Original Article

Evaluation of the effect of silicone residue after different surface treatments on shear bond strength of glass ionomer cement to the dentin surface

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Abstract

Aim: Use of silicone fit-checking material during cementation of fixed restoration has shown to leave residual silicone film after peeling off of fit checker (FC). This residual film reduced bond strength of cement to the inner surface of restoration. Silicone residue effect on tooth surface needs to be studied. The aim of this study was to evaluate the effect of residual silicone film on shear bond strength (SBS) of glass ionomer cement (GIC) to dentin surface and the efficacy of different surface treatments (STs) on dentin in the removal of silicone residue.

Materials and Methods: Fifty freshly extracted human molars were individually mounted on acrylic blocks and occlusal surfaces were ground flat until dentine surface was exposed. Specimens were divided into five groups as follows: Group I: without application of FC ($n = 10$) as control group; Group II: without any ST after peeling off FC ($n = 10$); Group III: ST using wet pumice after peeling off FC ($n = 10$); Group IV: ST with 37% phosphoric acid after peeling off FC ($n = 10$); and Group V: ST with 10% polyacrylic acid after peeling off FC ($n = 10$). GIC was placed on the dentinal surface using polyvinyl mold and subjected to SBS test using universal testing machine. The debonded specimens were observed under stereomicroscope for the mode of failure. Selected debonded dentinal surfaces from each group were examined under scanning electron microscope. One-way analysis of variance and Tukey's test.

Results: Group II (1.083 MPa) showed significantly lower SBS. Among the STs, Group III (2.047 MPa) was comparable to the control group whereas Group IV (1.376 MPa) and Group V (1.63 MPa) were significantly lower. There was no significant association between failures and groups at $P = 0.257$.

Conclusion: The residue of silicone was demonstrable on dentin surface after peeling off FC and caused a significant reduction in SBS between GIC and dentin. ST with wet pumice is found to be beneficial in removing silicone residue and improving SBS, followed by phosphoric acid and polyacrylic acid.

Keywords: Fit checker, shear bond strength, silicone residue, surface treatment

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INTRODUCTION

The clinical outcome of dental restoration depends mainly on bonding of luting cement to the surface of tooth and crown. Markedly reduced bond strength has been documented between the restoration and luting cement due to the contamination of the fitting surface of the crown from saliva, blood, or silicone fit-indicator during the try-in procedure of a restoration.[1-3]

Fit-checking material (G-C-fit checker, GC Corporation, Tokyo, Japan) is a modified polyvinyl siloxane material that can help to improve the fit of fixed restoration to the prepared tooth surface by detecting interferences on the internal surface of the restoration.[4-7] After peeling off the fit checker (FC), a residual film is evident on the fitting surface of the restoration.[8,9] This film was found to act as a barrier and alter the bond strength of the luting cement to the inner surface of cast metal and ceramic restoration.[1,10-12]

During silicone-disclosing procedure, *in vivo*, apart from the internal surface of the restoration, FC also makes contact to the dentin surface of the prepared tooth. However, there is no literature available supporting the presence of silicone residual film on the tooth surface. Hence, it was assumed that the residue may be present on the prepared tooth surface as well.

Glass ionomer cement (GIC) is the most commonly used cement for luting crown to the prepared tooth surface as it has several clinical advantages. They include physicochemical bonding to the tooth structure, fluoride release, and low coefficient of thermal expansion.[13]

The efficiency of STs on dentin in the removal of silicone residual film from the surface of the tooth to enhance the bonding between the luting cement and dentin has not been studied so far.

Hence, the present study was formulated to evaluate the effect of residual silicone film on SBS of GIC to dentin and the efficacy of different surface treatments (STs) on dentin in the removal of silicone residue.

The null hypothesis was that the STs used in this study could not effectively remove the residual silicone film from the tooth surface and improve the SBS.

MATERIALS AND METHODS

A total of 50 (n = 50) freshly extracted molar teeth were collected and stored in 10% neutral-buffered formalin. The radicular portion of prepared tooth was embedded into the autopolymerizing acrylic resin block (25*15*15) leaving the coronal portion open. The occlusal surface of the tooth was ground flat till the dentin was exposed using a model trimmer with diamond disk, followed by manual polishing of the dentinal surface using wet 600 grit silicon carbide paper and cleaned under running water. All the specimens [Figure 1] were prepared in the same manner. Specimens were divided into five groups with 10 specimens in each group.

In Group I (n = 10) (control group), fit-checking material was not applied on the flattened dentinal surface. No ST was done. For the remaining four groups, FC (GC Corporation, Tokyo, Japan) was manipulated following manufacturer's instructions and applied on dentine surface. It was peeled off 2 min after the final set. In Group II, no ST was done. In Group III, the surface was cleaned using wet pumice and polishing brush attached to contra-angled handpiece (NSK, Japan) at low speed for 10 s. In Group IV, the surface was treated with 37% phosphoric acid (Eco-Etch, Ivoclar vivadent) for 15 s. In Group V, the surface was treated with 10% polyacrylic acid (Dentine conditioner, GC Corporation, Tokyo, Japan) for 20 s. All the surface-treated samples were rinsed with water for 5 s, and moisture was removed using tissue paper.

A prefabricated polyvinyl mold of 3.5-mm diameter and 3-mm height was positioned on the flattened dentinal surface [Figure 2]. GIC Type I (GC Corporation, Tokyo, Japan) was mixed following the manufacturer's instructions (3:1). The mixed cement was placed in the mold and held under hand pressure. The polyvinyl mold was removed 10 min after setting of the cement. The samples [Figure 3] were prepared groupwise and subjected to shear bond strength (SBS) test using universal testing machine

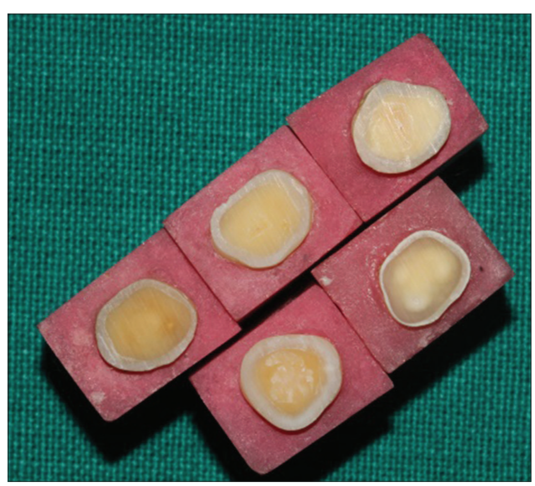


Figure 1: Specimens for the study with occlusal surface ground

(Instron 8801, Europe) until fracture. The specimen was placed in the lower assembly of the machine and force was applied onto the cement parallel to the occlusal surface of tooth at a crosshead speed of 0.5 mm/min [Figure 4].

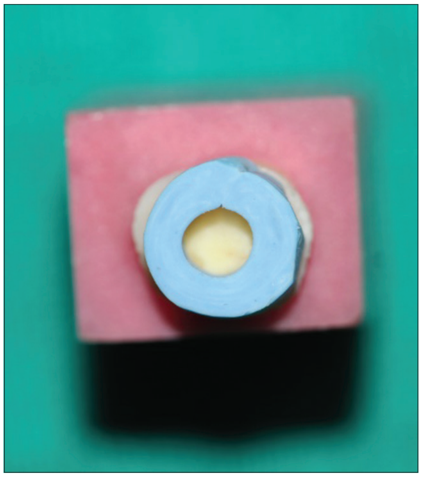


Figure 2: Polyvinyl mold positioned on the dentinal surface

Figure 3: Specimen with glass ionomer cement after the removal of polyvinyl mold

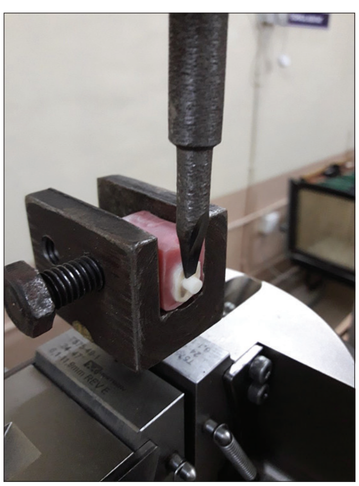


Figure 4: Shear bond strength testing using universal testing machine

The maximum force needed to debond the sample was recorded in newtons (N).

SBS was calculated using the formula:

$$\text{SBS (MPa)} = \frac{\text{Failure load (N)}}{\text{Surface area (mm}^2\text{)}}$$

Failure mode determination using stereomicroscope

Fracture analysis of all the specimens was observed under stereomicroscope (Olympus, Tokyo, Japan) at ×30. Images were captured and analyzed for the mode of failure. Failures were classified as cohesive (>75% of the failure was within the restorative material), adhesive (>75% of the failure was between tooth and restorative material), and mixed.

Scanning electron microscope evaluation

Randomly selected debonded specimens one from each group were desiccated, gold sputtered, and examined under scanning electron microscope (Zeiss Evo 18 special edition, Germany) at an acceleration voltage of 15 KV. Images were captured at ×200 and ×2000 to determine the presence or absence of silicone residue and the effect of ST on the GIC at the glass ionomer–dentin interface. The data obtained were subjected to statistical analysis using the one-way analysis of variance (ANOVA) test and Tukey's *post hoc* test. The Chi-square test was used to evaluate the association between failure modes among different groups at a statistical significance level of $P < 0.05$.

RESULTS

Descriptive statistics of the SBS (MPa) of the different groups is summarized in Tables 1 and 2. ANOVA showed statistical significant difference among the various groups ($P < 0.05$). Statistically significant reduction in SBS was found from Group I (2.599MPa) to II (1.083 MPa), IV (1.376 MPa), and V (1.63 MPa). No statistical significance was found between Groups I and III (2.047 MPa). Statistically significant reduction in SBS was found from Group III to II and IV. No statistical significance was found between Group II and IV; II and V; V and III; and V and IV. Three types of failures were recorded – cohesive failure [Figure 5], adhesive failure [Figure 6], and mixed

Table 1: Mean comparison of shear bond strength among five groups using the one-way analysis of variance test

Groups	Minimum	Maximum	Mean (MPa)	SD	*F*	*P*
Group I	1.60	3.28	2.5990	0.67621	18.137	0.000*
Group II	0.64	1.65	1.0830	0.36295		
Group III	1.56	2.68	2.0470	0.31411		
Group IV	0.85	2.01	1.3760	0.43221		
Group V	1.26	2.16	1.6300	0.30987		

*The mean difference is statistically significant at $P<0.05$.
SD: Standard deviation

Table 2: Intergroup comparison of mean shear bond strength values using the Tukey's *post hoc* test

Groups (I)	Groups (J)	Mean difference (I−J)	SE	*P*	95% CI	
					Lower bound	Upper bound
Group I	Group II	1.51600	0.19703	0.000*	0.9562	2.0758
Group I	Group III	0.55200	0.19703	0.055	−0.0078	1.1118
Group I	Group IV	1.22300	0.19703	0.000*	0.6632	1.7828
Group I	Group V	0.96900	0.19703	0.000*	0.4092	1.5288
Group II	Group III	−0.96400	0.19703	0.000*	−1.5238	−0.4042
Group II	Group IV	−0.29300	0.19703	0.576	−0.8528	0.2668
Group II	Group V	−0.54700	0.19703	0.058	−1.1068	0.0128
Group III	Group IV	0.67100	0.19703	0.012*	0.1112	1.2308
Group III	Group V	0.41700	0.19703	0.231	−0.1428	0.9768
Group IV	Group V	−0.25400	0.19703	0.699	−0.8138	0.3058

*The mean difference is statistically significant at $P<0.05$. SE: Standard error, CI: Confidence interval

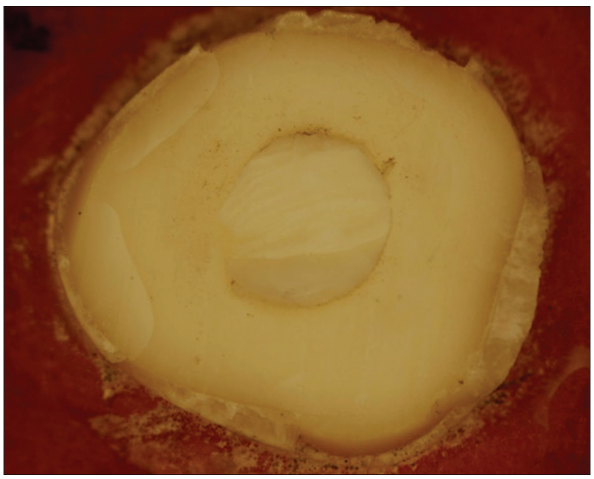


Figure 5: Cohesive failure

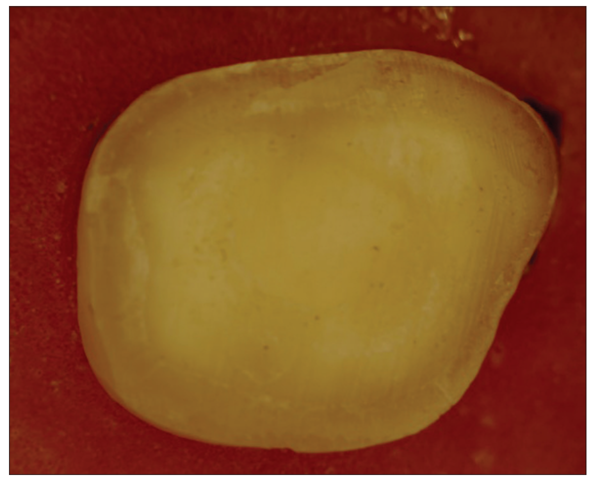


Figure 6: Adhesive failure

failure. Groups I and III showed predominantly cohesive failures, Groups II and IV showed adhesive failures, and Group V showed mixed failures [Table 3]. The Chi-square statistic (Pearson's χ^2) revealed that there was no significant association between the failures and the groups ($P = 0.257$). Figures 7-11 show representative scanning electron microscope (SEM) images of debonded interfaces after tensile bond testing.

DISCUSSION

The presence of residual silicone film on the inner surface of the restoration after the silicone-disclosing procedure has shown to alter the bond strength between the cast and ceramic restoration and the luting cement. The presence of residue of silicone may be due to chemical reactions[9] and covalent bonds[4] that may occur between silicone indicator film and restoration, leading to a stable adherence of silicone to bonding substrate, and reducing the bonding ability. Different STs have been used to remove the residual silicone and to disclose wax present on the inner surface of the crown to achieve optimal bonding conditions.[11,12,14,15] Similarly, there is a possibility of leaving a residual silicone film on the surface of the tooth also.

Conventional GIC Type I cement is the most popularly used material for luting of cast restorations on vital dentin. The bond formation between the carboxyl groups of polyacrylic acid and hydroxyapatite on the tooth surface makes it more adhesive. Efficiency of cement to bond depends on the availability of hydroxyapatite molecules on the tooth surface.[13] Any barrier between the calcium of dentine to the carboxylic group compounds would interfere in the formation of bond and thus reduce the retention of cemented restoration.

The mean SBS value obtained for Group I (control group) was 2.599 MPa [Graph 1] which was within the typical range shown in the previous studies.[16] Peeling off FC resulted in 58% loss of mean SBS in Group II (1.083MPa) compared to Group I. Similar kind of results were shown for the retention of the luting cement to the cast metal surface as well as ceramic restoration.[8,11] This loss of retention is suggestive of failure of cement to form a bond with hydroxyapatite of dentin due to the presence of silicone residue.

The drop in SBS emphasizes the need for some kind of ST to remove the silicone residue. In the present study, some of the most commonly used agents such as wet pumice, 37% phosphoric acid, and 10% polyacrylic acid were considered since they have shown to be effective in removing the smear layer containing both organic and inorganic contents. All the STs showed an improvement in SBS compared to Group II. The mean SBS value obtained for Group III (2.047 MPa) was 21.2% less compared to the control group indicating the incomplete removal of silicone film by pumice. Pumice, being an abrasive agent, was able to remove most of the smear layer from the dentinal surface.[17-20] Similarly, pumice was able to remove most of the silicone film from dentinal surface. ST with pumice improved the SBS by 47% compared to Group II. This could be due to the removal of most of the silicone films and facilitating the cement to react with hydroxyapatite of dentin.

Table 3: Comparison of failure modes using the Chi-square test

Groups	Failure mode		Total
	Adhesive	Cohesive	
Group I	3	7	10
Group II	7	3	10
Group III	3	7	10
Group IV	6	4	10
Group V	5	5	10
Total	24	26	50

The difference is statistically significant at $P<0.05$

The mean SBS value obtained for Group IV was 1.376MPa. Statistically significant difference was obtained between Group I and Group IV ($P = 0.000$) with a 47% decline in SBS in Group IV. This is in accordance with the previous studies that have shown reduced SBS of GIC to dentin surface treated with phosphoric acid in the removal of smear layer.[13,16] Compared to Group II, SBS of Group IV improved by 21%; however, statistically significant difference was not obtained ($P = 0.576$). This is suggestive of the limited efficacy of phosphoric acid in removing the silicone film.

There was a decrease in SBS of Group V compared to Group I by 37%. The difference was statistically significant

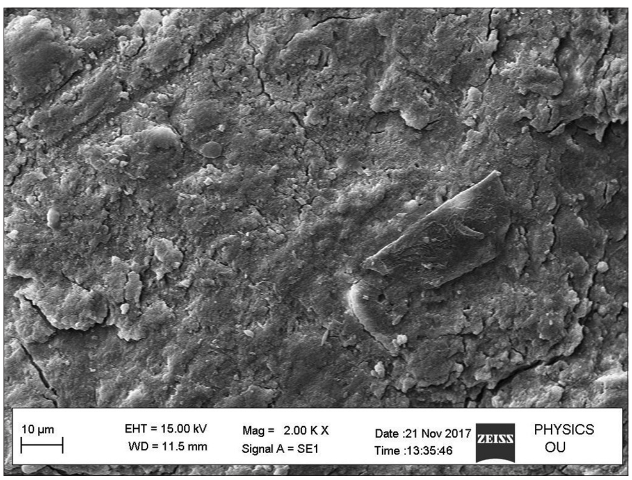


Figure 7: Scanning electron microscope of Group I at ×2000

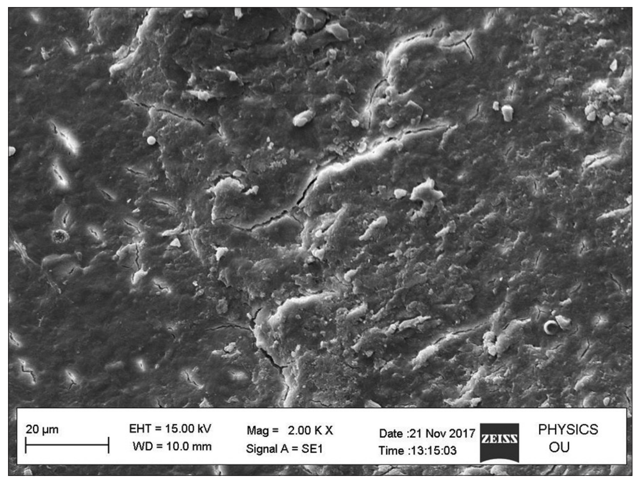


Figure 8: Scanning electron microscope of Group II at ×2000

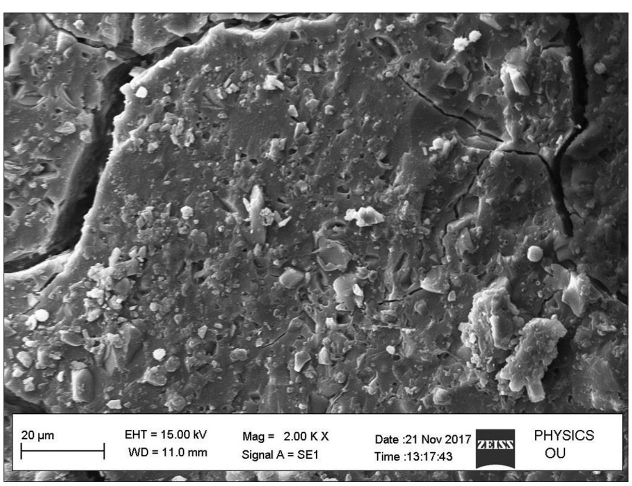


Figure 9: Scanning electron microscope of Group III at ×2000

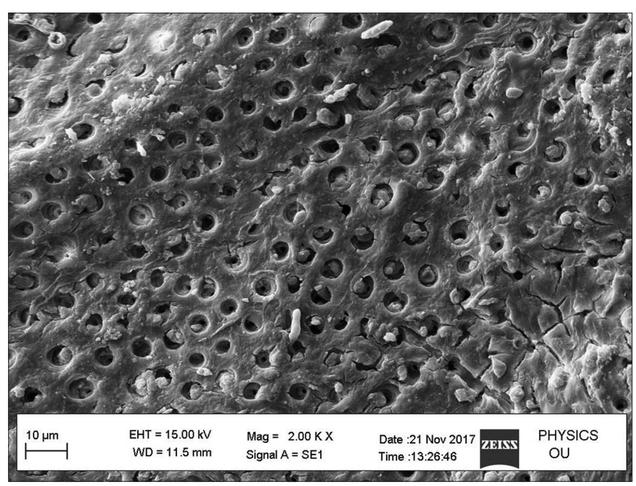


Figure 10: Scanning electron microscope of Group IV at ×2000

(P = 0.000) indicating that the efficacy of polyacrylic acid in the removal of silicone residual film from the dentinal surface was limited. SBS in Group V was higher compared to Group II by 33.6%, although statistically not significant (P = 0.058).

ST with Group V showed higher SBS compared to Group IV by 15%. The difference was statistically not significant (P = 0.699). The polyacrylic acid pretreatment is much milder than the phosphoric acid treatment. Polyacrylic acid partially demineralizes dentin, leaving hydroxyapatite around exposed collagen fibrils.[13,16] This condition is favorable for ion exchange where carboxylic groups from GIC interact with calcium and phosphate ions from hydroxyapatite crystals of dentin. The phosphoric acid treatment demineralizes superficial dentin to a variable thickness (depending on the time of application) and removes hydroxyapatite as well.[13,16] Lack of availability of hydroxyapatite crystals for bonding with GIC prevents the chemical interaction, thus reducing the bond strength. Results of the present study are in agreement with earlier studies where ST with 10% polyacrylic acid to remove smear layer has shown to increase SBS value of GIC to dentin than 37% phosphoric acid.[13,16-18]

Group III showed higher SBS compared to Group IV by 32.77%. The difference was statistically significant (P = 0.012). This is indicative of improved ability of pumice in removing the silicone residue than 37% phosphoric acid. SBS in Group III was higher compared to Group V by 20.37%. This suggests that pumice was more efficient in the removal of silicone film from the dentin surface compared to polyacrylic acid.

Stereomicroscopic analysis of the debonded dentin specimens revealed predominantly cohesive failures in Groups I and III, adhesive failures in Groups II and IV, and mixed failures in Group V [Graph 2]. Cohesive failure in Groups I and III indicates that the adhesion between the restorative material and tooth is higher than the tensile strength of the cement itself. The adhesive failures in Group II were indicative of the presence of residual silicone film resulting in reduced bond strength.[11] Adhesive failures in Group IV seemed to be due to its etching action by the removal of hydroxyapatite.

To further explore the effect of STs on the removal of residual silicone film from the dentinal surface, SEM analysis was performed. At ×2000, SEM evaluation of Group I revealed the presence of dense smear layer with few visible dentinal tubules. The prepared tooth surface was not subjected to any ST in Group I; hence,

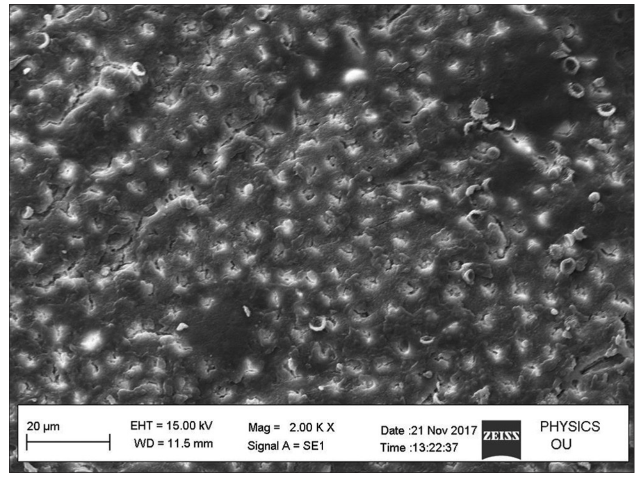
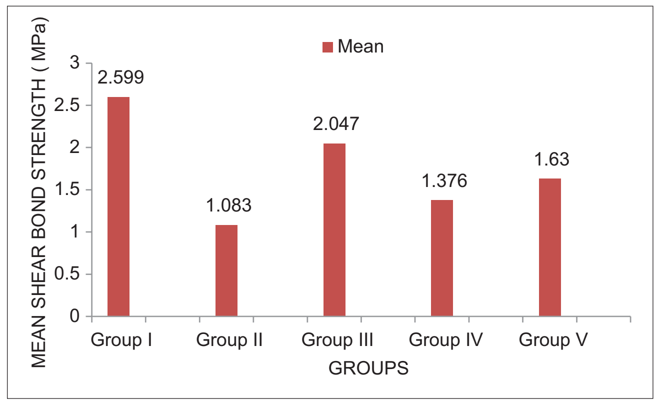
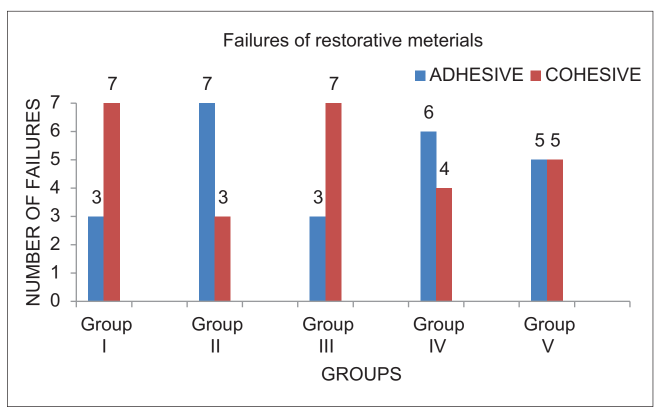


Figure 11: Scanning electron microscope of Group V at ×2000



Graph 1: Comparison of mean shear bond strength values between different surface-treated Groups I, II, III, IV, and V (I = control, II = fit checker, III = fit checker + pumice slurry, IV = fit checker + 37% phosphoric acid, and V = fit checker + 10% polyacrylic acid)



Graph 2: Types of failures in different surface-treated Groups I, II, III, IV, and V (I = control, II = fit checker, III = fit checker + pumice slurry, IV = fit checker + 37% phosphoric acid, and V = fit checker + 10% polyacrylic acid)

a layer of inorganic debris (smear layer) prevailed on the surface of the tooth.[13,17,18,21-23] Group II revealed the presence of residual silicone film interspersed with smear layer. Hence, the reduced SBS[24,25] indicating adhesive type of failure. Group III revealed cracking and the presence of GIC remnants on the dentin surface correlating with the cohesive type of failure. Group IV revealed poorly attached smear layer along with silicone layer over the intertubular dentin. Demineralizing action of phosphoric acid resulted in decreased availability of intertubular dentin for efficient bonding of GIC. Hence, adhesive type of failure was seen in Group IV. Group V revealed numerous dentinal tubules with their orifices closed and sparsely dispersed GIC remnants suggestive of mixed type of failure and incomplete removal of silicone residue.

Considering the amount of loss of SBS after silicone-disclosing procedure shows the evidence of silicone residue and its interference to form a bond at the tooth-cement interface. The null hypothesis was rejected as all the STs could partially remove the silicone residue.

Limitations of the study

Using X-ray photoelectron spectroscopy of dentin surface after various STs could reveal the effectivity of ST in removing silicone layer completely.

CONCLUSION

1. Residual silicone film significantly reduced SBS between GIC and dentin surface
2. Mechanical ST using wet pumice has shown higher SBS, followed by 10% polyacrylic acid and 37% phosphoric acid
3. Cleaning surface using wet pumice has shown comparable SBS to the control group indicating considerable removal of silicone residue
4. Chemical ST using 10% polyacrylic acid and 37% phosphoric acid were not efficient to remove silicone residue from the dentin surface.

Clinical significance

Whenever silicone-disclosing procedure is used for checking the fit of the indirect restorations, not only the inner surface of the restoration needs to be surface treated but also cleaning the dentin surface with wet pumice before cementation of the prosthesis will aid in improving the retention of the prosthesis by enhancing the bond strength.

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Conflicts of interest

There are no conflicts of interest.

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