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A comparative evaluation of the shear bond strength of monolithic zirconia to teeth enamel following surface treatment using a zirconia etching solution and airborne particle abrasion: An Invitro Study.

[1]Dr.Arghya Nayak, Post Graduate Student, Department of Prosthodontics & Crowns, Bridges & Implantology, D.A.P.M R.V Dental College and Rajiv Gandhi University of Health Sciences, Bengaluru, India.

[2]Dr.Roshan P Kumar, Reader, Department of Prosthodontics & Crowns, Bridges & Implantology, D.A.P.M R.V Dental College and Rajiv Gandhi University of Health Sciences, Bengaluru, India.

[3]Dr.Kalavathy N, Prof and HOD, Department of Prosthodontics & Crowns, Bridges & Implantology, D.A.P.M R.V Dental College and Rajiv Gandhi University of Health Sciences, Bengaluru, India.

Corresponding Author: Dr. Arghya Nayak, Post Graduate, Department of Prosthodontics & Crowns, Bridges & Implantology, D.A.P.M R.V Dental College and Rajiv Gandhi University of Health Sciences, Bengaluru, India.

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Abstract

Background: Zirconia was introduced in the early 1990s as an alternative to metals. The production of dental prostheses using zirconia has been at its peak in the recent years.Zirconia is a bio-inert material and this property of zirconia has made it very challenging to etch the surface of zirconia. This bio-inertness of zirconia has made bonding to tooth structure, titanium, or other ceramic materials challenging. As a result, surface treatments or conditioning techniques are required to achieve predictable and durable adhesion. Hydrofluoric acid (HF) (9.5%) solution has been used to etch zirconia by extending the immersion time (up to 24 hours) or by elevating the temperature (80°C) for up to 30 minutes. Additionally, HF solutions of higher concentrations have been used. However, these protocols have failed to improve adhesive bonding to zirconia.

Objective of this study: The aim of this study was to evaluate and compare the mean shear bond strength of monolithic zirconia to teeth enamel following surface treatment using a zirconia etching solution and airborne particle abrasion.

Methodology: A total of 20 samples of monolithic zirconia were fabricated as per the standard protocol, 10 of the specimens (Group A) were etched with Bioden Zircos-E Etching solution and the other 10 (Group B) were airborne particle abraded. The specimens were then tested for shear bond strength under a universal testing machine post bonding with a self-adhesive resin cement. A failure mode analysis was done using a scanning electron microscope to assess where and what might be the cause for bond failure.

Results: The mean shear bond strength of group A and group B was 63.13±14.70 and 175.14±34.26 respectively. Group A reported a predominantly adhesive failure and Group B reported a predominantly cohesive failure.

Conclusion: The mean shear bond strength of the monolithic zirconia specimens that were etched with the Bioden Zircos-E etching solution was 63.13±14.70 MPa. The mean shear bond strength of the monolithic zirconia specimens that were airborne particle abraded was 175.14±34.26 MPa. The mean shear bond strength of the zirconia specimens was evidently higher in the airborne particle abraded specimens in comparison to the etched specimens.

Keywords: Airborne particle abrasion, Bioden Zircos-E Etching solution, Monolithic Zirconia, Shear Bond Strength, Surface Roughness

Introduction

Zirconia was introduced in the early 1990s as an alternative to metals and has been used as a core material to support various veneering ceramics.[5] It exhibits exceptional mechanical properties and is the strongest of all the dental ceramics. However, the crowns with zirconia as the core material exhibited some amount of veneering failure. The rates of chipping of zirconia veneering ceramics have been reported to be 2%-9% for single crowns after 2-3 years and 3%-36% for FDPs after 1-5 years.[6] Several modifications were tried – sintering high-strength CAD/CAM fabricated porcelain veneer onto a zirconia coping, applying veneering ceramic via pressing it over zirconia copings (press-on technique), and a combination of press on and layering veneer ceramics on zirconia copings (double veneering technique), to overcome the cohesive failures.[9,10,11] Recently, monolithic zirconia crowns have emerged as an alternative to the aforementioned methods.

Monolithic zirconia restorations offer many advantages over the conventional veneered restorations. Clinical studies have substantiated their use as functional posterior restorative materials.[12] They exhibit high mechanical strength, bring about limited wear of the opposing dentition, require minimal tooth preparation and may be the material of choice in cases with limited interocclusal space. However, zirconia is a bio-inert material and this property of zirconia has made it very challenging to etch the surface of zirconia. Also, unlike feldspathic porcelain or lithium disilicate ceramic, hydrofluoric acid (HF) is unable to etch zirconia efficiently.

Therefore, the conventional approaches to adhesive bonding to silica-based ceramics are not applicable to zirconia. The bio-inertness of zirconia has made bonding to tooth structure, titanium, or other ceramic materials challenging.[1] As a result, surface treatments or conditioning techniques are required to achieve predictable and durable adhesion.

Hydrofluoric acid (HF) (9.5%) solution has been used to etch zirconia by extending the immersion time (up to 24 hours) or by elevating the temperature (80°C) for up to 30 minutes. Additionally, HF solutions of higher concentrations (48%) have been used. However, these protocols have failed to improve adhesive bonding to zirconia.[1]

Surface treatments such as selective infiltration technique, laser treatment, and coating with nanostructured alumina or tribochemical silica have been proposed. However, clinically significant improvements have been lacking, and these alternative treatments have caused surface micro-cracks, weakening the material and making it more prone to crack propagation. As an alternative method of bonding to zirconia restorations, phosphate ester primer

10-methacryloyloxydecyldihydrogenphosphate (MDP) has been used successfully to improve adhesion to zirconia restorations.[1] Airborne-particle abrasion has been used to clean the surface of zirconia, remove impurities, increase surface roughness, and modify the surface energy and wettability. In addition, airborne-particle abrasion provides mechanical impingement of particles on the surface which results in a roughened surface and allows the resin cement to flow into these micro-retentive areas and create a stronger micromechanical interlock. Airborne-particle abrasion with alumina has been identified as a key factor in achieving a durable bond for zirconia-based ceramics. Different sizes of abrasive alumina particles have been used, without evidence of the superiority of one over another.

Recent in vitro studies report that airborne-particle abrasion may have a deleterious effect on the zirconia surface due to the creation of micro-cracks, which might reduce the flexural strength.[6] Moreover, the tetragonal phase of Y-TZP is converted to the monoclinic phase with volume expansion (4–5%) under the high stresses caused by airborne-particle abrasion, and this unique transformation can produce different types of damage that affect the structural integrity.

Recently, an etching solution for zirconia containing HF solution and nitric acid, sulfuric acid, hydrochloric acid and phosphoric acid (Zircos E Etching system) has been introduced that can etch the surface of zirconia at room temperature.[1] The effects of this etching solution on the bond strength and mechanical properties of zirconia are not known. Therefore, the purpose of this in vitro study was to examine the effects of the Zircos E etching solution on the shear bond strength of zirconia to teeth enamel and compare it to surface treatment of zirconia by airborne particle abrasion.

Objectives

The aims and objectives of our study were –

1. To evaluate the shear bond strength of zirconia to teeth enamel following surface treatment of zirconia using an etching solution (Bioden Zircos E Etching Solution).
2. To evaluate the shear bond strength of zirconia to teeth enamel following surface treatment of zirconia by airborne particle abrasion.
3. To compare the shear bond strength of zirconia to teeth enamel following surface conditioning using a zirconia etching solution (Bioden Zircos E Etching solution) and airborne particle abrasion.

Methodology

Preparation of the zirconia specimens: 20 disc shaped specimens of monolithic zirconia, measuring 4mm in diameter and 2mm in thickness were fabricated.[3] Pre-formed blocks of zirconium oxide were milled to create disc shaped specimens as per the specified dimensions. The specimens were be sintered, followed by routine finishing and polishing procedures. After finishing and polishing, the specimens were measured with a digital caliper to confirm appropriate dimensions.

Group A The total sample size was 20. The samples were grouped as Group A and Group B, each group comprising of 10 samples each.	Monolithic zirconia etched with ZES (10 nos)
Group B	Monolithic zirconia airborne particle abraded (10 nos)

Preparation of the teeth for bonding

Maxillary premolar teeth that were extracted for orthodontic purposes were used in the study.

Immediately following extraction, the teeth (premolars) were cleaned of surface debris and sterilized in a liquid sterilant (0.5% sodium hypochlorite). Teeth were selected from the sterilization liquid based on the following criteria: no evidence of caries, no restorations, and no cracks or fractures in the crown.[2] The teeth were placed in distilled water at room temperature over a period of 1 month. The teeth were gently air dried and embedded in autopolymerizing acrylic resin. The mounting procedure were done with the experimental surface of the tooth exposed and specimens were stored in distilled water. Tooth preparation were performed with minimum enamel reduction (0.5–1mm), using a high-speed hand-piece with water, and a coarse flat-end cylindrical diamond bur (No.6837KR; Brasseler, Savannah, GA) to get a flat planar surface for bonding. The specimens were then finished with silicone carbide strips to create a uniform flat surface.[3] The teeth were cleaned with fluoride free pumice, rinsed with water and dried gently to maintain a dry working field. The primer was applied to the prepared tooth structure, left for 30 seconds and gently air streamed until the surface appears glossy.

Bonding of the zirconia to prepared enamel surface following conditioning of the Zirconia surface using the etching solution: The specimens belonging to Group A were placed in Zircos E Etching solution container containing the Bioden Zircos E Etching solution for 30 seconds and then placed in a cold water ultrasonic bath for 30 minutes. The specimens were cleaned with a steamer and then degassed using a porcelain furnace with glaze program. The specimens were cemented at room temperature with a phosphate methacrylate resin luting agent (PanaviaF2.0;NY) to the prepared teeth, following the manufacturer's recommendations. Cementation of the zirconia to prepared enamel surface following surface treatment of the zirconia by airborne particle abrasion:

The specimens belonging to Group B were subjected to airborne particle abrasion using. Alumina particles of 50μm size under 100kPa pressure for 10 seconds at less than 10mm distance. These specimens were then cemented at room temperature with a phosphate methacrylate resin luting agent (Panavia F2.0; NY) to the prepared teeth, following the manufacturer's recommendations.

Evaluation of Shear bond strength: The cemented specimens of both groups were stored in distilled water at 37°C for 24 hours, then thermocycled in water at temperatures between 5°C and 55°C for 500 cycles, with a 15-second dwell time at each temperature.[3] Following thermocycling, specimens from both groups were loaded to failure in a universal testing machine in the shear mode, with a crosshead speed of 0.5 mm/min.

The ultimate load to failure was recorded in Newtons (N). The average bond strength (MPa) was calculated by dividing the maximum ultimate load to failure (N) by the bonded cross-sectional area (mm^2).

$$SBS = F_{max} / S$$

SBS – Shear Bond Strength

F_{MAX}- Max ultimate load to failure

S – Bonded cross sectional area[7]

The means and standard deviations were recorded. The fractured surfaces were then examined under a scanning electron microscope.

Scanning Electron Microscope study: The fracture interfaces of all zirconia specimens were tested under a scanning electron microscope (SEM) at 15× magnification (SEM, Model ISI-DS130; Akashi Beam Technology, Tokyo, Japan) to determine failure mode. Zirconia surfaces were also selected randomly from each group and inspected at 200× and 2000× magnification.

An adhesive failure is characterized by a complete separation of the cement from one or both surfaces of a specimen. A cohesive mode is characterized by resin covering both test surfaces of a specimen. A specimen was considered a mixed failure when a portion of the test surface shows exposed zirconia or enamel, and other areas having islands of retained resin.[3]

Statistical Analysis: Statistical Package for Social Sciences [SPSS] for Windows Version 22.0 Released 2013. Armonk, NY: IBM Corp., will be used to perform statistical analyses.

1. Independent Student t Test will be used to compare the mean shear bond strength (in Mpa) between 02 study groups.
2. Chi Square test will be used to compare the different modes of failure between 02 groups.
3. The level of significance will be set at $P<0.05$.
4. And any other relevant test, if found appropriate during the time of data analysis will be dealt accordingly.

Results

The following results were obtained:

The mean Shear Bond Strength (in MPa) between Etched and Sandblasted Zirconia groups was compared using an Independent Student t Test.

(a) Shear Bond Strength

- Group A exhibited a mean shear bond strength of 63.13 with a standard deviation of 14.70. (Table 1)
- Group B exhibited a mean shear bond strength of 175.14. The standard deviation was 34.26. (Table 1)
- The mean difference among the specimens was found to be -112.00 (Table 1

Scanning Electron Microscope study

The fracture interfaces of all zirconia specimens were tested under a scanning electron microscope (SEM) at 15× magnification (SEM, Model ISI-DS130; Akashi Beam Technology,Tokyo, Japan) to determine failure mode. Zirconia surfaces were selected randomly from each group and inspected at 200× and 2000× magnification.

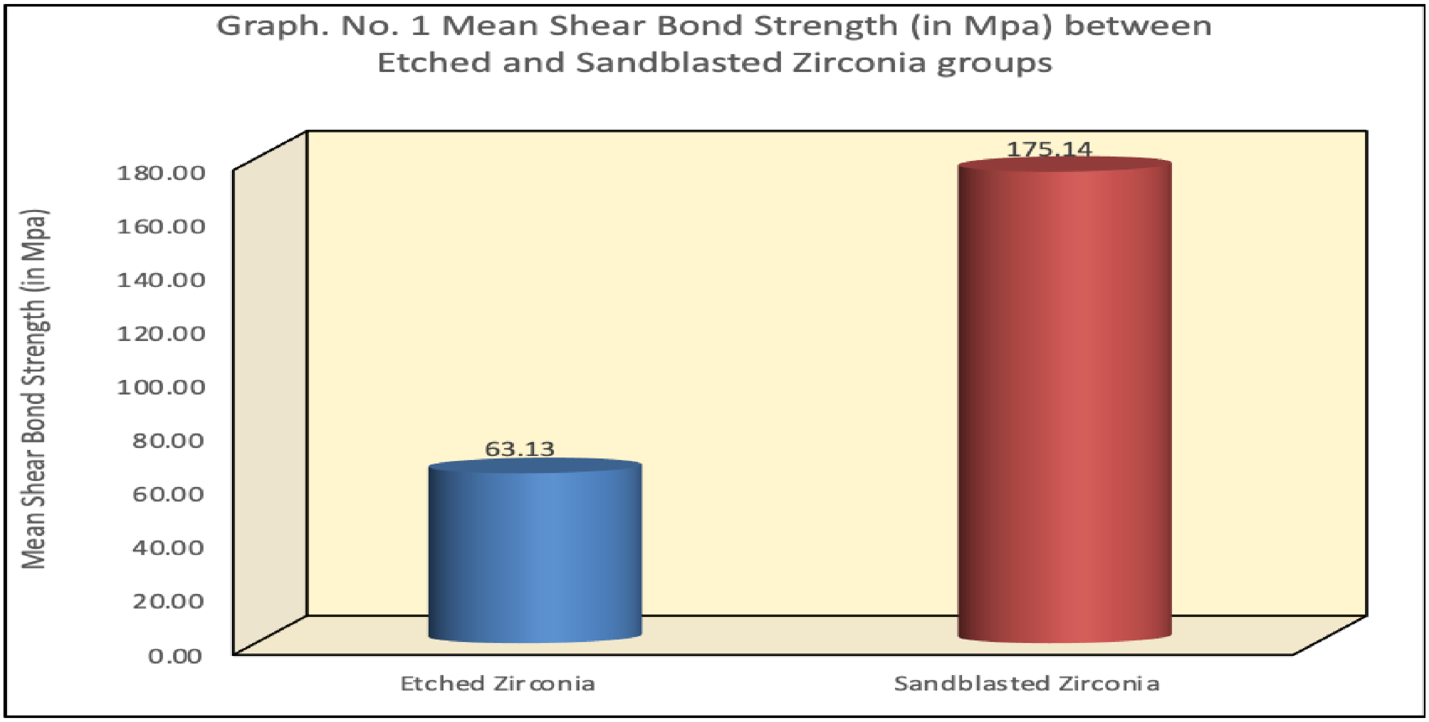
- Group A exhibited a predominantly adhesive type of failure. (Table 1)
- Group B exhibited a predominantly cohesive type of failure. (Table 1)

Table 1: Comparison of mean Shear Bond Strength (in MPa) between Etched and Sandblasted Zirconia groups using Independent Student t Test

Groups	N	Mean	SD	Mean Diff	Lower	Upper	t	P-Value
Etched Zirconia	10	63.13	14.70	-112.00	-136.76	-87.23	-9.501	<0.001*
Sandblasted Zirconia	10	175.14	34.26					

*** - Statistically Significant**

Table 1 illustrates the comparison of mean Shear Bond Strength (in MPa) between Etched and Sandblasted Zirconia groups. [Refer Graph no. 1]



This difference in the mean shear bond strength between Etched and sand blasted zirconia was statistically significant at P<0.001. [Refer Graph 1]

Table 2: Comparison of Modes of Failure between Etched and Sandblasted Zirconia groups using Chi Square Test

Mode of Failure	Etched Zirconia		Sandblasted Zirconia		$\chi 2$ Value	P-Value
	n	%	n	%		
Adhesive	7	70%	1	10%	8.100	0.02*
Cohesive	3	30%	7	70%		
Mixed	0	0%	2	20%		

*** - Statistically Significant**

Table 2 illustrates the comparison of modes of failure between Etched and Sandblasted Zirconia groups.

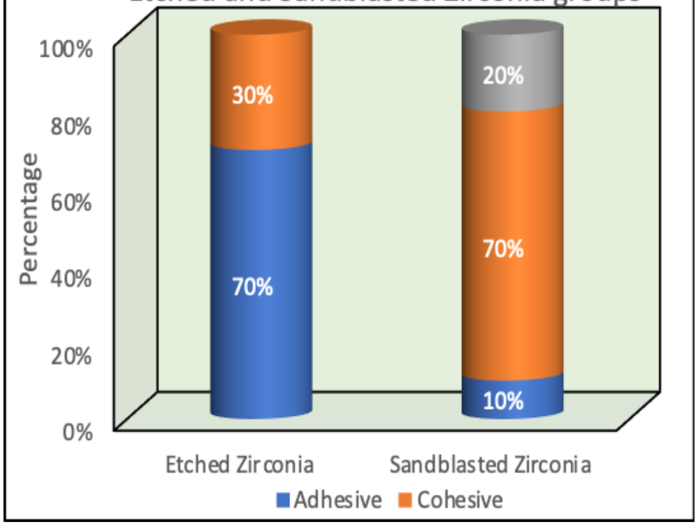
The test results demonstrate that the etched zirconia group predominantly showed adhesive type of failure [70%], followed by 30% cohesive failure as compared to sandblasted zirconia, which showed predominant cohesive failure [70%], followed by mixed failure of 20% & 10% adhesive failure. This difference in modes of failure between Etched and sand blasted zirconia was statistically significant at P=0.02. [Refer Graph no. 2]

* - Statistically Significant

Table 2 illustrates the comparison of modes of failure between Etched and Sandblasted Zirconia groups.

The test results demonstrate that the etched zirconia group predominantly showed adhesive type of failure [70%], followed by 30% cohesive failure as compared to sandblasted zirconia, which showed predominant cohesive failure [70%], followed by mixed failure of 20% & 10% adhesive failure. This difference in modes of failure between Etched and sand blasted zirconia was statistically significant at P=0.02. [Refer Graph 2]

Fig. No. 2 Modes of Failure between Etched and Sandblasted Zirconia groups



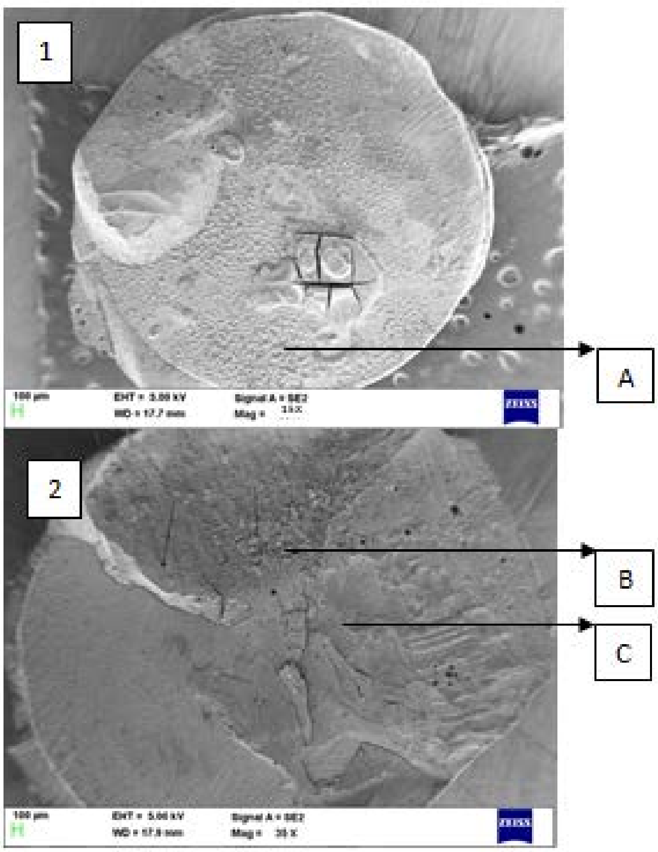


Fig. 3: Scanning Electron Microscope study for failure analysis, 1- SEM image of etched zirconia at 15x magnification, 2- SEM image of sandblasted zirconia at 15x magnification.

A- Etched zirconia surface

B- Sandblasted zirconia surface

C- Resin cement on sandblasted zirconia surface.

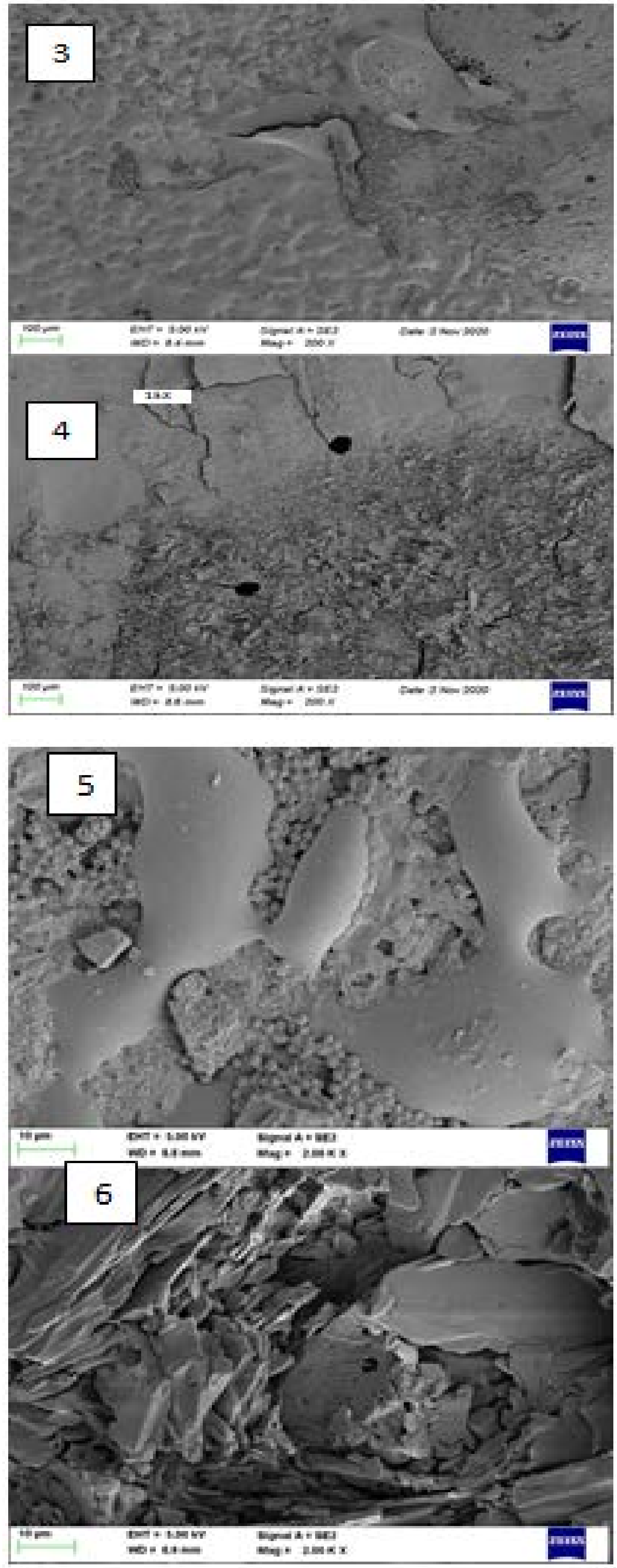


Fig.4 Scanning Electron Microscope study of Zirconia specimens 3- SEM image of etched zirconia at 200x, 4- SEM image of airborne particle abraded zirconia at 200x, 5- SEM image of etched zirconia at 2000x, 6- SEM image of airborne particle abraded zirconia at 2000x.

Discussion

The results of our study demonstrated that the mean shear bond strength testing in group B (Airborne particle abraded Zirconia specimens) was significantly higher than in Group A (Etched Zirconia specimens) with the values being 63.13±14.70 MPa in Group A and 175.14±34.26 MPa in Group B. This result can be attributed to the evident surface irregularities created by sandblasting on the group B specimens prior to bonding in comparison to Group A (Fig.5). On further evaluation, in Group A, 70% of the bond strength failures were adhesive and 30% of the bond strength failures were cohesive signifying that the failure that occurred was predominantly adhesive. In Group B, 70% of the failures were cohesive, 20% were mixed, and 10% were adhesive signifying that the failure was predominantly cohesive.

Furthermore, to understand the possible reasons for the kinds of failures occurred in our study and to assess the failure mode, a SEM analysis of the zirconia specimens was done which demonstrated significant morphological changes under the influence of acid etching with the Zircos-E etching solution and airborne particle abrasion. In Group A, the 10 samples that were etched with the Zircos-E solution predominantly demonstrated a significantly adhesive type of failure in 70% of the specimens which is characterized by complete separation of the cement from the zirconia specimen. The possible explanation for this is that the failure occurred within the resin cement to adhere itself to the zirconia specimen probably due to insufficient surface roughness created by the Bioden Zircos-E etching solution. The bond strength in Group A reported lower than Group B possibly because the concentration of the acid was insufficient or the etching time needed to be prolonged further. In Group B, the 10 samples that were airborne particle abraded with alumina particles of 50µm size under 100kPa pressure for 10 seconds at less than 10mm distance showed a predominantly cohesive type of failure in 70% of the specimens which is characterized by resin covering both the zirconia specimen and the tooth. The possible explanation for this is that the adherend failed before the adhesive leading to a predominantly cohesive failure in Group B. The bond strength in Group B reported higher values and a cohesive failure suggesting that the bond strength of the resin cement was insufficient and could be possibly influenced by thermocycling.

Conclusion

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

1. The mean shear bond strength of the monolithic zirconia specimens that were etched with the Bioden Zircos-E etching solution was 63.13±14.70 MPa.
2. The mean shear bond strength of the monolithic zirconia specimens that were airborne particle abraded was 175.14±34.26 MPa.
3. The mean shear bond strength of the zirconia specimens was evidently higher in the airborne particle abraded specimens in comparison to the etched specimens.

Appendix

Appendixes, if needed, appear before the acknowledgment.

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