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Evaluation of the effect of different conditioning methods on core-veneer bond strength of zirconia restorations an *in-vitro* study

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Abstract

The aim of this study was to evaluate and compare shear bond strength of zirconia core to veneering porcelain after different methods of surface conditioning of the core.

Materials and methods: 40 zirconia blocks were milled in the CAD/CAM system and divided into four groups (n=10). After sintering, Group A, control, no surface conditioning, group B specimens were subjected to air-borne particle abrasion with 110 μ aluminium oxide, Group C specimens were sandblasted with silica coated alumina particles. For Group D a mixture zirconia powder and adhesive were applied on the surface of the specimens before sintering.

All these specimens were veneered with dimensions 3mm length, 5mm width and 3mm in height. Specimens were subjected to shear force and the shear bond strength was calculated. Fractured specimens were observed under scanning electron microscope.

Results: The mean shear bond strength values in MPa were 40.06, 41.91, and 42.89, 47.24 for group A, B, C and D respectively and statistically significant ($p < 0.05$). Group D demonstrated higher shear bond strength than other groups. Scanning electron microscope evaluation showed cohesive fracture in the veneering porcelain in modified zirconia, while the other groups principally fractured at the core-veneer interface.

Conclusion: Surface conditioning of zirconia core with zirconia powder slurry significantly improved the shear bond strength.

Keywords: Shear bond strength, surface conditioning, zirconia core-veneer bond

1. Introduction

During the past 40 years the porcelain fused to metal restoration has proven to be a reliable treatment option for fixed partial denture. However, the rising interest in esthetic dentistry as well as questionable biocompatibility of some dental metals and alloys has accelerated the development of alternatives to metal ceramic restorations^[1].

The success of all-ceramic crowns and patient demand for metal free, tooth coloured restorations has led to the development and introduction of restorative systems for all ceramic fixed partial denture^[2]. Different All-Ceramic materials are available are glass ceramics like lithium disilicate, leucite and feldspathic, aluminium oxides and zirconia like yttrium-oxide partially stabilized zirconia (Y-TZP).

Zirconia based ceramics are recommended for posterior fixed dental prosthesis as they have the highest failure loads when compared to alumina and lithium-disilicate based ceramics^[3]. The main disadvantage of zirconia based restorations being delamination of veneering porcelain or chipping. The incidence of chipping or fracture of veneering porcelain ranges from 11.4% to 25% of all fixed dental prosthesis.

The core-veneer bonding strength may be affected by different factors such as mechanical retention due to the surface roughness of the core, mismatch on thermal expansion coefficient (TEC) of the core and veneer ceramics and its resulting pre-stresses, transformation of zirconia crystals at core-veneer interface due to the thermal influences or stress loading, structure defects or flaws development at the interface between zirconia core and veneering ceramics and wetting properties and firing shrinkage of the veneering ceramics^[4]. It was reported that the bond strength and the mode of failure were significantly affected by some surface treatments such as air-borne particle abrasion or use of liner material, surface modification of

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core and type of zirconia framework material [4, 5].

In the literature to overcome the delamination of veneering porcelain different surface conditioning processes like, grinding, airborne-particle abrasion with alumina, liner application, silica coating of zirconia core and zirconia powder coating were recommended over the zirconia core surface with varied results.

This proposed study was undertaken to evaluate the effect of different surface conditioning methods like air abrasion with Aluminum oxide, silica particles and modification with zirconia powder on the shear bond strength of one of the commercially available zirconia core to veneering ceramics. The null hypothesis was the shear bond strength of Y-TZP to veneering ceramic would not be altered by the proposed surface conditioning method.

2. Materials and methods

Testing method followed those standardised by the American National Bureau of standards for flexural strength.

2.1 Materials

Zirconia blanks (Ceramil®, Amannirbach, Austria, Germany), the veneering porcelain (VITAVM®9, Vita zahnfabrik, Germany), Aluminium oxide particles of size 110 microns (Korex, Bego), Silica particles of size 50 microns (Rocatector, 3M), Zirconia powder (Ceramil® Amannirbach Austria Germany), Adhesive (Zir-liner, Vita zahnfabrik, Germany), Araldite (Petro Araldite Pvt Ltd, India)

2.2 Preparation of the Zirconia core specimens

A total of 40 zirconia blocks were milled with a milling machine in the CAD/CAM system.

2.3 Surface conditioning and grouping of the specimens

The 40 zirconia core blocks were divided into four groups (A B C D) containing 10 blocks each, and were subjected to four different types of surface treatments as follows:

Group A (Control)-A group of 10 blocks were sintered to full density in a heat furnace at 1400⁰ C for 90 minutes. After sintering Adhesive (zir liner) was applied to these 10 zirconia blocks as a single coat on to the surface to be veneered with a thin brush and grouped as A.

Group B-A group of 10 blocks were sintered to full density in a heat furnace at 1400⁰ C for 90 minutes. These zirconia blocks were then subjected to air abrasion in the sand blasting machine using 110 μ aluminium oxide at a distance of 10mm for 20 seconds. Then single coat of adhesive was applied with a thin brush on the surface to be veneered and were grouped as group B.

Group C-Another set of 10 blocks were sintered to full density in a heat furnace at 1400⁰ C for 90 minutes. These sintered zirconia blocks were subjected to air abrasion in the sand blasting machine using 1:1 ratio of 50 μ silica mixed with aluminium oxide 110μ at a distance of 1cm for 20 seconds. Single coat of adhesive was applied to these specimens with thin brush on the surface to be veneered and were grouped as group C.

Group D- Zirconia powder of 3μ was mixed with an adhesive Araldite in 1:1 ratio to form a slurry and was applied twice with a thin brush to the surface of presintered 10 zirconia core blocks. These blocks were sintered to full density in a heat furnace at 1400⁰ C for 90 minutes. Grouped as group D.

All 40 zirconia core specimens (10x5x5mm) ultrasonically cleaned for 10 minutes and air dried.

2.4 Veneering the zirconia blocks

All the four groups (A B C D), were veneered with veneering porcelain (VITAVM®9, Vita zahnfabrik, Germany) to uniform dimensions of 3.0 mm in length, 5.0 mm in width, and 3.0 mm in height according to the manufacturer's instructions.

2.5 Shear bond strength test

The specimens were placed in a specimen holder and mounted in a universal testing machine as shown in (fig 1). Load was applied parallel to the long axis of the specimens, and as close as possible to the interface, with a piston at a constant cross head speed of 0.5 mm / min till it fractures. The maximum force (N) at which the veneering porcelain had chipped off from core material was evaluated by the universal testing machine. Shear bond strength (Mpa) was calculated by dividing this force (N) with bond surface area that is veneering porcelain surface area. The mean surface area of veneering porcelain was 15mm.

$$\text{Shear bond strength} = N/A$$

The values obtained were the load (N) required to fracture the specimens. Then the shear bond strength (Mpa) was calculated by dividing the load (N) by the bonding area (mm²).

The data obtained was statistically analysed.



Fig 1: Testing for bond strength on universal testing machine.

2.6 Scanning electron microscope (SEM) examination

A scanning electron microscope was used to analyse the surface morphology of the core specimens after receiving different surface conditioning.

3. Results: The data was statistically analysed using One-way ANOVA test ($\alpha=.05$) – for multiple group comparisons and Tukey's post hoc pairwise comparisons test ($\alpha=.05$) – to identify pairwise differences.

Table 1: Comparison of Shear bond strength between the four study groups using ANOVA

		N	Mean	SD	ANOVA	
					F value	p-value
Shear bond strength (mpa)	Group A	10	40.06	3.61	8.02	<0.001*
	Group B	10	41.91	2.61		
	Group C	10	42.89	2.68		
	Group D	10	47.24	4.39		

$P < 0.001$ highly significant * $P < 0.05$ statistically significant $P > 0.05$ non-significant, NS

On comparing the four study groups the mean shear bond strength obtained for group D was highest 47.24 ± 4.39 followed by group C (42.89 ± 2.68), group B (41.91 ± 2.61) and group A (40.06 ± 3.61) in the decreasing order. $P < 0.001$

which indicates the results are statistically highly significant. As the results were statistically highly significant ($P < 0.001$) the multiple comparisons (post hoc test) was done using Tukey's range test.

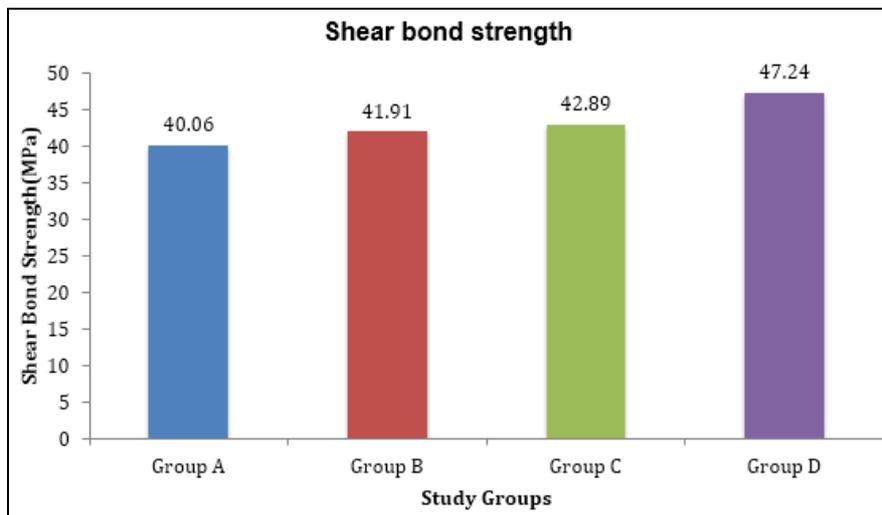
Table 2: Pairwise Comparison of Force and shear bond strength between the four study groups using Tukey's Post hoc test

Dependent Variable	(I) Group	(J) Group	Mean Difference (I-J)	Std. Error	p-value
Force (new tons)	Group A	Group B	-27.70	22.81	0.62(NS)
		Group C	-42.40	22.81	0.26(NS)
		Group D	-107.70	22.81	<0.001**
	Group B	Group C	-14.70	22.81	0.92(NS)
		Group D	-80.00	22.81	0.006**
	Group C	Group D	-65.30	22.81	0.03*
Shear bond strength (mpa)	Group A	Group B	-1.85	1.52	0.62(NS)
		Group C	-2.83	1.52	0.26(NS)
		Group D	-7.18	1.52	<0.001**
	Group B	Group C	-0.98	1.52	0.91(NS)
		Group D	-5.33	1.52	0.006**
	Group C	Group D	-4.35	1.52	0.03*

** 0.001 highly significant * $P < 0.05$ statistically significant. $P > 0.05$ non-significant, NS

On pairwise comparison among the four groups(A,B, C and D) force and shear bond strength was found to be highest in group D. when compared to other groups- group A vs group

D ($P < 0.001$), group B vs group D ($P < 0.001$), group C vs group D ($P < 0.03$).



Graph 1: Comparison of four groups (A B C D) with respect to shear bond strength

3.1. Scanning electron microscope (SEM) evaluation

3.1.1. Surface morphology of the core specimens

SEM images showed different surface morphologies of the specimens that received the various surface treatments. After airborne particle abrasion, the surface was rough, and the grains were no longer discernible. Modified zirconia was characterized by rugged surface and abundant micro porosities, which would allow the penetration of the veneering porcelain.

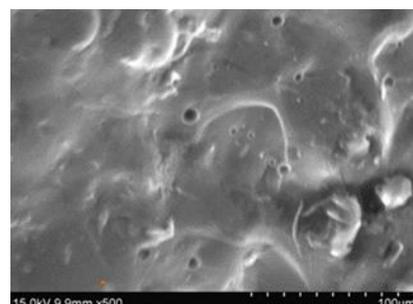
3.1.2. Fracture topography analysis

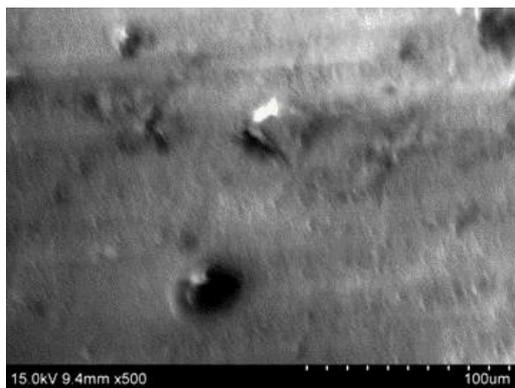
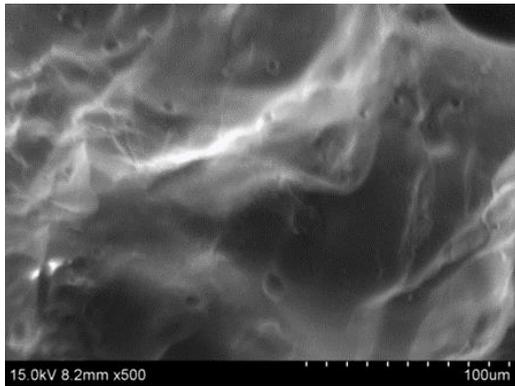
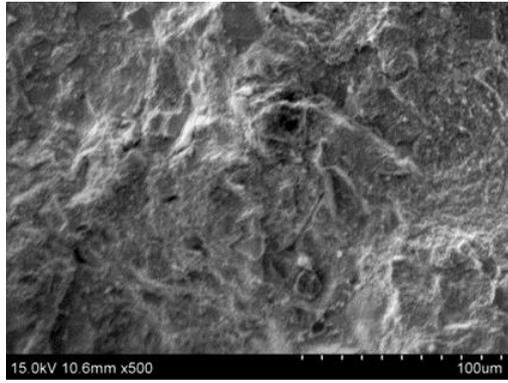
Cohesive fracture within the veneering porcelain was the principle failure mode of the modified groups, B, C, D, while the primary mode in the control group A was the adhesive fracture.

According to the SEM images, much less veneering porcelain remained on the surfaces of core materials than on the fractured zirconia specimens, characterized by a thin layer of veneering porcelain with embedded zirconia particles, which covered the zirconia surface. In some locations, the fracture of modified zirconia specimens propagated through the zirconia

particles within the coatings, while veneering porcelains filled the micro porosities and encapsulated the zirconia particles, the melted veneering porcelain infiltrated the intricate micro porosity and produced a hybrid layer on the zirconia surface. This significantly increased the bond strength of zirconia to porcelain and changed the primary failure mode from adhesive fracture to cohesive fracture within the veneering porcelain.

3.1.3. SEM images of groups A, B, C, D respectively





4. Discussion

Most of the ceramic systems are multilayer structures with high-strength core overlaid by weak but esthetic veneering porcelains. The long term success not only depends on the superior mechanical strength of the core, the strength usually determined by its weakest component [6]. The bond strength between the core- veneer the weak point of ceramic restorations. [7, 8], The literature in early ceramic systems shows that the primary failure modes were the fracture of both the veneering porcelain and the core but not the veneering porcelain fracture in the early ceramic systems, like alumina and lithium disilicate based ceramics) [9].

Recent advances in the ceramic systems demonstrated that adequate bond strength had developed between the core materials and their correspond-ing veneering porcelains [10, 11]. However, the most common failures of zirconia restorations was found to be delamination of veneering ceramics. Sufficient bond strength between the veneering ceramics and core is therefore a concern for the long-term clinical success of zirconia restorations. Although the bonding mechanisms between zirconia and veneering porcelains are not well understood, their mechanical interlocking is essential. Several methods, shear bond, 3- and 4-point flexure, tensile, and micro tensile bond tests, were suggested for evaluation of the bond strength of veneering porcelain to frameworks, shear

bond test being the most prevalent It is crucial that the bonding interface be the stressed region, regardless of the test method used [12].

There are several methods to measure shear bond strength. In the present study, was Schmitz-Schulmeyer test [13], a planar rectangular interface shear bond test was done. This test has minimal experimental variables and has proven to be a reliable well-suited test set up for metal ceramic bond strength measurements. The stresses during the shear tests were reported to be directed mainly at the interface resulting in a relatively uniform distribution of interfacial stresses [1]. Therefore this design was transferred for the application of all- ceramic systems and chosen for the present study.

Among the four groups tested, highest shear bond strength was observed in group D (modified zirconia, 47.24) followed by silica coated with alumina group C (42.89), air abrasion with alumina group B (41.91) and group A (40.06). The shear bond strength of those veneering ceramics intended for use on zirconia range from 21.9 to 31.0 MPa. The results achieved required the rejection of null hypothesis, since there is differences in the mean shear bond strength values between the groups.

On comparing, the mean shear bond strength of air-borne particle abrasion (group B) is greater than control group (group A). Which was comparable to that of previous studies done by Aboushelib *et al.* [14], Kim *et al.* [15] and Kosmac *et al.* [16]

Air-borne particle abraded with alumina was assumed to enhance the bond strength of core to veneering porcelain by increasing surface roughness and providing undercuts. However, the results of other studies demonstrated that air-borne particle abrasion did not effectively increase the shear bond strength between zirconia and veneering porcelain. Air-borne particle abrasion might compromise the mechanical strength of the ceramic by initiating surface defects that can become stress concentration sources, leading to failure. Air-borne particle abrasion not only increases surface roughness but also causes phase transitions from tetragonal to monoclinic in the zirconia surface. Superficial layer of the monoclinic phase results in tensile stress in the veneering layer, which has a negative effect on the bond strength because the coefficient of thermal expansion of monoclinic zirconia is significantly lower than that of tetragonal zirconia. In air-borne particle abrasion the SEM analysis shows that fracture pattern of veneer specimens was mainly combined as adhesive at the interface and cohesive in the veneering ceramic. These failure modes are comparable to the results of other *in-vitro* studies, where crack deflection has been identified at the core-veneering interface [4,5] Guess *et al.* [1]. interpreted reason for this kind of fractures in two ways: first, crack deflection could be an excellent property of Y-TZP to resist crack propagation. Second, the inter-laminar crack deflection could be related to the poor bonding strength between zirconia and veneering ceramic.

In the present study on comparing the mean shear bond strength of silica coated with alumina particles (group C) is greater than control group that is no surface treatment done (group A). Which was comparable to that of previous study done by Kosmac *et al.* [16]

The specimens were treated with 50µm silica coated alumina particles. Air-borne particle abrasion increases the surface roughness and surface contact areas, which reduces the interfacial failure of zirconia core and veneering porcelain. Kosmac *et al.* concluded that air-borne particle abrasion may be considered as a powerful clinical technique for

strengthening Y-TZP.

Silica coated with alumina particles with a shear bond strength of (group C) 42.89 MPa is lower than modified zirconia which had shear bond strength of (group D) 47.24 MPa. This proves that the use of silica as a surface-modifying agent is less effective than what is seen with modified zirconia group.

In silica coated alumina particles SEM analysis shows that fracture began at the zirconia-ceramic interface and proceeded into veneering porcelain or fracture began within the veneering porcelain itself. The interfacial image of the core and veneering ceramics showed a roughened surface created by air-borne particle abrasion.

In the present study on comparing the mean shear bond strength of modified zirconia (group D) is greater than control group. The results of the present study is comparable to that of previous study done by Phark *et al*, Teng *et al*.^[5]

As seen in various other studies, several approaches have been introduced to increase the surface roughness of ceramics to obtain satisfactory mechanical interlocking. Phark *et al*. introduced a porous surface for zirconia to composite resin. However, only Teng *et al*.^[5] Tried testing this surface-modifying method for zirconia to veneering porcelain bonding. In this present study, in group D the same method was tried by application of thin layer of zirconia powder over pre-sintered zirconia surface to obtain micro porosities. Thin layer of veneer porcelain was then fired which allowed the molten veneering porcelain to penetrate and fill the micro porosities to generate a hybrid layer of porcelain and zirconia at the interface. This modified surface possessed a rough, micro porous structure, which differed from the other surface treatments and provided interlocking bonding for veneering porcelain.

SEM analysis of the fractured specimens demonstrated that the fracture of modified zirconia specimens occurred within the veneering porcelain, leaving a thin layer of veneering porcelain on the zirconia surface. This is because porcelain infiltrated into the pores of the modified zirconia surface and interpenetrated with zirconia particles. These findings indicate that the weak link in the modified zirconia specimens was in the veneering ceramic rather than in either the zirconia coating itself or veneering ceramic bonding to zirconia substrate. Teng *et al*.^[5] concluded that modifying the zirconia surface with powder coating significantly increases the shear bond strength of zirconia to veneering porcelain. Thus it could be concluded that the bond strength of modified zirconia to the veneering porcelain was higher than the bulk strength of the porcelain.

The modification of zirconia surface in group D has shown a significant increase in the shear bond strength when compared with the group A, B and C. This is also evident from the studies conducted by Teng *et al*. who have shown in their research that the modified group has greater than or equal mean shear bond strength values when compared to metal ceramic system.

Thus with above mentioned reasons it can be concluded that the modifying the zirconia surface with powder slurry could significantly increase the shear bond strength of zirconia to veneering porcelain when compared to air-borne particle abrasion.

5. Conclusion

Within the limitations of the study following conclusions were drawn:

The effect of different surface conditioning methods increased

the shear bond strength of zirconia core and veneering ceramic. A porous surface for zirconia ceramic was produced by zirconia powder coating which significantly improved the zirconia to porcelain bonding as compared with alumina and silica air-abrasion surfaces.

No significant improvement in bond strength was observed in alumina and silica air-borne particle abrasion groups.

Scanning electron microscope evaluation showed cohesive fracture in the veneering porcelain was the predominant failure mode of modified zirconia, while the other groups principally fractured at the core-veneer interface.

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