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Effect of different surface treatments on shear bond strength of zirconia bonded to dentin with two resin luting cements

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Abstract

All-ceramic materials, especially silica-based lithium di-silicate and non-silicate-based Zirconia have become a topic of interest in the field of dentistry. It is still difficult to achieve a strong and durable resin-ceramic adhesion, especially resin-Zirconia bonding, focusing on the latest resin bonding techniques including surface treatment, priming and cementation.

The purposes of this study was to evaluate the effect of different surface treatments on shear bond strength of zirconia bonded to dentin with two resin luting cements.A total 60 zirconia discs 3mm thickness and 6 mm diameter subjected to three types of surface treatments,irradiated with CO2 laser,etched with hydrofluoric acid, sandblasted+ceramic primer application,then bonded to dentin with two types of resin cement (variolinkII and Dentocem). Shear bond strength was measured and the type of failures were determined. The data were statistically analyzed by one-way analysis of variance and Independent T-test. The results of this study showed that the groups which were sandblasted treated with ceramic primer produced highest shear bond strength followed by hydrofluoric etched groups and CO2 laser irradiated groups showed the lowest bond strength. Dentocem resin cement showed better bonding than variolink II.The failures were either adhesive (between discs and cement)or cohesive(cements on both disc and dentin).As a conclusion the bonding of Zirconia can be improved by the different surface treatments especially by sandblasting ceramic primer . Dentocem resin cement showed higher shear bond strength than variolinkII so promoted better adhesion between dentin and zirconia.

Key words:Zirconia,surface treatments,luting resin,bond strength.

Introduction

There is an increasing demand for metal free restorations due to the increasing interest in esthetics. Both patients and clinicians have been seeking Suitable metal free tooth colored restorations. Recently, all-ceramic restorations provide the most esthetically pleasing restorations currently available. The increasing need for the all-ceramic restorations led to the development of new high-strength ceramic materials with improved mechanical properties, such as aluminum oxide-based and zirconium-oxide based ceramics(1).

Zirconium oxide known as zirconia is a white crystalline oxide of zirconium. Zirconia is a very high performance material with low thermal conductivity and with excellent biocompatibility and mechanical properties (2).

The ultimate adhesion of an indirect restoration to prepared tooth is affected by two important interfaces namely, the interface between the tooth and the adhesive resin and the interface between the adhesive resin and the inner surface of restoration, however new generations of dentin bonding agents do micromechanical interlocking and chemical adhesion to both dentin and the zirconia surface that was treated with different surface treatments.(3'4)The aim of the present study is to evaluate the effect of different surface treatments and two luting resins on bond strength of zirconia to human dentin and the type of debonding failure.

Materials and Methods

60 Zirconium-based discs (Prettau,zircon zhan, Italy) (6mm in diameter, 3 mm thickness) were provided fully sintered by the manufacturer. Disc specimens were cleaned for five minutes in an ultrasonic bath containing distilled water (Quantrex , Kearny, NJ, USA), air-dried, then divided randomly into two main groups according to type of resin cement each contain (30 discs) then each main group subdivided into three subgroups according to the type of surface treatments each contain (10discs).

Types of surface treatment:

A- The discs in this group were subjected to CO_2 laser irradiation with a Power of 5, 10, 15 watt respectively for 10 seconds. Then a modification of the power and time was attempt to be 20 watt for (20) seconds and then to be 20 watt for 30 seconds. The laser treatment was achieved by using CO_2 laser machine (CO_2medical laser system model ATL-250, Optoelectronic Technology, China). A beam of 10.6 um wavelength was delivered in a continuous mode through an articulating arm. The entire ceramic surface was manually scanned in a circular motion to include the whole surface, since the CO_2 laser beam diameter is 0.4mm. The laser parameters used were (20 watt Power, 200 J Energy and 10 seconds application time repeated three times according to M.A. Rashad etal .(1)

B- The discs in this group etched with 9.5% hydrofluoric (HF) acid gel (Versa-link, Hannover-Germany) for (2) min. The etching procedures were performed in a laboratory under ventilation, with operators wearing protective glasses and acid-resistant gloves. The etching gel was rinsed in a polyethylene cup; the diluted solution was neutralized using a neutralizing powder made up of calcium carbonate ($CaCO_3$) and sodium carbonate (Na_2CO_3) for (5) min and thoroughly washed for (30) seconds. The etched substrates were thoroughly washed with water and rinsed to remove any acid residual. The treated samples were then cleaned in an ultrasonic bath for (10) min and dried with oil-free air.

C- The discs in this group sandblasted by using (50) µm grain sized aluminium trioxide powder at a pressure of (3) bar from a distance of approx. (10) mm, for (20) seconds.

Following air particle abrasion, the discs cleaned in ultrasonic cleaner with distilled water for five minutes then dried with oil free air.After that the discs treated with ceramic primer (Clearfil ceramic primer, Kurary Inc-japan) that had been applied to the zirconia samples by using disposable brush tips and allowed to react for one minute then dried with air blow for (50) seconds and waited for its evaporation for (5) min according to manufacturer's instructions.

Preparation of teeth specimen:

A total of (60) human mandibular third molars free of cracks, fractures or caries were selected, cleaned by scaling and kept in saline till use. All teeth were embedded in autoploymerized acrylic resin cylinders at level of cemento-enamel junction. The teeth were mounted in acrylic cylinders with the help of surveyor. Hollow plastic cylinders with (15) mm internal diameter, (19) mm external diameter and (18) mm height were used with a self cure acrylic resin for the teeth embedment. The occlusal surfaces of all the teeth were reduced to remove all the enamel till dentin was exposed using a diamond stone disc under copious water coolant .Then dentin surface was finished with hand using 600-grit silicon carbide abrasive paper under copious water coolant for (60) seconds. After that the teeth randomly divide into six groups each containing (10) teeth.

Bonding procedure:

Dual polymerization resin cements were used (variolink, ivoclar – vivadent, lichtenstein) and (Dentocem, Itena ,France) as resin cement.

For the first main group variolink II will be used to bond (30) zirconia discs to (30) teeth, the dentin will be etched using 37% phosphoric acid for (15) seconds, washed with copious amount of water and dried, then syntac primer applied followed by syntac adhesive and lastly the bonding agent heliobond applied according to manufacturer's instructions.The exposed dentin surface was covered with a thin polythin sheet having an inner diameter of (6) mm. The variolink resin dispensed and applied to the zircon surfaces. A special loading device was designed to apply a constant load of (250) mg to the discs. This load was used to create a uniform layer of cement. Initial photopolymerization was performed for (20) seconds .Oxygen inhibiting gel was applied to the free surfaces. After (3) minutes the gel washed away, rinsed and dried. Excess cement was removed using microbrush. A constant load is still applied during removing excess cement and curing process. The luting agent was polymerized from each direction (mesial, distal, buccal, ligual and occlusal) with a halogen light cure unit for (40) seconds (Astralis 5, vivadent, Austria).

For the second main group Dentocem will be used to bond (30) zirconia discs to (30) teeth, first the dentin will be etched using 37% phosphoric acid for (15) seconds , washed with copious amount of water and dried, then Quick bond primer and bond applied, light cured for (20) .The exposed dentin surface was covered with a thin polythin sheet having an inner diameter of (6) mm. The Dentocem resin dispensed and applied to the zircon discs, bonded to dentin and cured in same manner as prescribed for the first main group.

After bonding procedure was completed all samples stored in distilled water at 37°C for (24) hours, then thermocycled for 1000 cycles between 5±°2 C and 55±°2C,with dwell time of (20)seconds and transfer time (5)seconds.

Shear bond strength test:

The specimen were fixed to the lower part of universal testing machine (Gunt, Hamburg, Germany)so that the base of the specimen were parallel to the shear force direction load applied at speed of 0.5 mm/min. till debonding occur and the maximum load necessary to debond was recorded.

After debonding all debonded discs and teeth were examined under stereomicroscope to determine the type of failure according to Johnson etal(5) as follow:

1-cement mainly on prepared dentin (adhesive).
2-cements on both disc and dentin (cohesive).
3-cements mainly on disc (adhesive).

Statisticalanalysis:

Data were analyzed using the statistical package for social sciences (spss). Differences between the means of three surface treatments groups were examined by ANOVA, post hoc test (LSD) was used to compare between means of two each specific groups. Independent T-test was used to compare between each two resin cements groups.

Fisher`s exact test was used to compare any statistical significant differences between the testing groups of two types of cement for the failure results (proportion).

P values of 5% and more were regarded as statistically insignificant, whereas values less than 5% ($p<0.05$) were considered as significant and those values less than 1% ($p<0.01$) were considered as highly significant.

Results

The results of the shear bond strength values of variolink resin cement were 7.83 Mpa for laser irradiated group, 8.39Mpa for hydrofluoric etched group and 10.42 Mpa for sandblasted+ ceramic primer treated group Table (1), while the values of Dentocem resin cement 8.06 Mpa for laser irradiated group, 9.18 Mpa for hydrofluoric etched group and 11.37 Mpa for sandblasted+ ceramic primer group Table (2).

The highest shear bond strengths of variolink and dentocem resins were obtained in sandblasted ceramic primer groups and the lowest were obtained in laser irradiated groups.

Dentocem resin cement showed better bonding than variolink II with significant difference for CO_2 laser groups and highly significant difference for HF acid etching and sandblasting primer groups Table(3).

The mode of bond failure was either adhesive (between cement and zirconia) in test groups for Variolink II resin cement (CO_2 irradiated specimens: 80%, HF etched : 70 % and sandblasted+ ceramic primer:40%) or cohesive (part on disc and part on dentin) (CO_2 irradiated specimens: 20%, HF etched :30% and sandblasted+ ceramic primer 60%),while for Dentocem resin cement were (CO_2 irradiated specimens: 70%, HF etched : 60 % and sandblasted+ ceramic primer:40%) or cohesive (part on disc and part on dentin) (CO_2 irradiated specimens: 30%, HF etched :40% and sandblasted+ ceramic primer 60%)

The statistical analysis of data by ANOVA showed a statistically highly significant difference ($P<0.001$) in shear bond strength of all the surface treatments groups Table (1and 2).

Least Significant Difference test was used to investigate where the significant did occure.The Post Hoc test (LSD) showed significant results between all groups.

The statistically Fisher`s exact test showed a no significant difference between the groups in the site of bond failure of two types of cements (Variolink-P=0.238) and (Dentocem-P=0.249).Table (4,5).

Discussion

Selection of CO_2 laser type to be used in our current study based on the previous findings by stubinger etal(6) they reported that CO_2 laser revealed a distinct surface alterations to zirconia surface at various laser parameters. This could be attributed to that the CO_2 laser beam does not penetrate the ceramic surface or cause surface preservation as other laser types. And since the principal effect of laser energy is the conversion of light energy into heat and the most important interaction between laser and substrate is the absorption of the laser energy by the substrate(7) .The CO_2 laser showed complete absorption by the zirconia surface So, this explains why CO_2 laser was selected(6). CO_2 laser was applied manually on the discs in a circular motion to expose the entire surface, as the laser beam diameter is 0.4mm only. Many studies showed that using low power settings produced nearly no changes in the surface roughness measurements while using 20 watt Power for 30 seconds without resting periods produced excessive melting of the ceramic surface that was seen directly without SEM. The laser parameters applied were (20 watt Power, for 10 seconds repeated 3 times with resting periods of 10 seconds and 200J Energy for each shot. Using these parameters, the surface destruction that might be in the form of cracks or excessive melting was prevented.(1,7)

The higher roughness of the laser treated samples could be attributed to the effect of the laser energy discharge that promoted a distinct surface changes in the form of pores caused by material removal by the laser and elevations caused by fusing and melting of the most superficial zirconia layer and its resolidification. The studies found a diverse micromechanical retention pattern originated by laser application, which is completely different from the other surface treatments.(4,8)

The results of this study agree with the results of sankin etal (9) but disagree with the results of Kara etal(10) ,this may be due to different types of laser with different times and output power used.

Effect of HF acid on zirconia, Consequently, HF acid is widely used in dental laboratories in conventional fixed prosthodontics and in adhesive all-ceramic applications. The influence of the type and the concentration of the etching agent on the ceramic structure was clearly demonstrated and almost all the authors agree on the higher bond strength of HF acid-etched ceramics than that of polished or phosphoric acid etched ceramics. Different pore and groove dimensions were observed in etched zirconia samples, as some surface modifications were evident, such a phenomenon was due to the HF acid ability of selectively removing the glassy matrix of zirconia, exposing the crystalline structure. This occurrence resulted in an increase of the surface area, which would facilitate the penetration of the luting agent into the micro-retentions of the acid etched zirconia.(11,12)

HF acid etching was found to result in sufficient bond strengths in the present study, it achieves proper surface texture and several other studies have also demonstrated that HF acid etching is the most effective surface treatment. Akyil et al.(13)found the bond strength of HF acid etching higher than the present study, the difference can be attributed to the different concentration and etching time.

Primers in dentistry are organic compounds that contain polymerizable groups, such as (meth) acrylates, in one end and silane alkoxy groups in the other. The (meth) acrylate functional groups can polymerize with an organic matrix of dental resin materials (e.g., dental resin cements, composites, adhesives). The silane alkoxy group can react with a hydroxylated surface, like zircon material *via* a chemical covalent bond. (14,15) Studies showed that phosphate-base primers which contained no silane were not effective to improve resin bond strength. The primers containing only silane monomer was the most effective for improving resin bonding to zirconi material. (16)

The surface treatment with primers containing functional monomers such as Clearfil Ceramic Primer, Kuraray Medical Inc., Japan are often recommended to improve the bonding to zirconia. At the same line the combination of primers and air-abrasion methods tend to produce better bond strength, as the air abrasion tend to roughen the surface of zirconia and producing a mechanical pores and irregularities for resin cement interlocking.(17,18)

According to Kern et al(19) air-abrasion without primers can result in higher initial bond strength to zirconia ceramics that will be reduced to zero in long-term evaluation, independent of air-abrasion application pressure, so combination of air abrasion with primer found to produce long lasting bond of resin cement to zirconia.

The adhesion of all-ceramic restorations, surface morphological changes, just like pores and grooves, are considered important to rheologic interlocking of resin cement to zirconia. Both micro-mechanical interlocking and the chemical bonding to the zircon surface increase the fracture resistance of the restored tooth and the restoration; provide high retention and improve marginal adaptation, preventing microleakage.(11,20)

Regarding the type of resin cements the results of the present study showed that the discs bonded with dentocem resin cement show better bonding than discs bonded with variolink .This may be explained by the fact that dentocem resin cement is a phosphate-monemer containing cement as the phosphate-monemers were found to be effective in improving zirconia bonding, this may be due to the fact that reactions may be formed between hydroxyl groups in the adhesive phosphate monomer and hydroxyl groups on the zirconia surface. (21,22)

For the two types of cements used in this study the type of failure in sandblasted primer treated groups showed few adhesive failure at interface between discs and cement due to good mechanical and chemical bonding of resin cement to zirconia ,while HF acid etched groups showed less adhesive failure at interface between discs and cement than CO_2 irradiated groups, this may be explained by the fact that HF acid produced more roughness and irregularities on surface of zirconia than CO_2 laser so producing more surface area for mechanical interlocking of resin cement to zirconia and creating better bonding.(23,24).The cohesive failure that occurred in some specimen may be occurred due to that the resin cement used had a dual cure ,acid base reaction and resin monomer polymerization, this may cause increase the stiffness of the cement beside the good mechanical inter-lock that was obtained between the cement and the zirconia is greater than that within the cement itself.(25) No adhesive failure at the interface between the resin cement and dentin was noticed ,this may be due to stronger bonding of resin cement to dentin than to zirconia discs.

Conclusion

Within the limitations of this study, the following conclusion can be withdrawn, the bonding of Zirconia can be improved by the different surface treatments especially by sandblasting ceramic primer. Dentocem resin cement showed higher shear bond strength than variolinkII so promoted better adhesion between dentin and zirconia. .

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Table (1) : Mean of shear bond strength of Variolink II resin cement with different surface treatments.

Surface treatments	N	Mean	SD	P by ANOVA	Significance by LSD test	P (LSD Test)
a-CO2 laser	10	7.835	.225	< 0.001	a X b	< 0.001
b-HF acid	10	8.395	.293		a X c	< 0.001
c-Sandblasting+primer	10	10.423	.302		b X c	< 0.001
Total	30	8.884	1.162			

Table (2) : Mean of shear bond strength of Dentocem resin cement with different surface treatments.

Surface treatments	N	Mean	SD	P. by ANOVA	Significance by LSD test	P (LSD Test
a-CO2 laser	10	8.0620	.23682	< 0.001	a X b	< 0.001
b-HF acid	10	9.1820	.21953		a X c	< 0.001
c-Sandblasting+primer	10	11.3760	.35712		b X c	< 0.001
Total	30	9.5400	1.42541			

Table(3) : Mean shear bond strength by type of material and surface treatments.

Surface treatments	Material	N	Mean shear bond strength	SD	p
CO2 laser	Variolink II	10	7.835	.225	.041
	Dentocem	10	8.062	.237	
HF acid	Variolink II	10	8.395	.293	< 0.001
	Dentocem	10	9.182	.220	
Sandblasting+primer	Variolink II	10	10.423	.302	< 0.001
	Dentocem	10	11.376	.357	

Table(4):The failure site in shear test of variolink resin cement.

		Failure		
		Score 1	Score 2	p value (Fisher's exact test)
Surface treatments	a-CO2 laser	8 80%	2 20%	. 238
	b-HF acid	7 70%	3 30%	
	c-Sandblasting+primer	4 40%	6 60%	

Table(5):The failure site in shear test of Dentocem resin cement.

		Failure		
		Score 1	Score 2	p value (Fisher's exact test)
Surface treatments	a-CO2 laser	7 70%	3 30%	. 249
	b-HF acid	6 60%	4 40%	
	c-Sandblasting+primer	4 40%	6 60%	