



## RESEARCH ARTICLE

### MONOLITHIC VERSUS PRESSED/LAYERED CAD/CAM ZIRCONIA CROWNS: EFFECT OF CEMENT MATERIAL ON THE FRACTURE STRENGTH

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#### ABSTRACT

The purpose of this study was to evaluate the influence of different types of cements on fracture resistance of full zirconia and zirconia-based ceramic single crowns.

Maxillary premolar crowns in 2 mm thickness were fabricated on metal dies in 3 groups: Group MO: 24 monolithic zirconia crowns; Group ZL: 24 zirconia frameworks veneered with feldspathic by the layering technique; Group ZP: 24 zirconia frameworks veneered by the heat-pressed technique. Then groups separated in two subgroups and half of them were cemented with glass-ionomer cement and the other half were cemented adhesive resin cement. All specimens were stored in distilled water at 37 °C for 24 hours and then underwent thermal cycling. Single load to fracture test was performed in universal testing machine until failure. The mean fracture values were compared by an one-way ANOVA and a multiple comparison post-hoc Tukey HSD test ( $p < 0.05$ ). Scanning electron microscope was used to evaluate cracks and/or bulk fracture.

**Results:** Group MO cemented with adhesive cement showed the highest mean fracture strength ( $2,703.07 \pm 308,98$  N). There was no significant difference in the mean fracture resistance of Group ZP and ZL.

Monolithic zirconia full crowns had the highest fracture strength values and these single crown restorations did not effected by cementation.

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## INTRODUCTION

The demand for metal-free materials have promoted development of all-ceramic systems. Until the introduction of yttrium partially stabilized zirconia ceramics, several attempts have been made to improve the fracture strength and brittle properties of all-ceramic restorations. Due to its high mechanical properties and advances in CAD/CAM technologies; zirconia is used for the production of single crowns and fixed dental prosthesis (FDP) and known as a tough material with good long-term stability (Fonseca *et al.*, 2013). Traditionally, veneering ceramics are layered on zirconia copings to prevent the tendency for premature failure of the brittle ceramic. Ceramics also can be used onto the zirconia copings to fabricate single crowns or FDPs, using heat-pressing technology, which involves the simultaneous application of heat and pressure to prefabricated ceramic ingots in a previously invested mold cavity (Droge, 1972).

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However, in heat-pressing technique, high density of ceramic ingots; spherical porosities were observed within the veneer layer and also at the interface (Guess *et al.*, 2009). Moreover, these bilayered ceramic systems have several disadvantages including the multistep manufacturing process, low toughness of the veneer material, and weak bonding between veneer ceramic and zirconia coping (Zhang *et al.*, 2013). The most important characteristics for layering or heat-pressed ceramic materials are their fracture strength and fracture toughness. Fracture strength is also known as breaking strength which means the stress of a specimen that fails through fracture (Guess *et al.*, 2010). The strength of veneer ceramic materials is limited by the size of the cracks in microstructure or defects that appear during processing, manufacturing and manipulating (Guess *et al.*, 2010). The initial purpose for zirconia material was decreasing the large number of veneering failures: both adhesive (delamination) and cohesive (chipping) failures but the most common clinical complication reported by systematic reviews is the chipping of bilayered all-ceramic restorations (Guazzato *et al.*, 2004; Raigrodski *et al.*, 2012).

The alternative to preclude all bilayered zirconia system's disadvantages is to replace the veneer/core bilayer with a

monolithic restorative system (Guess *et al.*, 2010). Many manufacturers have shifted their attention to the development of monolithic all-ceramic materials to simply remove the most common failing layer of the system and to avoid inherent residual thermal stresses in bilayered all-ceramic systems. Monolithic zirconia restorations were recently introduced and offer a single CAD/CAM procedure and also exhibit higher fracture strength when compared with bilayered zirconia restorations (Lawn *et al.*, 2001; Raigrodski *et al.*, 2012). The most important advantage of monolithic zirconia restorations is elimination of veneer ceramic chipping but on the other hand the application of these restorations is only reported in single case studies with a short observation period (Marchack *et al.*, 2011). To prevent mechanical fracture of the monolithic zirconia full crown, the thickness of zirconia crown and proper sintering process should be considered. The relevance of cementation is an important point for the fracture load tests. Currently, zirconia restorations for fracture load tests are differently cemented in various studies (Rosentritt *et al.*, 2011; Stawarczyk *et al.*, 2011). Conventional zinc oxide-phosphate, glass ionomer or adhesive resin cements are used (Stawarczyk *et al.*, 2011; Stawarczyk *et al.*, 2012). Zirconia shows high values of fracture strength when luted with adhesive resin cements, however, zirconia material offers the possibility of conventional cementation. Therefore, the purpose of this in vitro study was to evaluate the influence of different cement types on the fracture resistance of bilayered zirconia or monolithic zirconia single crowns. The null hypothesis were different veneer ceramic techniques would not effected the fracture strength of zirconia crowns and resin cement type would not effect on the fracture strength of both bilayered zirconia and monolithic zirconia single crowns.

## METHODS

A prefabricated Frasaco® acrylic tooth (Frasaco® GmbH, Tettnag, Germany) representing maxillary first premolar was used as a master model. The tooth preparation of model was arranged with 1 mm 360 ° chamfer preparation and occlusal reduction of 2.5 mm on buccal tubercule and 2 mm on palatal tubercule. A silicone impression was made to duplicate the artificial tooth into Co-Cr metal alloy and 72 Co-Cr master dies were fabricated using metal casting technique. A Co-Cr master die was scanned with 5-axis 3D scanner device and designed for zirconia copings and monolithic zirconia anatomic crowns. Then 48 zirconia copings and 24 monolithic zirconia crowns were manufactured by Zirkozahn CAD/CAM system (Zirkozahn, Steger, Ahrntal, Italy) using pre-sintered zirconia (ICE Zirconia Translucent blank size 95H10, Zirkozahn, Steger, Ahrntal, Italy) and monolithic zirconia (Zirconia Prettau blank size 95H14, Zirkozahn, Steger, Ahrntal, Italy) blocks.

### **Group ZL: Zirconia based ceramic crowns / Layering technique**

40 µm virtual spacer layer and a wall thickness of 0.5 mm were chosen for zirconia copings. After 4 hours milling procedure, % 20 enlarged 24 zirconia copings were removed from CAM machine and manufacturer's appropriate colour liquid was applied for 3 seconds onto the copings. All the copings were put under the manufacturer's infrared drying lamp and dried for 5 minutes. Final sintering was performed at 1500 °C in a zirconia sintering furnace for 8 hours. The

copings were examined and cleaned with hot steam. All copings were seated on Co-Cr master dies and controlled in terms of marginal adaptation. An experienced dental technician applied the traditional layering technique to veneer the copings of group ZL. First, feldspathic ceramic powder and liquid was mixed according to manufacturer's instructions and applied onto the zirconia frameworks. All specimens fired in a calibrated ceramic furnace under vacuum at a temperature of 830 °C and glazed. A silicone impression was taken from this specimen and used as a control key to maintain the standardization of veneering ceramic size of the other zirconia based ceramic crowns.

### **Group ZP: Zirconia based ceramic crowns / Heat-Pressing technique**

In this group, 24 zirconia copings were fabricated in the same way with the layering technique group. The silicone impressions taken from the outer surface of group ZL were used to give the standardized shape of the crown to these restorations. Buccal and lingual parts of the impressions were filled with casting wax in the area of the crown, and the testing models with the fixed copings were put into one part of the impression. The two parts of the impression were set together and the resulting wax-up was arranged onto the coping in order to obtain an equivalent veneering structure of the corresponding crown from Group ZL. The wax surface was invested into the phosphate-bonded investment material in a muffle according to the manufacturer's instructions and burnt out. The muffle was heated and the zirconia copings were overpressed by a fluor-apatite ceramic in a special ceramic furnace (1075 °C). After cooling at room temperature for 1 hour, the investment was removed in a sandblasting unit using 50 µm aluminum oxide grains at 2 bar pressure. After final firing at a temperature of 790 °C, the zirconia based ceramic crowns were glazed.

### **Group MO: Monolithic zirconia full crowns**

MO crowns were designed using CAD's special software with 40 µm virtual spacer layer and 2 mm occlusal wall thickness. Milling procedure continued for 10 hours and repeated 2 times for fabrication of 48 MO full crowns. After milling of MO blocks in partially sintered stage considering of % 20 shrinkage. MO crowns were stained using special colour liquids and dried for 5 minutes under the infrared drying lamp. Sintering was performed at 1600 °C in sintering furnace for 8 hours. All specimens were fully sintered and reached the original crown size thereafter. Glaze powder and liquid were mixed and applied onto the MO crowns. Glaze sintering was done in the ceramic furnace at a temperature of 840 °C according to manufacturer's instructions.

### **Crown Cementation**

After glazing, sandblasting using 50 µm aluminum oxide grains at 2 bar pressure applied before cementation. Each group were subdivided equally and cemented with Fuji I conventional glass ionomer cement (GC Corporation, Tokyo, Japan) (Groups ZL-C, ZP-C, MO-C) and adhesively luted with RelyX™ Ultimate Clicker™ dual-cure adhesive resin cement (3M ESPE GmbH, Seefeld, Germany) (Groups ZL-A, ZP-A, MO-A). The glass ionomer cement powder and liquid (2/1 ratio) was mixed for 20 seconds and applied inside of the crowns.

**Table 1. Static fracture load of all groups. Medians, means, standard deviations, minimum and maximum in N**

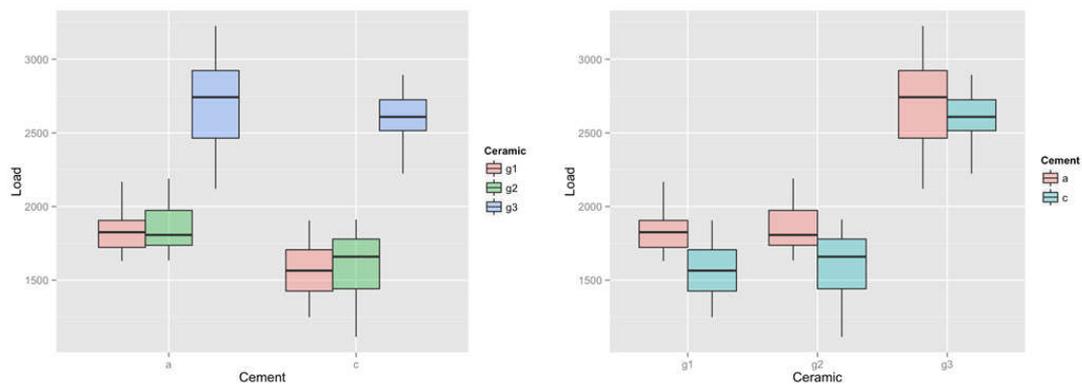
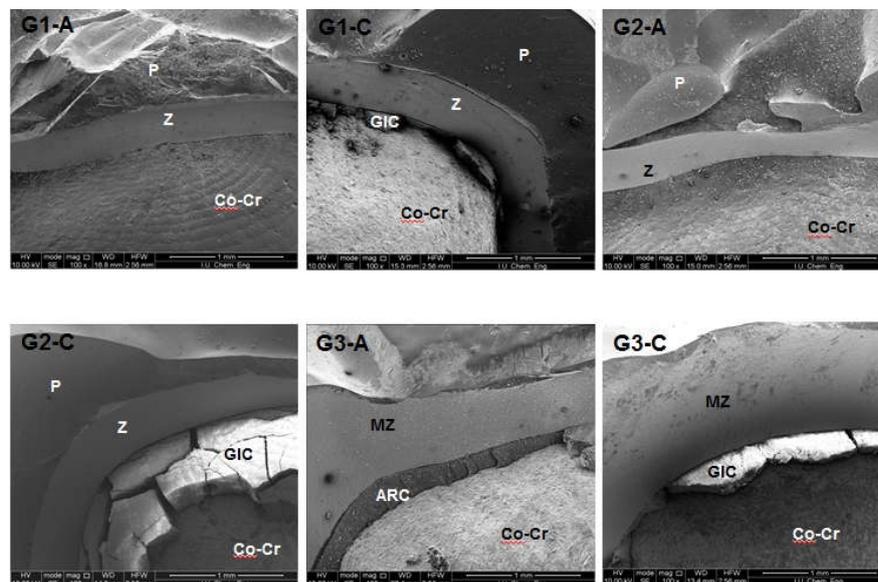
Group	Median	Mean $\pm$ SD	Min (N)	Max (N)
GZT-C G1	1,564.51	1,566.84 $\pm$ 194.81	1,248.15	1,905.10
GZT-A G1	1,825.34	1,845.99 $\pm$ 178.66	1,630.33	2,167.62
GZP-C G2	1,659.11	1,586.40 $\pm$ 273.10	1,114.73	1,912.12
GZP-A G2	1,820.94	1,828.17 $\pm$ 184.44	1,577.85	2,128.99
GMO-C G3	2,608.62	2,593.57 $\pm$ 214.25	2,223.56	2,893.79
GMO-A G3	2,742.43	2,703.07 $\pm$ 308.98	2,436.09	3,225.97

**Table 2. One-way ANOVA of experimental groups**

	d.f.	Sum of Squares	Mean Squares	F	P value
G1	1	467556	467556	13.38	0.00138 **
G2	1	428261	428261	8.05	0.00959 **
G3	1	71940	71940	1.018	0.324
Signif. codes	0 '****'	0.001 '***'	0.01 '**'	0.05 '*'	0.1 ' '

**Table 3. Tukey multiple comparisons of means – 95% family-wise confidence level**

	Ceramic Groups	diff	lwr	upr	P adj	
Adhesive Resin Cemented Group Comparisons	G2A - G1A	7.573008	-223.2033	238.3493	0.9964321	
	G3A - G1A	857.074638	626.2984	1087.8509	0.0000000 ***	
	G3A - G2A	849.501629	618.7254	1080.2779	0.0000000 ***	
Glass Ionomer Cemented Group Comparisons	G2C - G1C	19.56095	-210.6563	249.7782	0.9763384	
	G3C - G1C	1026.72766	796.5104	1256.9449	0.0000000 ***	
	G3C - G2C	1007.16672	776.9495	1237.3839	0.0000000 ***	
Signif. codes		0 '****'	0.001 '***'	0.01 '**'	0.05 '*'	0.1 ' '

**Figure 1. Boxplots of fracture strength of all tested groups for each cementation and ceramic type****Figure 2. SEM views of the fracture surfaces belong to groups, magnification x100. P: Porcelain, Z: Zirconia, Co-Cr: Metal alloy master die, GIC: Glass ionomer cement, ARC: Adhesive resin cement, MZ: Monolithic zirconia**

The crowns were fixed onto the Co-Cr metal alloy master dies. A special cementing device was used to ensure that the crown was loaded centrally at a force of 50 N for 10 min and then the excess cement was removed. In adhesive resin cement groups; the prepared surfaces of Co-Cr metal alloy master dies was roughened with metal alloy primer and dried with air for 3 seconds. MDP including adhesive was applied inside of the crowns and dried 5 seconds until the solvent evaporated. The adhesive was polymerised using a LED light device for 10 seconds according to manufacturer's instructions. Dual-cure adhesive resin cement was mixed for 20 seconds to get a homogenous consistence and put into the crowns and fixed onto the master dies. The same special cementing device was used to remove the excess cement. All crown restorations were polymerised 40 seconds for each with LED light device and waited under pressure for 6 minutes to complete polymerisation totally. All cementations were done by the same team of experienced dentists.

### Fracture Testing

All crown restorations were stored in distilled water at a temperature of 37 °C for 24 hours to assure hydration and eliminate effect of water uptake dimensional expansion after cementation. Prior to mechanical testing, all cemented specimens underwent thermal cycling in thermocycle machine water baths at temperatures between 5 – 55 °C ( $\pm 2$ ) with waiting time 30 seconds in each compartment for 5000 cycles. All Co-Cr master dies were embedded in 10 mm internal diameter stainless steel cylindrical tubes using auto-polymerised acrylic resin. Specimens were subjected to single-load to fracture test and the fracture strength values were calculated in Newton (N) units. For single load-to fracture, the specimens were mounted on the universal testing machine and load-to-fracture was applied axially with a 4 mm diameter stainless steel ball indenter on the occlusal surface of lingual cusp (functional cusp) at a crosshead speed of 0.5 mm/min. The maximum fracture load was measured by applying compressive load to the occlusal surface until the crown failed. Catastrophic fracture failure was considered as either the presence of visible cracks or sudden load drops or even acoustic events of chipping or fracture. The crowns were optically examined after fracture testing, and failure modes were separated as total core fracture (mixed type), chipping of the veneer (cohesive type), or fracture at core/veneer interface (adhesive type). One representative specimen from each group was gold-sputtered and examined using scanning electron microscopy. Data results were analyzed statistically using SPSS 19.0 for Windows (SPSS Inc., Chicago, IL, USA). The loads at fracture were registered, and differences between the groups were calculated using a one-way analysis of variance (ANOVA) test at a significance level of % 5. Additionally a multiple comparison post-hoc test (Tukey-HSD) was performed to evaluate differences between the experimental groups.

### RESULTS

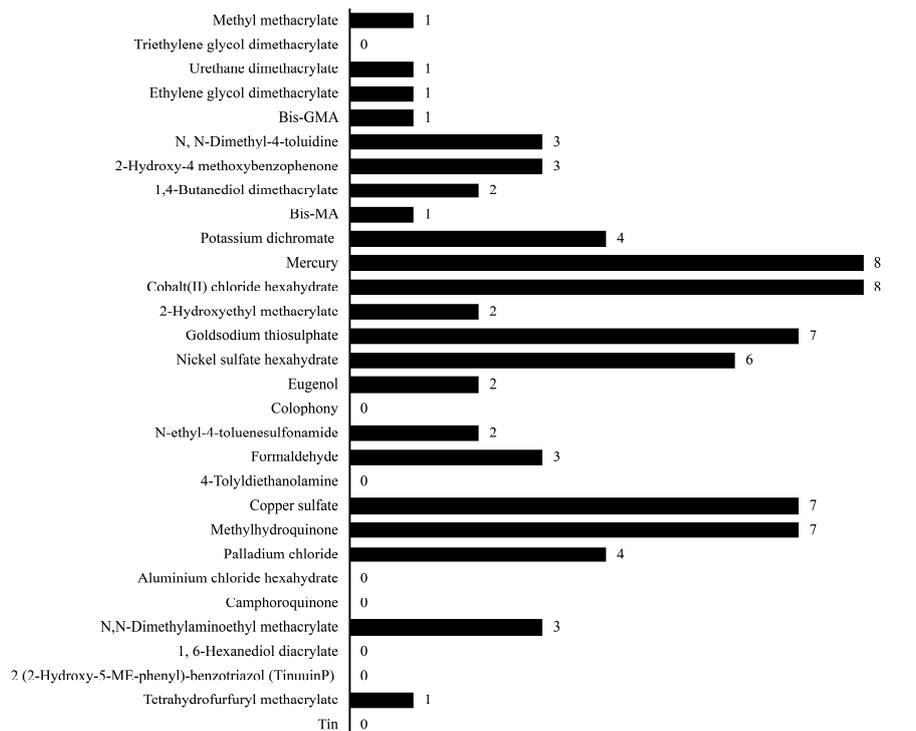
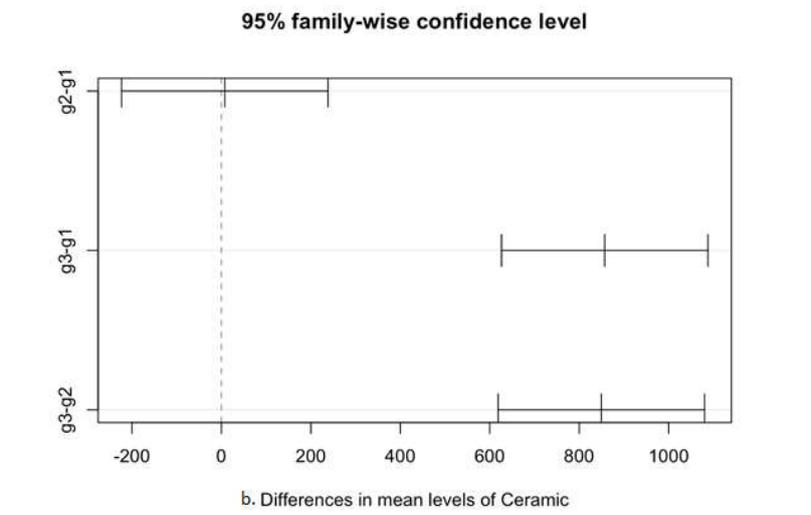
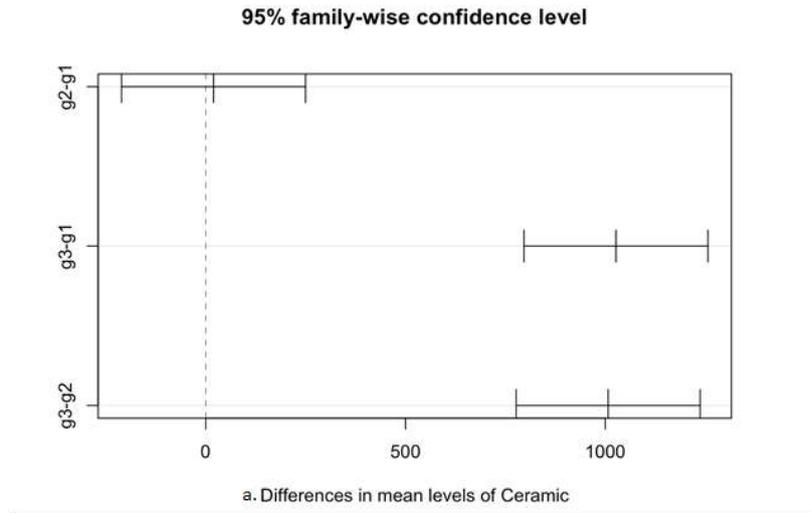
The medians, means, standard deviations, minimum and maximum fracture loads for all groups are listed in Table 1. Figure 1 shows the boxplots for the tested groups, separately for each cementation type and each ceramic material. The SEM views of the interface between crowns and metal alloy master dies were shown in Figure 2. Among the MO groups; [Group MO-C (2,593.57  $\pm$  214.25 N); MO-A (2,703.07  $\pm$

308.98 N)] no statistically significant influence of cementation type on fracture load results was observed ( $p = 0.324$ ). For group ZL, ZL-A (1,845.99  $\pm$  178.66 N) showed significantly higher fracture load compared to the group ZL-C (1,566.84  $\pm$  194.81 N) ( $p = 0.00138$ ). Also in groups ZP; group ZP-A (1,828.17  $\pm$  184.44 N) showed higher fracture load compared to the group ZP-C (1,586.40  $\pm$  273.10 N) and this comparison was found to be statistically significant ( $p = 0.00959$ ). The results of One-way ANOVA statistical analysis for all groups were shown in Table 2. For the analysis of cementation types with the ceramic groups, post-hoc Tukey multi comparison test was performed and the results are shown in Table 3 and the pairwise comparisons were plotted in Figure 3. The comparison of groups ZL-C / ZP-C and groups ZL-A / ZP-A showed similar fracture strength values and these group comparisons revealed no statistically significant difference ( $p > 0.05$ ). Ceramic veneering techniques had no effect on fracture strength of these zirconia based ceramic crowns. MO crown groups MO-C and MO-A showed the highest fracture strength values than other all bilayered zirconia crown groups and the results were found to be statistically significant ( $p < 0.001$ ).

### DISCUSSION

Based on the results of the present study; the first null hypothesis were different veneer ceramic techniques would not effected the fracture strength of zirconia crowns was rejected. Also the collected data support to rejected the second null hypothesis which was resin cement type would not effected on the fracture strength of both bilayered zirconia and monolithic zirconia single crowns. In combination with CAD/CAM fabrication, monolithic/full-anatomic crown restorations seemed to be reliable and robust. Previous studies evaluating the fracture strength of all-ceramic monolithic crowns indicate a superior performance for the monolithic design. According to the authors, the reason of enhanced performance of monolithic crowns is the elimination of the interface between core and veneer, which is believed to be the weak link in bilayer systems with no doubt (Guess *et al.*, 2010; Silva *et al.*, 2011). Another in vitro study evaluated the load-bearing capacity of four different zirconia based crowns, including zirconia core with veneer layer produced either by powder build-up or CAD/CAM technique, glazed monolithic zirconia, and polished monolithic zirconia.

The results showed that zirconia in bilayer configuration had significantly lower load-bearing capacity than the other crowns' design (Beuer *et al.*, 2012). In this present study, the monolithic zirconia anatomic crown groups (MO-C and MO-A) showed the highest fracture strength values in comparison of all groups. In all-ceramic systems, the flaw population (size, number and distribution) can be related to the material, or be affected by the fabrication process. The heat-pressing ceramic technique introduces fewer flaws than layering, and better strength properties are expected, as it is a more controlled procedure. By comparison, the layering technique is subject to variability due to the individual veneering and firing procedures. Nevertheless, no statistically significant differences were found in the fracture loads between the groups ZL and ZP. A study which compared fatigue of layered and heat pressed zirconia crown systems also found no statistical difference between layering and by heat pressing techniques in terms of mechanical stability (Beuer *et al.*, 2009).



**Figure 3. a. 95% family-wise confidence level for glass ionomer cemented groups, b. 95% family-wise confidence level for adhesive resin cemented groups**

Therefore, the present study showed fracture strength of bilayered zirconia crowns did not influenced by using different veneer ceramic techniques. The null hypothesis that different veneer ceramic techniques have an effect on the fracture strength of bilayered zirconia crowns was rejected. Sand-blasting is an alternative widely investigated for improved resin bonding to reinforced ceramics (Qeblawi *et al.*, 2010). Many studies demonstrated that surface grinding significantly increased the fracture strength of zirconia by inducing a tetragonal to monolithic phase transformation that could inhibit microcrack extension, thus increasing the strength of zirconia material (Guazzato *et al.*, 2005; Qeblawi *et al.*, 2010). In this present study, inside surfaces of the zirconia and monolithic zirconia crowns were sand-blasted with 50 µm aluminum oxide particles. Zirconia based ceramic restorations can be cemented with both conventional and adhesive resin cement agents. Adhesive resin cements and primers containing 10-MDP monomer (10-methacryloyloxydecyl dihydrogen phosphate) have been considered for the cementation of zirconia ceramic restorations because the chemical interaction established between the hydroxyl groups of the zirconia ceramic and the phosphate ester monomer of the MDP-containing material (Oyague *et al.*, 2009; Tanaka *et al.*, 2008). Supporting materials, such as abutment material and cement, will influence the fracture resistance of all-ceramic crowns (Mormann *et al.*, 1998; Yucel *et al.*, 2012). Indeed, earlier studies have demonstrated that the fracture resistance of monolithic all-ceramic crowns made of feldspar ceramic, leucite glass-ceramic and lithium disilicate glass-ceramic, which possessed much lower flexural strength than zirconia, increased by using a resin-based cement (Bindl *et al.*, 2006).

The result achieved from our study that adhesive cementation has significantly increased the fracture strength except the monolithic zirconia groups. This result can be explained by the zirconia material thickness, which is more resistant than the veneering ceramic. The results of the present study also are in concordance with other results achieved. Cementation with resin cement does not necessarily result in higher fracture resistance of monolithic zirconia crown. One of the possible reasons would be due to the high strength of zirconia by which monolithic zirconia crowns might withstand the load that can fracture other types of all-ceramic crowns in combination with the effect of mechanical and adhesive properties of cement. In the case of zirconia-based restorations, it is considered that conventional cementation is acceptable, although RC might be a first choice even if adhesion between zirconia and RC can be difficult to achieve (Papia *et al.*, 2014).

Conversely to the results of this study regarding Rosentritt *et al.* did not observe significant differences for fracture resistance between adhesively bonded and conventionally cemented zirconia restorations. The second null hypothesis of this present study that adhesive resin cement would not effected the fracture strength of both bilayered zirconia and monolithic zirconia single crowns was partially rejected. Using adhesive resin cement increased the fracture strength of bilayered zirconia ceramic crowns but had no effect on monolithic zirconia full crowns. The mechanical stability of a prosthetic restoration consisting of the framework with or without veneering ceramic is of clinical importance and can be tested in vitro using fracture load tests (Stawarczyk *et al.*, 2012). Strength values obtained from fracture tests are partially the result of simple geometric shape of specimen (e.g. bar, disk or cube) that do not mimic typical clinical situations. Fracture

test on ceramic specimens which have the shape of anatomic configuration of teeth can be a useful tool for identification of their behavior (Koutayas *et al.*, 2002; Oh *et al.*, 2002). For better standardization, Co-Cr metal alloy tooth analogs were used in this study to support the tested crowns. Being aware of the strong influence of the abutment material and its mechanical properties on the fracture resistance results, natural teeth were not preferred to avoid the natural heterogeneity of biologic samples. In comparison with natural tooth; the abutment material has a significant influence and increased the fracture load in this study (Rosentritt *et al.*, 2000). Problem of this type of mechanical test setup is the failure loads are usually very high (more than 1000 N) compared with the range of mean load values reported in the mouth (about 100 to 600 N) (De Boever *et al.*, 1978). This situation shows that stress state at failure and failure mechanism during in vitro experiments might be different from clinical conditions. The results should be validated by well-designed clinical trials (Kelly, 1999). Several factors, such as preparation design, crown thickness, method of luting, method of cyclic loading and thermal cycling can influence the results (Friedlander *et al.*, 1990). Therefore, the results of different studies cannot be compared directly.

## Conclusion

According to the results of this study; heat-pressing ceramic or layering feldspathic ceramic techniques to veneer zirconia frameworks did not show significant differences in the fracture strength of zirconia-based single crowns, although using adhesive resin cement increased the fracture strength of zirconia-based single crowns. The overall strength of a dental prosthesis is determined by a combination of multiple factors, such as; bond strength, fracture strength, fracture toughness, framework design, stresses created in fabrication, and flaws. Prior to a general recommendation, the data results of this study have to be supported by comprehensive clinical studies.

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