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RETENTION OF ZIRCONIUM OXIDE AND RESIN NANO-CERAMIC COPINGS CEMENTED WITH TWO DIFFERENT ADHESIVE SYSTEMS

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ABSTRACT

Objectives: The aim of this in-vitro study was to evaluate the influence of two types of cements on the retentive force of Zirconia and resin nanoceramic copings.

Methods: Thirty two freshly extracted maxillary premolars were collected and prepared in a standardized manner. The prepared premolars were randomly divided into two equal groups (n=16) according to the material type of the constructed coping. Group I (LZ): Sixteen prepared premolars were restored with yttrium partially stabilized zirconia (Lava Zirconia). Group II (LU): sixteen prepared premolars were restored with resin nano-ceramic (Lava Ultimate). Each group will be further subdivided into two equal subgroups (n=8) according to the type of the cement used for cementation. Subgroup A (RXU): the copings were cemented with self-adhesive resin luting cement (RelyX UniCem). Subgroup B (RX ARC): the copings were cemented with a total etch adhesive resin cement (RelyX ARC). The copings were designed with an outer cylindrical handles located on buccal and palatal surfaces to provide a mean for removal of the coping during the pull-out testing. The cemented copings were thermal cycled at 5° C and 55° C for 2000 cycles and then removed along the path of insertion using a universal testing machine at 0.5mm/min. The retentive force was recorded, and the stress of dislodgement was calculated using the surface area of each preparation.

Results: The mean dislodgement stresses were 6.03MPa for group I (LZ) and 4.66MPa for group II (LU) where there was a statistically significant difference and LZ showed the higher value. Regarding the type of the cement, there was no statistically significant difference between RelyX unicem (mean=5.32MPa) and RelyX ARC (mean=5.73MPa) for the two coping materials. However RelyX ARC showed a slightly higher value.

Conclusions: Retention of Lava zirconia is better than Lava Ultimate regardless of the cementation technique and material used whether it is self-etch or total-etch.

KEYWORDS: Retention, Lava Zirconia, Lava Ultimate, Self-adhesive resin cement, Pull-out testing, resin nanoceramics, yttrium partially stabilized zirconia.

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INTRODUCTION

The popularity of all-ceramic restorations has increased due to superior esthetic appearance. Over the last years several all-ceramic systems have become established on the market.[1] Computer aided design and manufacturing (CAD/CAM) has become an increasingly interesting alternative to the conventional casting or pressing techniques.[2] The use of yttria partially stabilized tetragonal zirconia polycrystalline (Y-TZP) ceramics to fabricate metal-free esthetic restorations has been added to the routine prosthetic work[3]. The mechanical properties of zirconia are the highest reported for a dental ceramic. It presents high values of fracture strength (>1000MPa) and toughness (5-10MPa $m^{0.5}$) compared with other traditional ceramics [4,5,6,7]. Y-TZP ceramic has been introduced to restorative dentistry, based on these improved physical properties. CAD/CAM technologies have contributed to the ease of working with this high crystalline material, allowing the fabrication of frameworks for complete coverage all-ceramic crowns and fixed partial dentures. [8,9]

A new resin nanoceramic, recently introduced by 3M ESPE, called Lava Ultimate CAD/CAM restorative, has a unique chemical composition combining the advantages of highly cross-linked, heat-cured composite combined with nanoceramic technology for strength and polish retention. This truly hybrid material has several proposed advantages as ease of use, tooth-like properties, durability and aesthetics. [10]

Successful cementation of ceramics is important for clinical success of the restoration [11,12]. Y-TZP crowns and fixed partial dentures have high fracture resistance and can be cemented using conventional methods recommended by the manufacturers. [13] However, resin bonding between a dental substrate and a restoration is favorable for improved retention, marginal adaptation,[11, 12] prevention of microleakage, and increased fracture and fatigue resistance.

Strong resin bonding relies on micromechanical interlocking and adhesive chemical bonding to ceramic surface requiring surface roughening for mechanical bonding and surface activation for chemical adhesion. [14] Presumably, sandblasting the cementation surface creates considerable roughness enabling a strong micromechanical bond with resin cement. [15] In addition strong chemical adhesion would lead to enhanced long term fracture and fatigue resistance in the oral environment. [14]

Restorations made with a low modulus of elasticity like Fiber-reinforced composites show an increased degree of debonding when used with luting agents that demonstrate low adhesive properties. [16] The adhesive properties refer to both the bond to the prepared tooth as well as the bond to the indirect material that covers the tooth. [17]

Resin based cements are widely used for luting inlays, crowns, and veneers because they adhere to metal and ceramics. However, teeth and restorations require surface treatments, such as etching and bonding, when conventional resin based cements are used,[18,19] making the luting operation technique sensitive. Thus, if the surface treatment is insufficient, the bond strength will be impaired. Self-adhesive cements do not require any surface treatment of the teeth or restorations, so they are easier to handle and have clinically effective bond strength. It is reported that self-adhesive resin cements provide the equivalent bond strength of conventional resin cements to dentin[20,21], gold alloy, glass ceramics[22], and zirconia. [23,24]

The adhesive properties of a luting agent to ceramics can be assessed using several laboratory tests. From the most common laboratory tests are bond strength tests, such as shear, tensile, microtensile, or push out tests. The advantages of bond strength tests, if designed properly, are the reproducibility of results within same test lab and the ease of conducting the test. The main criticism is that the bond strength test does not reflect the clinical situation. Therefore laboratories developed crown pull-off tests where crowns were luted to extracted human teeth to simulate the clinical procedure. [3] This testing procedure is complex and technique sensitive but provides information on retentive performance of a material. [25]

Thus, the aim of the present study was to determine the retentive strength of two different crown materials (Y-TZP and Lava Ultimate nanoceramic) using two different cementation protocols: The conventional resin based cements and self-adhesive resin cement. The null hypothesis tested was the retentive force of Lava copings is influenced by the cement bonding mechanism.

MATERIALS AND METHODS

A total number of thirty two freshly extracted maxillary premolars for orthodontic reasons were collected. The collected teeth were examined for absence of caries, restorations, and cracks. The teeth were selected to be of similar size and shape. The buccolingual and mesiodistal dimensions at the cemento-enamel junction, height of contours and the occluso-axial line angles were measured in millimeters using digital caliper (0-50mm, 0.01mm, Germany). The premolars were selected to be of two divergent roots. The teeth were cleaned from any surface debris and disinfected in 0.5% sodium hypochlorite solution, then stored in distilled water for maximum one month until the experiment began.

The roots of the selected teeth were notched for retention and embedded along their vertical alignment in an autopolymerizing acrylic resin (Acrostone, acrostone dental factory , Egypt) with the cement-enamel junction positioned 1mm above the top of the acrylic resin. The occlusal surface of each mounted tooth specimen was sectioned flat 6mm above the top of the acrylic resin using a diamond disc (Diamond discs 910P, Drendel+Zweiling DIAMANT Gmbh, Germany). All the samples were prepared for full coverage all-ceramic crowns. The finish line was designed to be chamfer finish line with 1mm depth placed 1mm above the cement-enamel junction. The preparation of all samples was performed using a coarse diamond stone (Drendel zweiling Diamant GMBH, Germany) at high speed with water spray, the diamond stone was specially designed with a central non-cutting shaft so that the thickness of the finish line can be standardized for all samples. The mesiodistal and buccolingual dimensions of the prepared samples were measured at the finish line, 3mm above the finish line and at the occluso-axial line angle using digital caliper. This was done to assure a standardized preparation in all the specimens. The final prepared tooth dimensions were 6mm height, 5mm mesiodistal diameter at the cervical margin,7mm buccolingual diameter at the cervical margin, 4 mm mesiodistal diameter and 6mm bucco lingual diameter at the occlusal circumference. All the samples of this study were prepared by the same operator.

The prepared teeth were randomly divided into two equal groups of sixteen teeth each, according to the material used for the construction of the coping. Group I (LZ): Sixteen prepared premolars were restored with yttrium partially stabilized zirconia (Lava Zirconia, 3M ESPE, Seefeld, Germany). Group II (LU): Sixteen prepared premolars were restored with resin nano-ceramic (Lava Ultimate, 3M ESPE, Seefeld, Germany). Each group will be further subdivided into two equal subgroups of eight teeth each according to the type of the cement used for cementation. Subgroup A (RXU): the copings were cemented with self-adhesive resin luting agent (RelyX UniCem, 3M ESPE, Seefeld, Germany). Subgroup B (RX ARC): the copings were cemented with a total etch adhesive resin cement (RelyX ARC, 3M ESPE, Seefeld, Germany).

The chemical composition and the manufacturers of the materials used in this study are presented in table 1.

Each specimen was scanned using an optical scanner (ZirkonZahn, Gais, Italy). Both types of

TABLE (1) The chemical composition and the manufacturers of the materials used in this study.

Material	Composition	Manufacturer
Lava Zirconia	- 3% yttrium oxide treated tetragonal zirconia polycrystals with a very small concentration of alumina (< 0.25 %) to prevent leaching of the yttrium oxide.	3M ESPE, Germany
Resin Nano Ceramic Lava Ultimate "LU"	Total nanoceramic material content is 80% by weight: -silica nanomers (20 nm), -zirconia nanomers (4 - 11 nm). -zirconia-silica nanoclusterparticles (0.6-10 μm). Highly cross-linked methacrylate-based resin matrix Silane coupling agent	3M ESPE, Germany
RelyX ARC	**Paste A:**zirconia/silica filler 68%, amine, photo-initiator, pigment **Paste B:** zirconia/silica filler 67%, benzoyl peroxide	3M ESPE, Germany
Rely X Unicem Resin Cement	**Powder:** - Alkaline (basic) fillers - Silanated fillers - Initiator components - Pigments **Liquid:** -Methacrylate monomers containing phosphoric acid groups - Methacrylate monomers - Initiator components - Stabilizers	3M ESPE, Germany

copings were designed using Modellier (ZirkonZahn, Gais, Italy) and milled using 5 Tec milling machine (ZirkonZahn, Gais, Italy) according to manufacturer instructions.

The restorations were designed to be copings without any added veneering porcelain. The coping thickness was adjusted to be 0.7mm. The copings were designed with an outer cylindrical handles located on buccal and lingual surfaces below the occluso-axial line angles by 2mm. The length and the diameter of the handles were 3mm and 3mm respectively. These handles were added, milled and sintered to the copings to provide a mean for removal of the coping during the pull-out testing (Fig 1).Cement space was adjusted to be of 40μm.

Prior to cementation, the areas of the axial and the occlusal surfaces of each prepared tooth were calculated. For each specimen, the boundaries of the buccal surface were marked as an area extending from mesiobuccal line angle to distobuccal line angle and from cervical finish line to the occluso-axial line angle using indelible pencil. The same was done for the palatal, mesial, distal and occlusal surfaces.

For each specimen, the area of interest was captured by CCD digital camera (DP10-Olympus, Japan) mounted on a stereo microscope. The area of each surface on each specimen was assessed at the pre-determined marked areas. These reading were calculated as an area using image analysis software (Image J- 1b, USA).

The sum of the areas of the mesial, distal, buccal, palatal and occlusal surfaces was calculated to obtain the total surface area of each prepared tooth.

In subgroup A (RXU), self adhesive resin cement with phosphate monomer (RelyX Unicem) was used. The fitting surfaces of the copings were blasted with aluminum oxide ≤40μm then the blasted surfaces were cleaned with alcohol and dried with water and oil-free air. RelyX Unicem capsule was mixed in a high frequency mixing unit (DeGötzen, Italy) for 15 seconds. The cement was applied to the fitting surface of each coping. All the copings were seated onto their corresponding abutments and 5 Kg force was applied for 10 minutes. The excess cement was removed. Light curing was activated on buccal, palatal, mesial, distal and occlusal surfaces for 20 seconds for each surface using Light curing unit (Mini LED, 1250mW/cm^2, Satelec, Acteon).

In subgroup B (RX ARC), RelyX ARC adhesive resin cement was used. The scotchbond etchant (3M Scotchbond, Etchant, 3M ESPE, Germany) was applied to dentin for 15 seconds, then rinsed for 10 seconds. Two consecutive coats of single bond adhesive (Single bond adhesive, 3M ESPE, Germany) were applied to etched dentin for 15 seconds, and air thinning for 5 seconds was done. Light curing for 10 seconds was done per bonding surface. The fitting surfaces of the copings were blasted with aluminum oxide ≤40μm then the blasted surfaces were cleaned with alcohol and dried with water and oil-free air. RelyX Ceramic primer (3M scotchbond ceramic primer, 3M, ESPE, Germany) was applied to the fitting surfaces of each coping and dried for 5 seconds. The cement was mixed onto a mixing pad for 10 seconds. A thin layer of cement was applied to the fitting surface of the copings. The copings were seated onto their abutments and 5 Kg force was applied for 10 minutes, excess cement was removed 3-5 minutes after seating. Light curing was activated on buccal, palatal, mesial, distal and occlusal surfaces for 40 seconds for each surface. All specimens were subjected to 2000 thermal cycles between 5°C and 55°C (Willytecthermocycler, Germany) with a dwell time of 30 seconds in each bath.

Each acrylic embedded tooth with its own cemented coping was secured with tightening screws to the lower fixed compartment of a materials testing machine (Model LRX-plus; Lloyd Instruments Ltd., Fareham, UK) with a loadcell of 5 kN and data were recorded using computer software (Nexygen-MT Lloyd Instruments). The coping was suspended from the upper movable compartment of the testing machine through custom made double loop made of orthodontic wire. This wire was hooked around the cylinderical handles and and the cemented copings were pulled off along the path of insertion. Fig (2)

A tensile load with pull out mode of force was applied via materials testing machine at a crosshead speed of 0.5 mm/min. The force at dislodgment was

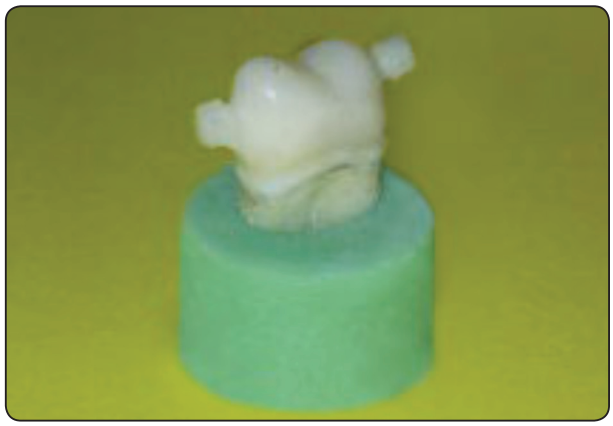

Fig (1) The cemented copings designed with cylindrical handles to facilitate pull-out testing.

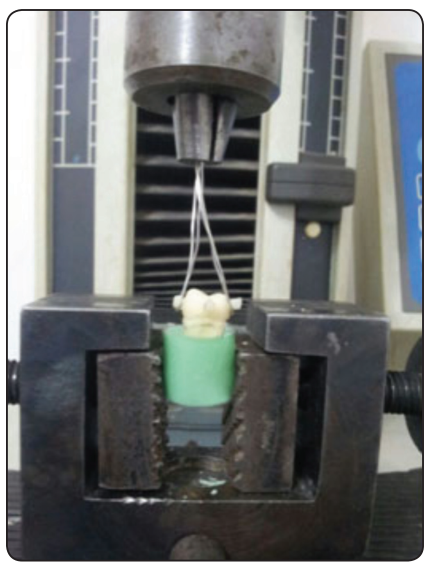

Fig (2) Pull-out testing.

recorded and the stress of removal was calculated using the surface area of each preparation.

Statistical analysis was performed by Microsoft Office 2013 (Excel) and Statistical Package for Social Science (SPSS) version 20.

Data were presented as mean and standard deviation (SD) values. The significant level was set at P ≤ 0.05.

Kolmogorov-Smirnova and Shapiro-Wilk tests was used to assess data normality and data was assumed normally distributed

Two ANOVA was used to assess effect of coping material and cement over retentive stresses.

RESULTS

The mean and standard deviation (S.D.) of the two tested groups are shown in table 2 and figure 3.Group I (LZ) exhibited higher retentive stress value (6.03MPa) than group II (LU) (4.66MPa) and the difference was statistically significant.

Regarding the types of the cement (Table 3, Figure 4), there was no statistical significant difference between retentive stress value of subgroup A-RXU (5.32MPa) and subgroup B-RX ARC (5.73MPa), however, subgroup B-RX ARC had slightly higher values.

There was no significant effect of the interaction between coping material and cement over retentive stress values (Table 4, Figure 5).

TABLE (2) Retentive stress values of the two tested groups (LZ, LU) expressed in MPa.

	Group I (LZ)		Group II (LU)		P value
	Mean	SD	Mean	SD.	
Retentive stress (Mpa)	6.03	0.55187	4.66	0.37	<0.001

TABLE (3) Retentive stress values of the two subgroups (RXU, RX ARC) expressed in MPa

	Subgroup A-RXU		Subgroup B-RX ARC		P value
	Mean	SD	Mean	SD	
Retentive stress value Mpa	5.32	0.78145	5.73	0.93577	0.84

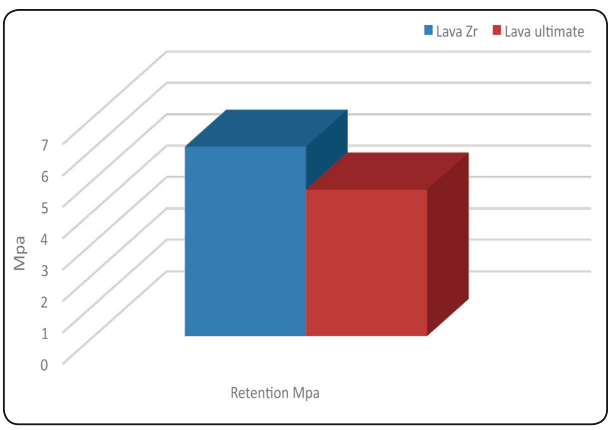

Fig (3) Retentive stress values of the two groups showing the difference between coping materials, expressed in MPa.

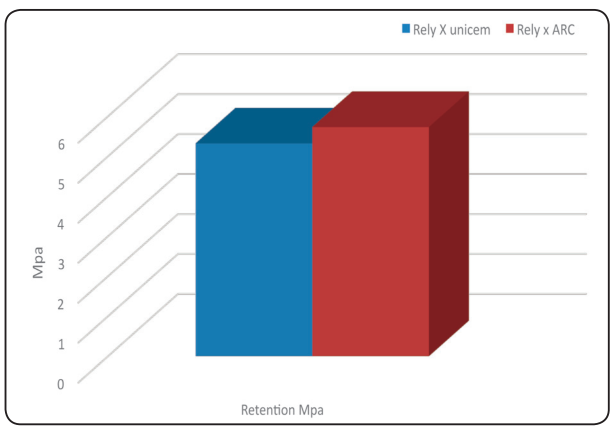

Fig (4) Retentive stress values of the two subgroups showing the difference between cement types, expressed in MPa.

TABLE (4): Effect of interaction between coping material and cement over retention

Coping material	Cement type	Mean	SD	P value
LZ	RXU	5.9400	.57271	0.565
	RX ARC	6.1200	.58052	
LU	RXU	4.7000	.29155	
	R X ARC	4.6200	.47645	

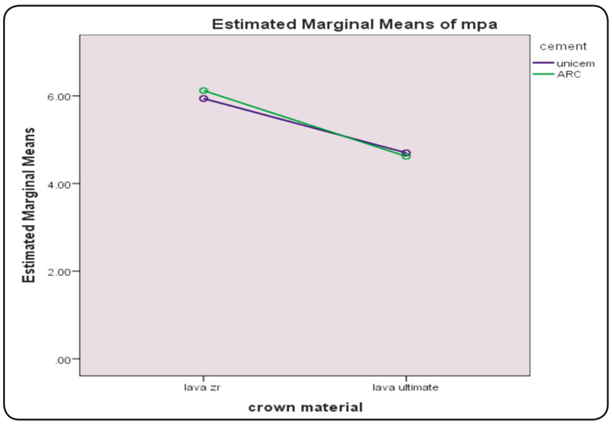

Fig (5) Estimated marginal means expressed in MPa.

DISCUSSION

The hypothesis that the retentive force of Lava Zirconia and Lava Ultimate copings is influenced by the cement type was rejected. The adequate adhesion between ceramic and tooth structure is one of the requirement for successful function of ceramic restorations over years. Bond strengths are influenced by several factors, one of which is the bonding mechanism. Another factor is the restorative material itself. [17] Bonding to zirconia restorations still represent a challenge as neither hydrofluoric acid etching nor silanization result in substantial modification of the surface due to the high crystalline content and the limited vitreous phase of this high-strength core ceramics.[33,40,41] However, sandblasting is supposed to roughen zirconia increasing the bonding area and modifying the ceramic superficial energy and wettability, thus facilitating the formation of resin-ceramic micromechanical interlocks. [42, 43]

It was proven previously that a durable bond to zirconium-based ceramic can be achieved after airborne particle abrasion of the ceramic and the use of resin composite containing an adhesive phosphate monomer. [44, 45]

Satisfactory bonding to zirconium oxide ceramics using multi-step phosphate monomer-based resin cement had been also demonstrated in the two studies conducted by Luthy et al [46] and Yoshida et al. [47]

The new self-adhesive modified composite resin cement was represented and developed with the goal of combining the ease of handling and absence of required pretreatment steps, along with favorable esthetics and firm adhesion to tooth structure. [24] This single-step luting agents contain a resin matrix packed with multifunctional acid methacrylates that should ideally interact with porcelain substrate. [20, 23] A controversy has been occurred claiming that these self-adhesive cements have comparable results to the multi-step total etch cements.

RelyX ARC, a conventional commercial Bis-GMA was used as a control. This cement does not contain methacrylate phosphoric acid ester. However, it was concluded previously by Matinlinna et al [48] that RelyX ARC has a coefficient of thermal expansion that matches more closely with zirconium oxide thus resulting in enhanced bonding. Moreover, artificial aging using thermocycling showed that only RelyX ARC was able to maintain its shear bond strength after using silane coupling agents.

Unfortunately, there are no reported researches in the literature about retentive strength of Lava Ultimate restorations. However, RelyX Unicem luting cement is recommended by the manufacturer.

In the present study, Lava Zirconia and Lava Ultimate copings were constructed using the same CAD/CAM technology, so that standardization of ceramic coping thickness, shape and even the dimensions of the handles used for pull-out testing have been assured. In addition, standardization of scanning process, software designing, milling procedures and shrinkage effect which may affect the final retentive strength of the restoration have been reached.

To better simulate the clinical environment, the retentive strength of luting agents was studied using pull-out test with copings cemented on extracted human teeth. The same was done previously in several studies. [17, 23, 24] However, in the pull-out test, the shape of the abutment preparation should be similar. This was done by choosing the abutment teeth of similar sizes which was measured at specific predetermined points before and after the preparation.

The cementation process was carried out using specific constant load to prevent the variations between different samples during cementation process.

The results of this study revealed that Lava zirconia copings exhibited higher retentive stress value (6.03MPa) than Lava ultimate copings (4.66MPa). This may be attributed to the heterogenous structure of the Lava Ultimate copings (Nanoceramics and resinous matrix) which may lead to variation of the bonding mechanism to each component in the lava ultimate material itself creating several stress concentration areas which accelerates debonding. Moreover the lowered modulus of elasticity of Lava Ultimate may have led to increased flexibility and enhanced debonding more easily than lava zirconia during pull-out testing.

The results of this study showed that the total etch bonding mechanism exhibited a comparable mean retentive stress values as the self-adhesive bonding mechanism regardless of the coping material used. This may be attributed to several factors: total-etch mechanism (RelyX ARC) contributed to complete removal of smear layer within dentin, therefore, total-etch system is applied directly on demineralized dentin collagen preserving the collagen in the dentin matrix and finally improved the stability of the hybrid layer [49]. In addition, the superior performance of the phosphate ester group (MDP functional monomer) of the self-adhesive mechanism (RelyX Unicem) promoted etching of dentin surface so enhanced the bond with zirconium oxide layer. [47]

This was in accordance with Oyague et al, [50] who concluded that bond strength of conventional BIS-GMA to air-abraded zirconia was statistically similar to that of Rely X Unicem. They concluded that the increase in surface roughness after sandblasting could have somewhat compensated the absence of functional monomers in the conventional BIS-GMS cement.

The results of this study provide an indication of the possible performance of two types of resin luting agents to zirconia and resin nanoceramics. Clinical trials are needed to refine these conclusions as all-ceramic restorations are subjected to more complex thermal-biomechanical interactions than those incorporated in in-vitro tests.

Further research is needed to examine the retentive strength of Lava zirconia and lava ultimate restorations using different cement types after long -term storage and cyclic loading.

CONCLUSIONS

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

1. Regarding the coping material, Lava zirconia copings exhibited higher mean retentive stresses and forces than Lava ultimate copings and the difference was statistically significant.

2. There was no statistical significant difference between retentive stress values of RelyX Unicem and RelyX ARC for the two coping materials, however, RelyX ARC showed slightly higher mean values.

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