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Comparative Assessment of Early and Long-Term Shear Bond Strength and Fracture Mode of Contaminated and Cleaned Zirconia Restorations Cemented with Two Different Cements – An In Vitro Study

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Abstract

Background: Yttria partially stabilized tetragonal zirconia polycrystalline restorations have gained widespread use because of its enhanced strength and esthetics. During the try-in process of the restoration, zirconia is likely to be contaminated with saliva. This contamination can later on lead to weakening of the bond between restorative material and cement and eventually failure of the restoration. For this reason, zirconia surface should be cleaned before cementation.

Method: Sixty samples of extracted human premolar teeth were used in the study. Full ceramic tooth preparation was done and zirconia copings of 2mm thickness were fabricated. They were divided into three groups based on salivary contamination. Group A: saliva-contaminated zirconia copings that are cleaned with Ivoclean (Group A-1, n=20) and distilled water (A-2, n=20) and Group B: Uncontaminated zirconia copings (n=20). Each group was further subdivided into two subgroups of each (n = 10). Subgroup A consisted of zirconia copings cemented with Resin Modified Glass Ionomer cement and Subgroup B consisted of zirconia copings cemented with Self-Adhesive Resin Cement. Prior to de-bonding, all samples were immersed in artificial saliva for 24 hours and 3months to check for the early and delayed SBS. This was followed by thermo cycling to stimulate the intra-oral conditions. After aging, all samples were tested to check for shear bond strength using universal testing machine with cross head speed of 0.5 mm/min. The fractured samples were tested under stereomicroscope to analyze fracture mode as adhesive, cohesive or mixed.

Results: The results of the present study results showed the maximum bond strength values for control group (uncontaminated zirconia copings) followed by salivary contaminated, cleansed with Ivoclean and least bond strength values for salivary contaminated, cleansed only with distilled water. The group comparisons for early SBS after 24 hours showed that all groups presented higher mean SBS results in comparison to the long term SBS mean values after 3 months of artificial aging followed by thermocycling. Spontaneous debonding also occurred during thermocycling. This was significant and was particularly pronounced in the distilled water test groups, where the thermocycled zirconia samples all debonded. Interestingly, the thermocycled uncontaminated samples only had one sample that spontaneously debonded.

Self-adhesive resin cement showed highest mean SBS values that were statistically significant at both early and long term shear bond testing in comparison to resin modified glass inomer cement for saliva contaminated and uncontaminated groups.

Conclusion: Salivary contamination significantly reduced shear bond strength of resin cements to zirconia. Ivoclean showed highest cleaning efficiency compared to distilled water cleaning. Also, the highest bond strength for both early and long term SBS was noticed with self adhesive resin cement in comparison to resin modified glass inomer cement.

Keywords: Ivoclean, salivary contamination, artificial saliva, self-adhesive resin cements, resin modified glass inomer cement, early shear bond strength, long term shear bond strength, zirconia copings.

Introduction

Zirconia ceramics with their ability for phase transformation and crack propagation arrest have provided enhanced mechanical properties like excellent flexural strength and high fracture toughness along with great aesthetics. These unique properties of zirconium have paved a way for extended applications and increased use of this material in clinical dentistry.[1]

One of the limitations of zirconium is the poor bonding of resin cements to zirconia due to contamination of the restorative surface with saliva, blood and silicone indicators during clinical try-in procedure. Saliva and gingival crevicular fluids contain various forms of phosphates, e.g phospholipids. Zirconium shows high affinity towards these phosphate groups and react irreversibly with the surface producing zirconium phosphate.[2,3] This coating could not be effectively removed only by cleansing with water and it interferes with the stable resin zirconia bonding resulting in reduced shear bond strength values. [3]

Recently, a new cleaning agent called Ivovlean (Ivoclar-Vivadent, schaan, Liechtenstein) has been developed to remove the contamination from zirconia after clinical try in and improve the bonding of the zirconia to the luting cements. Ivoclean has not been evaluated for zirconia-based restorations and as a replacement for currently accepted methods like cleaning withwater and air abrasion. Therefore, the commercially available cleaning agent (Ivoclean) and commonly practiced clinical method of using water were used in this study to check the cleaning efficiency of saliva contaminated zirconia copings. [4,5,6,7]

Another challenge faced is the effective bonding of zirconia and luting cements. Zirconia restorations can be luted with traditional luting cements like glass inomer cement but bonding of zirconia with resin cements can be beneficial in many ways such as providing increased retention and marginal adaptability[8] Hence, this study aimed to compare the shear bond strength of zirconia restorations bonded with two different resin cements i.e self-adhesive resin cement and resin modified glass inomer cement.

Also, there is limited information on the long-term bond strength between the luting cement and zirconia after cleaning with different agents. It is also equally important to examine the fracture mode under an optical microscope after de-bonding. It provides viable information to determine if the fracture was adhesive, cohesive or mixed.

Therefore, the aim of this study was to compare the early and long term shear bond strength and failure mode of saliva contaminated zirconia restorations that were cleaned with distilled water and Ivoclean and cemented onto extracted human teeth using two different cements- resin modified glass ionomer cement and self adhesive resin cement.

Materials and Methodology

Preparation of specimens: 60 non carious extracted maxillary premolar teeth indicated for orthodontic and periodontal extractions were collected and stored in 10% formalin. All specimens were cleaned with distilled water and with ultrasonic cleaner. Samples were mounted onto green (group A) and pink (group B) PMMA resin blocks for easy differentiation. Further, all samples were marked mesial, distal, buccal and lingual. (Figure 1)

Tooth preparation: Before tooth preparation a putty index was made for each tooth individually and sectioned into half bucco- lingually. Further, all preparations were performed by a single operator to standardize the procedure. Teeth were prepared with flat end tapered diamond burs. (Mani burs- TF13, TR13, TF13EF, TR13EF). All the specimens of group A and B were prepared with 2 mm shoulder margin and 6degree taper. Tooth preparations were evaluated using the respective silicone index of the specimens that were made prior to the preparation. (Figure 2)

Sample preparation: Sixty samples of Zirconia copings were fabricated with 2mm thickness using CAD-CAM technology. Cement space was established at 30 microns and the design blueprint was saved as STL file format. After the fabrication of zirconia copings, the fit between the coping and prepared tooth at the marginal area was checked visually for all samples. The samples with the zirconia copings were then randomly distributed among the subgroups. (Figure 3)

Sample distribution: (n=60)

GROUP A: Zirconia copings contaminated with saliva – 40 samples

Group A samples contaminated with saliva were divided into two groups depending on the cleaning agent used (20 samples each) (Figure 4)

GROUP A 1: 20 Zirconia copings contaminated with saliva and cleaned with Ivoclean for 20 seconds and rinsed with water for 15 seconds and then air dried for 15seconds. (Figure 5)

GROUP A 2: 20 Zirconia copings contaminated with saliva and cleaned with distilled water for 15 seconds and air dried for 15 seconds. (Figure 6)

GROUP B: Non contaminated Zirconia copings (control group) – 20 samples.

Both group A (A1, A2) and group B were further sub divided into 2 sub - groups.

SUB GROUP A: Zirconia copings cemented with Resin Modified Glass Ionomer cement according to manufacturers instructions and left to polymerize under constant finger pressure. (Figure 8)

SUB GROUP B: Zirconia copings cemented with Self Adhesive Resin Cement according to manufacturers instructions and left to polymerize under constant finger pressure (Figure 7). Excess cement was removed with the help of explorer.

Storage of samples: Prior to de- bonding all bonded samples were stored in artificial saliva for 24hours and 90 days. This was followed by thermocycling for 1,200 cycles between 5 to 55°C and dwell time of 30 seconds to stimulate the intra oral conditions. (Figure 9)

Testing for SBS: Samples were subjected to check for shear bond strength using universal testing machine with cross head speed of 0.5 mm/min. The specimens were mounted onto a stable fixture and subjected to shear loading using a flat shearing blade until de-bonding occurred. Force in Newtons required to debond the zirconia copings were then measured. This force was applied parallel to the area of the bonding surface to yield the bond strength in megapascals (MPa = 1 N/mm). Shear bond strength was calculated from the peak failure load applied. (Figure 10)

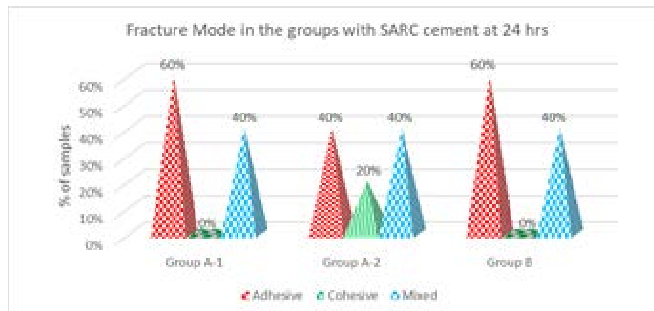
Testing for fracture mode: The fractured samples were tested under stereomicroscope to analyse mode being adhesive, cohesive or mixed. Adhesive fracture is the fracture at the interface of luting cement and zirconia surface and the fracture at the interface of luting cement and tooth surface. Whereas cohesive fracture is the fracture within the luting cement and the fracture occurring within the zirconia coping. Mixed fracture is a combination of these fracture mode. (Figure 11)

Statistical analysis

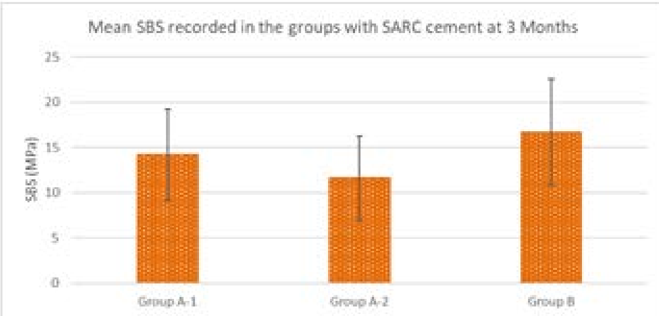
The categorical variables were presented as frequency tables with percentages and represented graphically. The quantitative data was described using Descriptive Statistics like Mean and Standard Deviation or Median and Inter-Cordial Range, whichever is applicable. For Mean, 95% confidence interval was calculated. Graphical representations were made wherever necessary.

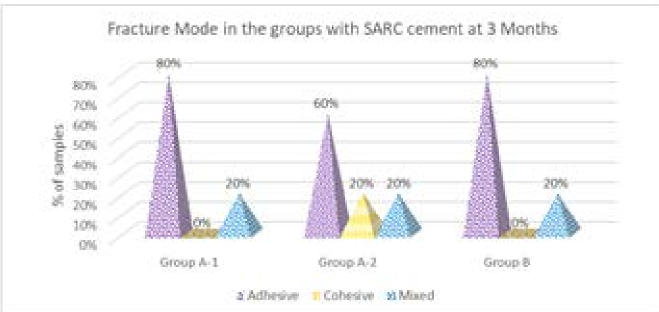
The decision criterion is to reject the null hypothesis if the p-value is less than 0.05. If there was a significant difference between the groups, multiple comparisons were calculated (post-hoc test) using Bonferroni test.

Results

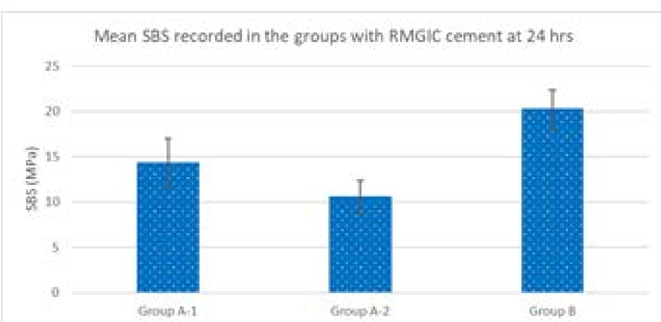


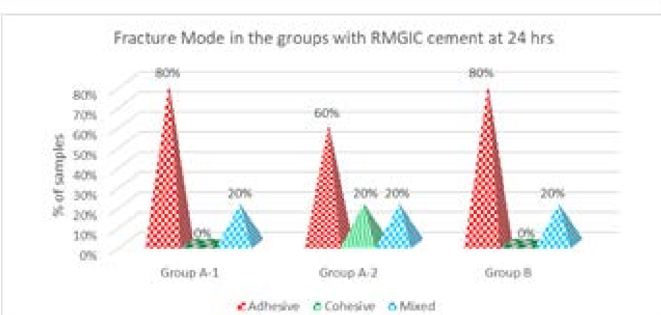
The difference in mean SBS (MPa) was found to be statistically significant between Group A-1 (Ivoclean) and Group A-2 (distilled water) (P<0.05) as well as between Group A-2 (distilled water) and Group B (control group) (P<0.01). However, the difference in mean SBS (MPa) was not statistically significant between Group A-1 (Ivoclean) and Group B (control group) (P>0.05).



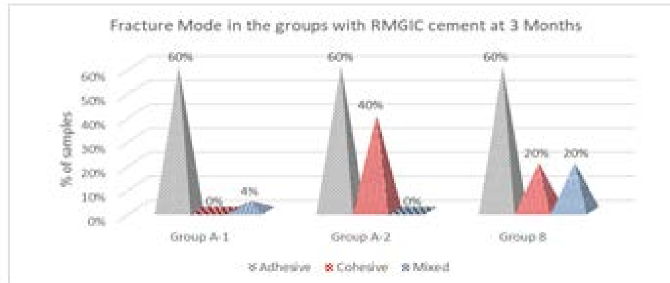


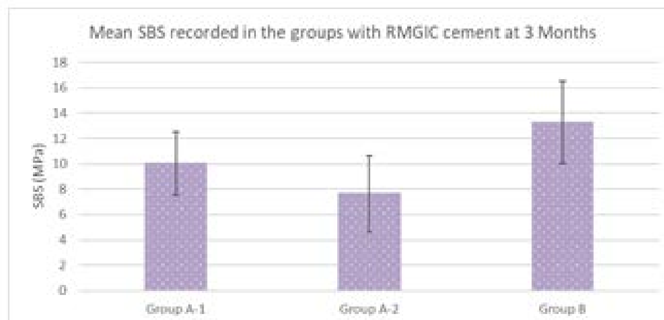
The difference in mean SBS (MPa) was found to be statistically significant between Group A-1 (Ivoclean) and Group A-2 (Distilled water) (P<0.01) as well as between Group A-2 (distilled water) and Group B (control group) (P<0.001). However, the difference in mean SBS (MPa) was not statistically significant between Group A-1 (Ivoclean) and Group B (control group) (P>0.05).





The difference in mean SBS (MPa) was found to be statistically significant between Group A-1 (Ivoclean) and Group A-2 (Distilled water) (P<0.01) as well as between Group A-2 (Distilled water) and Group B (control group) (P<0.001). However, the difference in mean SBS (MPa) was not statistically significant between Group A-1 (Ivoclean) and Group B (control group) (P>0.05).





No significant difference was observed between any pair of the groups (P>0.05).

Discussion

A successful restoration is not only defined by its strength, but mainly by the stability of the cement-restoration adhesion. Zirconia is widely applied in the dental field due to its strength, aesthetics and biocompatibility. Unfortunately, debonding of zirconia restorations has been commonly observed in clinical practice. According to the literature, most clinical failures begin at the cementation stage or after cementation as a cohesive failure within the cement. Failure rates due to high strength ceramic fractures range between 2.3%-8.0%, indicating that the integrity of the bond between the luting cement and the ceramic surface plays an important role in the longevity of the restoration.[9,10]

In the present study two widely used resin cements for luting to zirconia crowns were used i.e resin modified glass inomer cement (RMGIC) and self-adhesive resin cement (SARC).

Another common challenge is saliva contamination of the zirconia surface during the clinical try-in procedure. Saliva contamination inhibits the stable bond formation of resin cement with zirconia. The previous study by Kweon and Hakansson has demonstrated that zirconia has a strong affinity for phosphate groups present in saliva. [11,12,13]

Studies have reported different cleansing protocols, such as water, alcohol (70%-96% isopropanol), phosphoric acid (35%-37%) and additional airborne particle abrasion (Al_2O_3). Studies using X-ray photoelectron spectroscopy (XPS) showed that phosphoric acid (H_3PO_4) seems to be an effective cleansing method to remove organic contaminants from saliva and blood although it leaves phosphorous residues that could negatively impair the bonding ability. As a result, the adhesion between zirconia and resin cement was shown to decrease, consequently changing the surface energy and failure to re-establish the original bond strength value of the uncontaminated zirconia surface. [14,15,16,17,18,19]

Some authors have suggested that an additional particle abrasion may provide good bonding results after contamination. However, the use of a second particle abrasion could be controversial because of the potentially deleterious effect on zirconia phase transformation that could possibly weaken the zirconia ceramic. Also, in a clinical practice it may not be feasible to use air abrasion chair side. [15,16,19]

In the present study, group comparisons for early SBS after 24 hours showed that all groups presented higher mean SBS results in comparison to the long term SBS mean values after 3 months of artificial aging followed by thermocycling. The higher values obtained were statistically significant as well. Spontaneous debonding also occurred during thermocycling. This was significant and was particularly pronounced in the distilled water test groups, where the thermocycled zirconia samples all debonded. Interestingly, the thermocycled uncontaminated samples only had one sample that spontaneously debonded. This illustrates two things. The first was that thermocycling reduced bond strength. The second was that cleaning methods especially by distilled water in comparison to Ivoclean do not completely get rid of the contamination from the ceramic structures that caused reduced bond strength.

Prior studies reported water rinsing not to be an effective agent to remove saliva contaminants from the zirconia surface.[19] However, the present study made use of distilled water as an alternative cleansing agent. Distilled water is water that has been boiled into vapor and condensed back into liquid in a separate container with a neutral pH of 7. There is a significant difference noticed between the mean SBS of water as a cleansing agent documented in previous studies to the SBS of distilled water used as cleansing agent in the present study after saliva contamination. Due to its ease of availability and economic compatibility, it can be used as a regular practice instead of water from the tap or dental unit which might account to some impurities. However, the SBS values of distilled water as a cleaning agent were significantly lower compared to the control group and the new commercial agent, Ivoclean used in the study for both early and delayed SBS testing.

The use of a fairly new cleaning agent called Ivoclean uses the principle that zirconium has a strong affinity for phosphate groups. According to the manufacturer, due to the size and concentration of zirconium particles in Ivoclean, the phosphate contaminants are much likely to bond to Ivoclean than to the zirconium surface. Thereby

Ivoclean absorbs phosphate contaminants like a sponge leaving behind a clean zirconium surface for resin

In the present study, the Ivoclean group showed bond strength results comparable to those of the control group before and after artificial saliva storage for 3 months and TC. Even though saliva storage and TC reduced the SBS values for the Ivoclean group, the results showed that the Ivoclean and control groups maintained similar SBS values indicating optimum efficiency of cleansing action provided by Ivoclean.

Also, the data from this in vitro study showed the highest bond strength values for both early and delayed testing for self-adhesive resin cement (RelyX 200, 3M ESPE) compared to resin modified glass inomer cement (RelyX Luting 2, 3M ESPE).

Also, artificial aging and thermocycling showed a remarked reduction in the SBS values as compared to the early SBS values for both cements but significantly less difference was noted with SARC before and after aging.

The self-adhesive cement exhibited higher percentage of adhesive fracture i.e fracture between the cement and tooth interface compared to RMGIC that showed predominantly cohesive fractures within the cement and adhesive fractures at the ceramic-cement interface for initial and long term SBS testing.

These findings are in contrast to those of Palacios et al. who showed no significant difference in the retentive strengths of zirconia ceramic crowns cemented with a composite resin cement-RMGIC (RelyX Luting) and an SARC (RelyX Unicem).[20] Similarly, Capa et al. found no significant difference in the shear bond strength of composite to Y-TZP between SARC (RelyX Unicem) and RMGIC (FujiCem).[21] However, the long term shear bond strength and fracture mode has never been documented in literature before.

Self-adhesive resin cement contains methacrylate monomers, adhesive phosphate monomer and silanated fillers in its chemical composition. Adhesive phosphate monomer enhances the self-bonding to zirconia ceramics.[22] On the other hand, RMGIC contains BisGMA, HEMA and zirconia filler. Several studies previously have also reported the lower bond strengths of BisGMA containing resin cements due to their weak mechanical properties but were not compared with the self-adhesive resin cement for delayed SBS. [23]

Luting cements with high mechanical properties are more resistant to aging conditions.[24] According to the manufacturers the flexural strength for RelyX U200 (self - adhesive resin cement) is 99 MPa and that for Rely X luting 2 is 32.6 MPa. Therefore, the variations in the mechanical properties of the two luting cements used could be another contributing factor for the derived bond strength results.

The limitations of the present study were that the saliva contamination of the materials was not quantified. Quantifying the amount of contamination by X-ray photoelectron spectroscopy (XPS) would have shown if samples were uniformly contaminated. Further, Artificial saliva contains only inorganic components, such as calcium and phosphate, and does not contain human salivary proteins. However, use of human saliva in experimental studies may lead to problems in reproducibility and standardization of experiments due to human variation. Consequently, artificial saliva was used for aging process and standardization.

Conclusion

1. Salivary contamination significantly reduces shear bond strength of resin cements to zirconia.
2. In a clinical practice, a simple application of Ivoclean can be an effective alternative to water rinsing and air abrasion in removing salivary contaminants.

3. Self-adhesive resin cement showed higher SBS values for both early and delayed testing compared to resin modified GIC. Although, both resin cements used in the study showed clinically acceptable bond strength values for clinical use.
4. A significant reduction in SBS values were noted for delayed testing after artificial aging and thermocycling compared to the early SBS values for all groups.
5. However, long-term clinical studies are required to evaluate the efficacy of the cleaning solutions.

Legend Figures

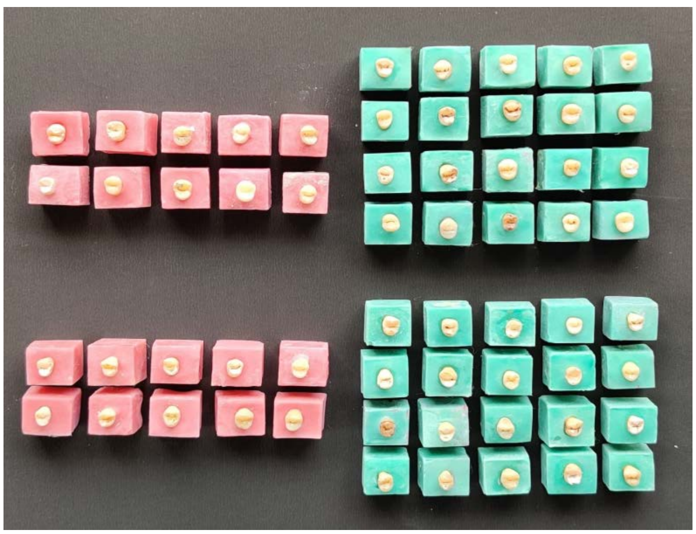


Figure 1: Teeth mounted onto PMMA resin blocks

Figure 2: Tooth preparation and Putty index

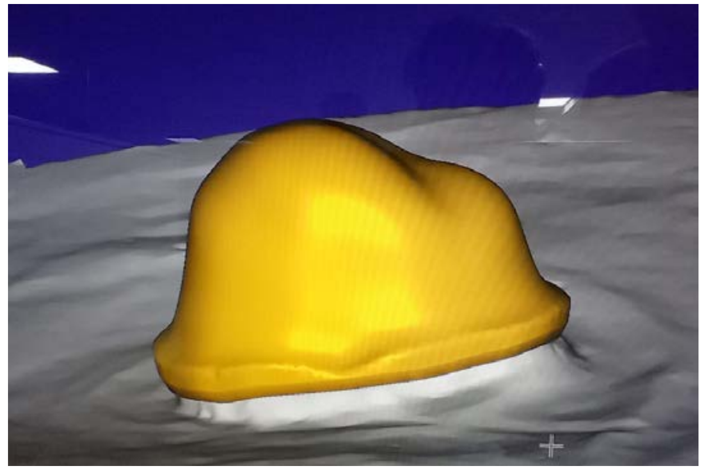


Figure 3- CAD/CAM fabrication of zirconia copings

Figure 4: Contamination of zirconia copings with human saliva

Figure 5: Contaminated copings cleaned with distilled water

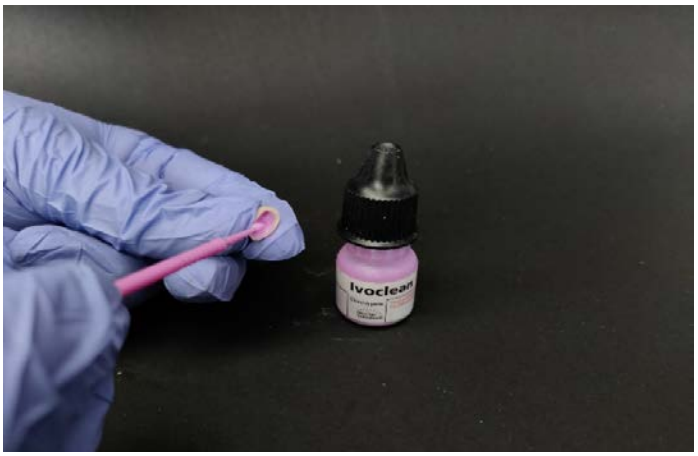


Figure 6 :Contaminated copings cleaned with Ivoclean

Figure 7: Cementation with SARC

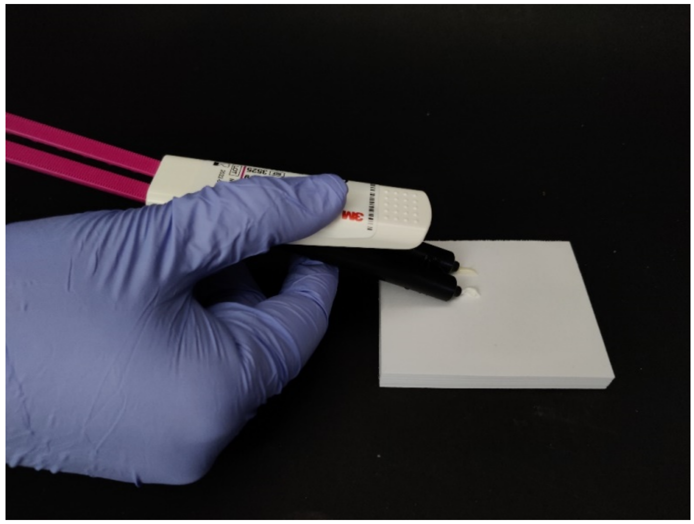


Figure 8: Cementation with RMGIC

Figure 9: Storage in artificial saliva

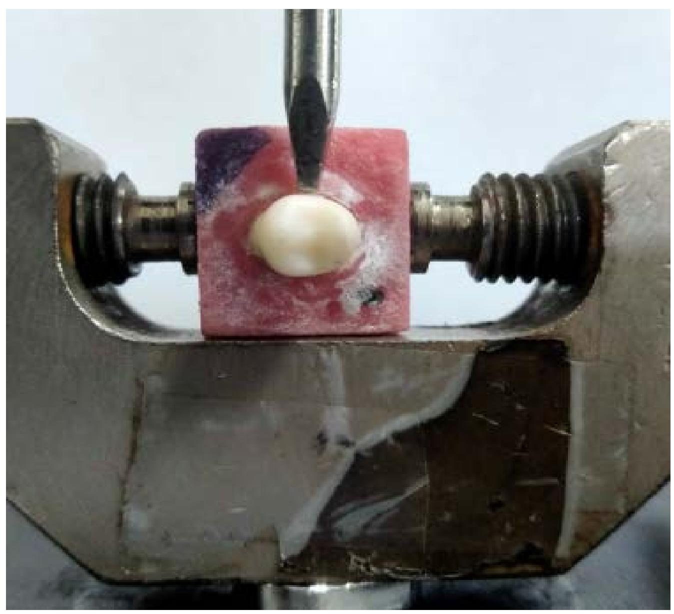


Figure 10:Testing SBS values under UTM

Figure 11: Cohesive, Adhesive, Mixed fracture mode

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