Original Article

Comparative Evaluation of Shear Bond Strength of Newer Resin Cement (RelyX Ultimate and RelyX U200) to Lithium Disilicate and Zirconia Ceramics as Influenced by Thermocycling

Rinki Kansal, Sapna Rani, Manoj Kumar, Shalabh Kumar, Gaurav Issar

Department of Prosthodontics, ITS-CDSR, Ghaziabad, Uttar Pradesh, India

Abstract

Background/Purpose: The purpose of this study was to compare the shear bond strength of high strength ceramics to cut dentine as influenced by different resin cement types after thermocycling. **Materials and Methods:** Shear bond strength testing was carried out for 56 sound, freshly extracted first permanent molars. Specimens were divided at random into 2 groups (n = 28) Lithium disilicate and Zirconia. Lithium disilicate and Zirconia specimens were further subdivided depending upon luting with Rely X ultimate cement bonded with single bond universal adhesive and Rely X U200 cement. Half of the specimens of each material luted with cements were subjected to thermocycling. Shear bond strength was evaluated using Universal testing machine at a crosshead speed of 5 mm/min. Results were compared and evaluated using t-test at a significance level of 0.05. The nature of bond failure was observed under a stereomicroscope for each sample. **Results:** The mean difference of Lithium disilicate test specimens bonded with Rely X ultimate cement bonded with single bond universal adhesive with and without thermocycling was found to be 42.95+/-17.41MPa and 120.62+/-56.46 MPa respectively. The mean difference of Zirconia test specimens with Rely X ultimate cement bonded with single bond universal adhesive, with and without thermocycling, was found to be 8.74+/-2.90 MPa and 164.28+/-43.78 MPa respectively. The mean difference of Lithium disilicate test specimens bonded with Rely X U200 with and without thermocycling was found to be 2.36+/-0.63 MPa and 36.79.62+/-17.21MPa respectively. The mean difference of Zirconia test specimens bonded with Rely X U200 with and without thermocycling was found to be 5.96+/-3.11MPa and 122.46+/-23.01MPa respectively. **Conclusion:** Zirconia was found to have better shear bond strength than Lithium discilicate. The use of single bond universal adhesive improves bonding to newer higher strength ceramics such as Zirconia. Cohesive failure was predominant at cement dentine interface.

Keywords: *Lithium disilicate, resin cements, shear bond strength, zirconia*

Introduction

In search for the ultimate esthetic restorative material, many new all-ceramic systems have been introduced in dentistry. Ceramics offer the potential for excellent esthetics, biocompatibility, and long-term stability.[1]

Despite their good mechanical properties, the porcelain fused to metal restorations did not always provide optimal esthetic values due to metal substructure on the marginal gingival border.[2]

Lithium disilicate is among the best known and most widely used types of glass ceramics as it is a highly esthetic, high strength material that can be conventionally cemented or adhesively bonded. It offers a unique solution with its ability to cater a full contour restoration fabricated from one high strength ceramics to be used in all areas of the mouth, thereby eliminating the challenge of managing two dissimilar materials. It can be processed either using lost wax heat-pressing technique or state-of-the-art computer-aided design/computer-aided manufacturing (CAD/CAM) milling procedure.

Zirconia ceramics are the most recently introduced dental materials. They exhibit high strength, good cosmetics (layered), excellent mechanical properties, and good biocompatibility. As compared to alumina, Zirconia has increased strength, decreased elastic modulus, and the remarkable

Address for correspondence:
Dr. Sapna Rani,
House No 2062, Sector 7D, Faridabad - 121 006, Haryana, India.
E-mail: drsapnadaksh@gmail.com

Access this article online
Website: www.contempclindent.org
DOI: 10.4103/ccd.ccd_601_18
Quick Response Code:

How to cite this article: Kansal R, Rani S, Kumar M, Kumar S, Issar G. Comparative evaluation of shear bond strength of newer resin cement (RelyX ultimate and RelyX U200) to lithium disilicate and zirconia ceramics as influenced by thermocycling. Contemp Clin Dent 2018;9:601-6.

property of transformation toughening.[3-5] Transformation toughening restricts the crack propagation by transformation of tetragonal particles to monoclinic form at the crack tip.[1] Zirconium oxide is primarily used as core ceramics and is fabricated either by CAD/CAM or copy milling technique. CAD/CAM zirconia dental frameworks can be fabricated following two different techniques: "soft machining" of unsintered blanks and "hard machining" of fully sintered blanks.[6]

A high-quality adhesion of the resin cement to the tooth structure and restorating surface is primordial for the success of bonding. Resin cement as compared to the traditional luting materials, have improved retention, reduced dissolution in the oral environment, less microleakage, high strength under tension, ease of manipulation, biological compatibility, excellent esthetic, shade matching potential and acceptable clinical performance.[7] Retention mechanisms are reported to be chemical, mechanical and micromechanical or a combination of these. Resin cement may be classified as total etch, self-etch, and self-adhesive, depending upon their application to the dental tissues.[8] Bonding to zirconia and lithium disilicate to prepared tooth may be advantageous in various ways, especially masking the discolored teeth.

The purpose of this study was to evaluate the shear bond strength of newer resin cement to current high-strength ceramics as influenced by thermocycling. The research null hypothesis was that there was no difference in the shear bond strength between the two resin cement (RelyX Ultimate and RelyX U200) when bonded with lithium disilicate and zirconia after subjecting to thermocycling.

Materials and Methods

The present study was a cross-sectional *in vitro* study to compare the shear bond strength of lithium disilicate and zirconia test specimens adhesively bonded to two different resin cement and effect the thermocycling on shear bond strength of two types of test specimens.

Preparation of tooth structure

For the present study, 56 unrestored caries-free sound permanent molars with no signs of attrition were selected within one month of extraction. Teeth were cleaned of debris and stored in normal saline at room temperature. Any surface of the tooth structure was prepared with the help of air rotor and crown preparation burs (Crown preparation kit, Shofu, Germany) to achieve uniform dentinal surface. The prepared teeth were embedded in autopolymerizing resin (DPI. Mumbai. India) such that the exposed tooth surface was available for cementation of test specimens.

Preparation of test specimens

In the present study, two types of test specimens in the form of discs (3 mm × 3 mm) were fabricated using lithium disilicate and zirconia material. The sample size was kept to be 28 (*n* = 28) in each group as determined by statistician.

Lithium disilicate discs (*n* = 28) were fabricated using heat-pressing technique. Putty index of one wax pattern measuring 3 mm × 3 mm was made to standardize the procedure. Molten inlay wax was poured in putty index and sprue was attached to wax pattern. Investment of wax pattern was done in silicone ring with the phosphate-bonded investment material (IPS PressVEST Speed). The IPS e.max Press ingot was inserted in hot investment ring. Investment ring was inserted in the center of hot press furnace (Multimat 2 touch + press) using investment tongs; selected program was started. After cooling of the ring, the sprue and reaction layer on the test specimens were removed. Dimensions of lithium disilicate discs were verified with a digital caliper.

Zirconia discs (*n* = 28) were fabricated using CAD/CAM technique by dry milling followed by sintering. The disc dimension data were transferred to the software with the help of stereolithographic file. The discs (*n* = 28) were fabricated of the same dimension as lithium disilicate discs.

The sample discs underwent surface treatment with airborne particle abrasion with 50 μm aluminum oxide for 15 s using 4–5 bar pressure followed by cleaning in an ultrasonic bath containing isopropyl alcohol for 3 min. The prepared samples were then divided as described in Table 1.

Half discs of lithium disilicate were bonded with RelyX Ultimate resin cement combined with single bond universal adhesive IA (*n* = 14). Another half discs were bonded with RelyX U200 resin cement IB (*n* = 14). Similarly, zirconia discs were divided into two halves and bonded with aforementioned resin cement (IIA and IIB) (*n* = 14).

For cementation with RelyX Ultimate resin cement with single bond universal adhesive, adhesive was applied to

Table 1: Distribution of test specimens

Lithium disilicate (*n*=28) [I]				Zirconia (*n*=28) [II]			
RelyX ultimate bonded with single bond universal adhesive (*n*=14) [I_A]		RelyX U200 (*n*=14) [I_B]		RelyX ultimate bonded with single bond universal adhesive (*n*=14)[II_A]		RelyX U200 (*n*=14) [II_B]	
With thermocycling (*n*=7) [I_{A1}]	Without thermocycling (*n*=7) [I_{A2}]	With thermocycling (*n*=7) [I_{B1}]	Without thermocycling (*n*=7) [I_{B2}]	With thermocycling (*n*=7) [II_{A1}]	Without thermocycling (*n*=7) [II_{A2}]	With thermocycling (*n*=7) [II_{B1}]	Without thermocycling (*n*=7) [II_{B2}]

Figure 1: Lithium disilicate/zirconia discs cemented

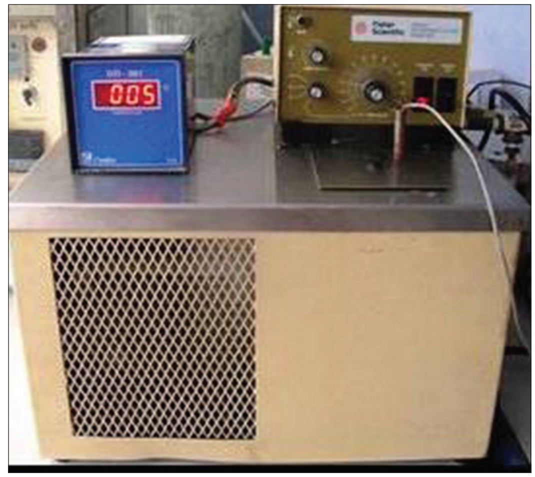


Figure 2: Thermocycler

Figure 3: Sample loaded in the universal testing machine

Figure 4: Testing nature of bond failure under stereomicroscope

prepared tooth surface and resin cement was applied to disc. Disc was pressed onto the tooth surface with finger pressure. The excess cement was removed after initial light cure, and then the cement was completely cured according to manufacturer's instructions [Figure 1]. RelyX U200 cement was also cemented according to manufacturer's instructions.

After cementation, half of the samples of each group were subjected to thermocycling in two different thermal [Figure 2] baths with temperature maintained at 5ºC and 55ºC using distilled water. A temperature regulating button and thermometer was used to monitor temperature fluctuation. Each sample was exposed to thermocycling for a period of 15 s at 5ºC and 55ºC with 15 s interval between each cycle. A total of 5000 temperature cycles were carried out for each sample.

Testing of samples for shear bond strength

Samples were tested for shear bond strength in Universal testing machine with a blunt end chisel at a crosshead speed of 0.5 mm/min until debonding of the discs from the tooth occurred [Figure 3]. The maximum force at which debonding occurred was recorded. The shear bond strength (tr values (expressed in effects of mycophenolic acid [MPa]) were calculated using the formula: $\partial = L/A$ where L is load (in N) and A is the adhesive area (in m^2).

Testing of type of failure

After the shear bond strength testing procedure, all the samples were observed under ×50 magnification using a stereomicroscope to identify the nature of bond failure, namely, cohesive, adhesive, or a combination of both [Figure 4].

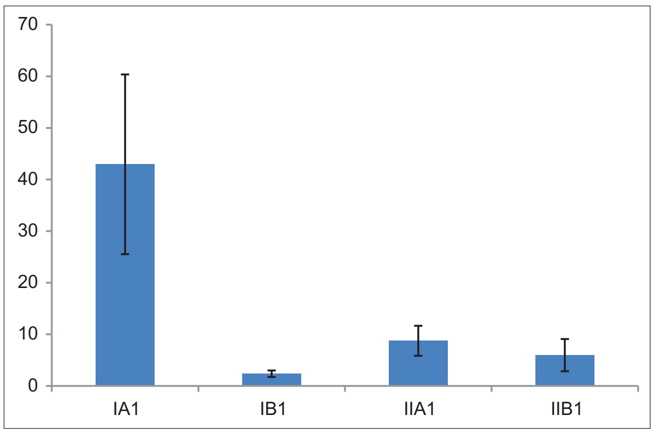
Statistical analysis

The shear bond strength was evaluated by pull-off test for all test specimens. All calculations were performed using the SPSS (version 16) for Windows (SPSS Inc., Chicago, IL, USA). The data thus obtained were entered into MS Excel spreadsheet and the statistical analysis applied was Student *t*-test (to compare between the two

Table 2: Statistical analysis results

	Mean±SD	95% CI for mean		Mean difference	*P*
		Lower bound	Upper bound		
SBS					
IA1	42.954±17.414	26.849	59.060	77.6671	0.005**
IA2	120.621±56.467	68.398	172.845		
IB1	2.366±0.632	1.781	2.950	34.4257	0.000**
IB2	36.791±17.210	20.874	52.708		
IIA1	8.747±2.901	6.064	11.431	155.5386	0.000**
IIA2	164.286±43.78	123.795	204.777		
IIB1	5.962±3.1150	3.081	8.843	116.5040	0.000**
IIB2	122.466±23.014	101.181	143.751		
Mean SBS	**Mean difference**	***P***	**Mean SBS**	**Mean difference**	***P***
IA1 and IIA1	34.2071	0.000**	IA1 and IB1	40.5886	0.000**
IB1 and IIB1	3.596	0.011*	IA2 and IB2	83.8300	0.003**
IA2 and IIA2	43.6643	0.132^	IIA1 and IIB1	2.7854	0.109^
IB2 and IIB2	85.6743	0.000**	IIA2 and IIB2	41.8200	0.045*

^Not significant $P>0.05$, *Significant $P<0.05$, **Highly significant $P<0.01$. SD: Standard deviation; CI: Confidence interval; SBS: Shear bond strength

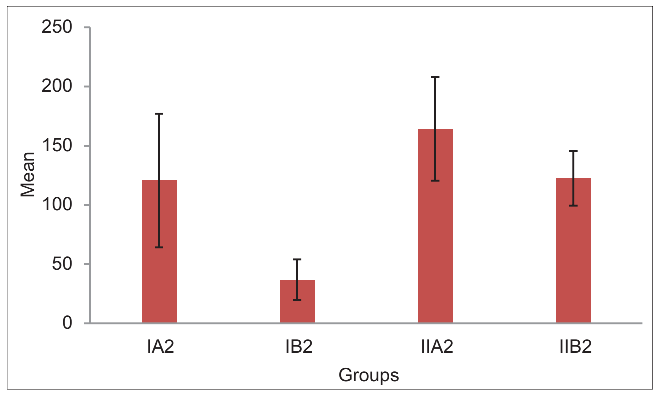


Graph 1: Comparison of mean shear bond strength of lithium disilicate and zirconia specimens bonded with both types of cement without thermocycling

materials). Student *t*-test was applied to analyze the data at a significance level of 0.05.

Results

The mean difference of lithium disilicate discs bonded using RelyX ultimate with single bond universal adhesive with and without thermocycling was 42.954 ± 17.4140 MPa and 120.621 ± 56.4670 MPa, respectively. The mean difference of shear bond strength of zirconia discs bonded with and without thermocycling was 8.747 ± 2.9017 MPa and 164.286 ± 43.7815 MPa, respectively. The mean difference of lithium disilicate discs bonded using RelyX U200 with and without thermocycling was 2.3666 ± 0.6320 MPa and 36.791 ± 17.2104 MPa, respectively. The mean difference of shear bond strength of zirconia discs bonded using RelyX U200 with and without thermocycling was 5.962 ± 3.1150 MPa and 122.466 ± 23.0147 MPa, respectively [Graph 1 and Table 2].



Graph 2: Comparison of mean shear bond strength of lithium disilicate and zirconia specimens bonded with both types of cement with thermocycling

On intragroup comparison of the effect of thermocycling on two test specimens (lithium disilicate and zirconia discs), it was found that there was a significant difference of shear bond strength of lithium disilicate as well as zirconia discs using RelyX ultimate with single bond universal adhesive and RelyX U200. It shows that thermocycling affects shear bond strength of both discs bonded with both types of cement.

On intergroup comparison of lithium disilicate and zirconia discs bonded using RelyX ultimate with single bond universal adhesive with thermocycling, it was observed that there was significant difference between two high-strength ceramics. There was no significant difference between both high-strength ceramics when bonded with RelyX ultimate with single bond universal adhesive without thermocycling. On comparison of two high-strength ceramics using RelyX U200 with or without thermocycling, there was a significant difference with high shear bond strength for lithium disilicate discs.

RelyX ultimate with single bond universal adhesive had significantly high bond strength than RelyX U200 when bonded with lithium disilicate with and without thermocycling. Highly insignificant difference was found in shear bond strength of RelyX ultimate with single bond universal adhesive and RelyX U200 when bonded with zirconia after thermocycling while without thermocycling results were highly significant.

On examination of the type of bond failure for each sample, it was found that cohesive failure was predominant in all groups having high percentage of zirconia discs bonded with RelyX ultimate with single bond universal adhesive without thermocycling.

Discussion

In recent years, esthetic demands have caused dental professionals to opt for the use of metal-free ceramics in prosthodontics. The development of leucite, lithium disilicate, zircônia, and alumina-reinforced ceramics has allowed the substitution of metallic infrastructures in diverse clinical situations, due to their high flexural and compressive strength. The present study was undertaken to compare the shear bond strength of two different high-strength ceramics (lithium disilicate and zirconia) bonded to dentin using resin cement RelyX ultimate with single bond universal adhesive and RelyX U200 (Self-etch–self-adhesive); with and without thermocycling. The results of this study led to the rejection of null hypothesis that there was no significant difference in the shear bond strength of the tested groups.

Fifty-six sound freshly extracted human first molars were collected for this study and stored in distilled water at room temperature. Sample preparation was done within a week of extraction to prevent changes in surface chemistry and physical properties over time.[9] All the zirconia discs were then bonded, according to manufacturer's instructions, to the prepared buccal surfaces of mounted samples as the buccal surface has a more favorable structure due to a lower number and area percentage of tubule openings than that of occlusally positioned dentin which shows a greater regional variability in dentin wetness. Zirconia cores were bonded using digital pressure, as described by Pashley *et al.* and Leloup *et al*.[9,10] and Pekkan and Hekimoglu and Strub and Beschnidt[11,12] who suggested that the polymerization of dual-polymerizing resin cement is not complete until after 1 week of placement. Hence, bond strength evaluations were performed 1 week after specimen preparation, assuming the polymerization of the resin cement to be complete, and the maximum bond strength would have been achieved.

The results in this study showed that the mean bond strength of RelyX ultimate with single bond universal adhesive bonded to lithium disilicate with and without thermocycling was, respectively, 42.954 MPa and 120.621 MPa and with zirconia was 8.747 MPa and 164.286 MPa. The mean bond strength of RelyX U200 bonded to lithium disilicate with and without thermocycling was, respectively, 2.366 MPa and 36.791 MPa and with zirconia was 5.962 MPa and 122.466 MPa. While comparing two resin cement (RelyX ultimate with single bond universal adhesive and RelyX U200), it was observed that a statistically significant ($P > 0.05$) difference in mean shear bond strength was observed. RelyX ultimate with single bond universal adhesive presented highly significant results than RelyX U200. The variation in the bond strength observed could be due to variation in the chemical composition of the two types of cement used. RelyX U200 is composed of methacrylate monomers [Graph 2] containing phosphoric acid groups which are all frequently used cross-linkers in adhesive systems. RelyX U200 contains multifunctional phosphoric acid methacrylates that are claimed to react with the hydroxyapatite of the hard tooth tissue when these monomers dissociate into methacrylate and the acidic phosphoric acid in an aqueous solution. It seems that the solvent was unable to generate enough interfibrillar spaces to accommodate the infiltrating adhesive. These findings are coincident with the studies of Elsayed *et al*.[13] who concluded that the tensile bond strength to zirconia ceramic and lithium disilicate ceramic is significantly influenced by the primer/adhesive used. The effect of silane incorporated in a universal multimode adhesive might be limited. In general, so-called universal primers/adhesives achieve more durable bonding to zirconia than to lithium disilicate.

Thermal cycling was done for half of the samples of each group to evaluate the effect of changing intraoral conditions in mouth on the shear bond strength of ceramics and dentin. In this study, the samples were subjected to 5000 cycles with bath temperatures of 5°C and 55°C with a dwell time of 15 s according to ISO standardization. The results showed that the difference between the mean shear bond strength of RelyX ultimate with single bond universal adhesive with lithium disilicate and RelyX U200, with and without thermocycling was 40.5886 MPa and 83.830 MPa. On the other side, the difference between the mean shear bond strength of RelyX U200 and RelyX ultimate with single bond universal adhesive with zirconia, with and without thermocycling was, respectively, 2.7854 MPa and 41.8200 MPa which was statistically significant difference ($P > 0.05$) in bond strength after thermal cycling in both the groups. According to Blatz *et al.*,[14] the difference in the coefficient of thermal expansion between tooth structure and restorative materials might induce degradation of dentin/restoration surface.

Bond quality, however, should not be assessed on bond strength data alone, because the mode of failure is also important; this information may yield predictions of clinical performance. Following the shear bond testing procedure, all the samples were observed under a stereomicroscope at ×50 magnification to identify the nature of bond failure, namely, cohesive, adhesive, or a combination of both.

Results obtained showed that percentage of failure of RelyX U200 with lithium disilicate before thermocycling were 17% adhesive, 53% cohesive, 28% mixed and after thermocycling were 14%, 70%, 16%, respectively, and with zirconia were 30%, 54%, 14% before thermocycling and 15%, 45%, and 40% after thermocycling, respectively. On the other side, type of failure percentage of lithium disilicate luted with RelyX ultimate and universal adhesive before thermocycling were 12% adhesive, 70% cohesive, 18% mixed and after thermocycling 10%, 68%, 22%, respectively, and with zirconia type of failure percentage were 18% adhesive, 42% cohesive, 40% mixed before thermocycling and 20%, 70%, 30% after thermocycling, respectively. Failure analysis revealed that failures were predominantly cohesive nature in the resin cement.

Monticelli *et al*. and Eick *et al*.[15,16] in their studies tested on dentin bonding observed that resin tags generally break off at the dentin surface rather than pulling out of the dentinal tubules suggesting that the bonding forces holding the resin tags to the tubule walls exceed the cohesive strength of the resin tags. It can, thus, be stated that higher bond-strength values of the resin luting agent to both dentin and ceramic materials increases the cohesive failure rate within the adhesive cement. This finding is also in agreement with those of Altintas *et al*.[17] who observed similar results.

This *in vitro* study also enabled us on an assessment of the bond created by resin bonding agent between dentin and the restorative material. However, *in vitro* tests cannot adequately simulate clinical conditions in every detail. Subjecting the specimens to dynamic loading in artificial saliva before testing may closely resemble intraoral conditions with respect to hydrolytic degradation of the bond due to pH changes of saliva and the effect of temperature change in the mouth. Furthermore, other clinically relevant factors such as configuration of cavity or crown preparation, dentin wetness, pulpal pressure, remaining dentin thickness, and type of dentin (normal or sclerotic) should be considered when testing adhesive systems *in vitro*. The final evaluation of material performance should be determined using long-term clinical studies which take the maximum number of parameters into account, least to mention, individual clinical determinants.

Conclusion

Within the conditions and limitations of this *in vitro* study, RelyX ultimate with single bond universal adhesive was found to be superior as compared to self-adhesive resin cement RelyX U200. Thermocycling affected the mean shear bond strength of both the high strength ceramics bonded with both types of resin cement. Zirconia was found to have higher mean values for shear bond strength as compared to lithium disilicate, with and without thermocycling. Mode of failure was seen to be mostly cohesive in the resin–dentin interface in both the groups.

Financial support and sponsorship

Nil.

Conflicts of interest

There are no conflicts of interest.

References

1. McLaren EA, Giordano RA. Zirconia-based ceramics: Material properties, esthetics, and layering techniques of a new veneering porcelain, VM9. Quintessence Dent Technol 2005;28:99-111.
2. Blatz MB. Long-term clinical success of all-ceramic posterior restorations. Quintessence Int 2002;33:415-26.
3. American Society for Testing and Materials. Engineered Materials Handbook: Ceramics and Glasses. Vol. 4. Philadelphia, PA: American Society for Testing and Materials; 1991.
4. Stevens R. Engineering Properties of Zirconia. Engineered Materials Handbook: Ceramics and Glasses. Vol. 4. Philadelphia, PA: American Society for Testing and Materials; 1991. p. 775-85.
5. Kosmac T, Oblak C, Jevnikar P, Funduk N, Marion L. The effect of surface grinding and sandblasting on flexural strength and reliability of Y-TZP zirconia ceramic. Dent Mater 1999;15:426-33.
6. Palacious RP, Johnson GH, Phillips KM, Raigrodski AJ. Retention of zirconium oxide ceramic crowns with three types of cements. J Prosthet Dent 2006;96:104-14.
7. Malament KA, Socransky SS. Survival of dicor glass cceramic dental restorations over 16 years. Part III: Effect of luting agent and tooth or tooth-substitute core structure. J Prosthet Dent 2001;56:511-9.
8. Diaz-Arnold AM, Vagas MA, Haselton DR. Current status of luting agents for fixed prosthodontics. J Prosthet Dent 1999;8:135-41.
9. Leloup G, D'Hoore W, Bouter D, Degrange M, Vreven J. Meta-analytical review of factors involved in dentin adherence. J Dent Res 2001;80:1605-14.
10. Pashley DH, Sano H, Ciucchi B, Yoshiyama M, Carvalho RM. Adhesion testing of dentin bonding agents: A review. Dent Mater 1995;11:117-25.
11. Pekkan G, Hekimoglu C. Evaluation of shear and tensile bond strength between dentin and ceramics using dual-polymerizing resin cements. J Prosthet Dent 2009;102:242-52.
12. Strub JR, Beschnidt SM. Fracture strength of 5 different all-ceramic crown systems. Int J Prosthodont 1998;11:602-9.
13. Elsayed A, Younes F, Lehmann F, Kern M. Tensile bond strength of so-called universal primers and universal multimode adhesives to zirconia and lithium disilicate ceramics. J Adhes Dent 2017;19:221-8.
14. Blatz MB, Sadan A, Kern M. Resin-ceramic bonding: A review of the literature. J Prosthet Dent 2003;89:268-74.
15. Monticelli F, Osorio R, Mazzitelli C, Ferrari M, Toledano M. Limited decalcification/diffusion of self-adhesive cements into dentin. J Dent Res 2008;87:974-9.
16. Eick JD, Gwinnett AJ, Pashley DH, Robinson SJ. Current concepts on adhesion to dentin. Crit Rev Oral Biol Med 1997;8:306-35.
17. Altintas S, Eldeniz AU, Usumez A. Shear bond strength of four resin cements used to lute ceramic core material to human dentin. J Prosthodont 2008;17:634-40.