:1)] at 100°C for 25 minutes.

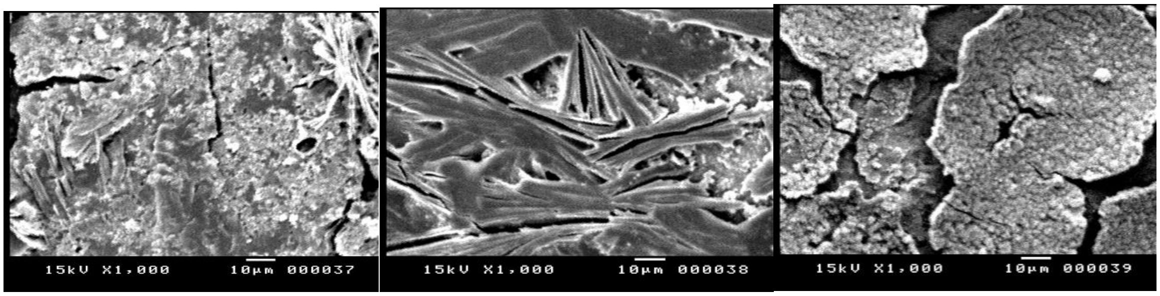

Figure 4 The scanning electron microscopy images of three zirconia groups according to Yttria content (5Y, 7Y, and 9Y) at 1000x magnification after air abrasion then hot acids at 1000x

4. DISCUSSION

The first null hypothesis states that hot acids and sandblasting improve the retention of the YTZ crown. The second null hypothesis states that the percentage of yttrium would not affect the retention of the zirconium crown. This study showing, hot acid etching increased the retention of the crown made by zirconia. Moreover, the Y content affected the retention of zirconium crowns. In this study, human teeth were chosen for their more clinically relevant bonding properties, thermal properties, modulus of elasticity and strength (Liu et al., 2015). Teeth were prepared using CAD CAM technology as it allows a uniform design for all teeth in vitro testing and delivers a reliable procedure used in studies to facilitate comparisons and eliminate biases caused by inconsistencies in sample processing (Kasem et al., 2020).

Regarding the retention strength of the three zirconia crowns (variable yttrium concentration), this study found that the 5Y sample had the highest average retention strength, followed by the 7Y and 9Y samples, which is consistent with previous studies. earlier studies, have shown that an increase in the yttrium content to 5 mole percent (as seen in the YTZP 3rd generation material) results in decreasing the mechanical properties and increase the cubic phase up to 50% (Burgess, 2018). Fully stable zirconia is a mixture of metastable and cubic tetragonal phases. While this increases translucency and resistance to aging, the mechanical quality decreases, as Zirconia cannot transition from the tetragonal phase to the monoclinic phase (Kontonasaki et al., 2020). An increase in the concentration of yttrium oxide resulted in a decrease in the retention force in this investigation. This can be explained by increasing the yttrium oxide content and grain size in the cubic phase to improve the optical characteristics leading to a decrease in the tetragonal phase (Zhang et al., 2019). The tetragonal phase is critical for transition hardening of zirconia, which occurs when the crack tips because transforming the tetragonal grains into monoclinic grains with a 3%-5% of local expansion. This increase in volume induces a compressive force at the crack tip, which acts in opposition to the external tensile stress, thereby slowing crack propagation. This is consistent with a study examining the fracture strength of zirconia ceramics with various levels of yttrium oxide and determined that increasing the percentage of yttrium oxide can decrease the mechanical quality of ceramics during aging (Ali et al., 2012).

In terms of surface pretreatment, air abrasion and hot acid etching groups had the highest retention force, followed by air abrasion samples and lastly only hot acid etching samples. This is in line with the results of the study (Zarif Najafi et al., 2015), which showed that the adhesion value of Zirconia Copran and Zirconia Katana ML abraded with air particles was better than untreated Zirconia but comparable to Zirconia Metoxit ZCAD HTL abraded with air particles. The lowest values found particularly in the 5Y group, while the difference between the other two groups was not significant. This may be due to the fact that hot acid etching protocols used in this study was carried out according to (Kasem et al., 2020), who confirmed that etching zirconia using a 1:1 mixture of 49% HF acid and 69% HNO3 was effective.

SEM examination showed that the surface of the zirconia appeared rough with irregular grooves due to wear of particles in the air (Harb et al., 2021). However, hot acid etching due to particle abrasion in air increases roughness due to the wide distribution of pore networks with varying depths and widths. These results are congruent with (Kasem et al., 2020), who found that hot acid etching disintegrate the zirconia grain, creating multiple craters of various sizes that increase the surface roughness of the zirconia compared to air abrasion. Compared with the combined group, the roughness is increased, the large pores and indentations are increased, and the blocks are separated. This may result in cracking and weakening of the zirconia crown, because the value of the retention force when combined with the yttrium content is lower than when air abrasion or hot acid alone is used. This could be due to the deepening of the cracks and caves.

5. CONCLUSION

The 5Y group showed the highest retentive force with the combined air abrasion and hot acids. Study observed indirect relation among amount of yttria content and the retentive force whereas, pre-treatment with a hot acid and air abrasion increases the zirconia crown retention. We recommend further clinical study to confirm these in vitro results especially with different types of zirconia.

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Ethical approval

The research proposal was approved by the Regional Research and Ethics committee Al-Azhar University, Egypt, with Ethical approval number (No. AUAREC20210315-11).

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Conflict of interests

The authors declare that there are no conflicts of interests.

Data and materials availability

All data associated with this study are present in the paper.

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