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The effect of Fit-checking material and various subsequent cleaning methods on the wettability of the dentin surface: an *in vitro* study

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ABSTRACT

Background: GC Fit-checker is a modified polyvinyl siloxane impression material exclusively used to check the internal fit and improve the marginal fit of indirect restorations. An unpolymerized organic film is known to be leftover on the bonding surfaces after the silicone disclosing procedure. Residual silicone film being hydrophobic may alter the wettability of the cement to the tooth/ metal surface, thus having a detrimental effect on the bond strength and retention of the restoration.

Aim: This study aimed to evaluate the wetting of luting liquid (GIC) to tooth dentin surface after application of Fit-checker and evaluate the efficiency of various surface treatments in removing the residual silicone film.

Materials and methods: Extracted human molars were mounted on the acrylic block, and the tooth occlusal surface was ground flat till the dentin exposure. All the specimens were assigned into five groups: Group 1: without application of Fit-checker (control group); Group 2: without any surface treatment after peeling off Fit-checker; Group 3: surface treatment with wet pumice; Group 4: 37% phosphoric acid treatment; Group 5: 10% polyacrylic acid treatment. Later, Type 1 Glass Ionomer Cement (GIC) liquid drop was placed on the dentin and photographs were made horizontally using a standardized procedure. Contact angles were measured using AUTOCAD software. Obtained values were statistically analyzed using the One-way ANOVA test and Tukey's Post hoc test. Samples of each group were examined using the scanning electron microscope.

Results: Statistically significant difference was observed among all the groups except between Group 4 and Group 2 ($p > 0.05$). SEM images of various groups showed a significant difference in roughness patterns.

Conclusion: Surface treatment with pumice and the rotary brush was an effective method among the three in cleaning the residual silicone film.

1. Introduction

The fit of indirect restoration and maintenance of optimum cement space (25µm) can be affected by various irregularities/interferences on the internal surface of the casting [1,2]. These interferences can be identified using Fit-checking material and eliminated by the selective grinding procedure [3]. Fit-checking material (GC Fit-checker) is a modified polyvinyl siloxane material exclusively used to

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check the internal fit and improve the marginal fit of the restorations [4]. The silicone disclosing procedure using Fit-checker leaves a thin layer of residual unpolymerized organic film on bonding surfaces [5]. Residual silicone film being hydrophobic alters the wettability of the cement to the metal surface [5].

During the luting procedure, the GIC cement flows (wetting) between the irregularities of both tooth and the internal surface of the restoration and results in micromechanical bonding between restoration and luting cement interface and chemico-mechanical bonding between tooth and luting cement interface. This adhesion results in enhanced bond strength between the cement to the restoration and the tooth, respectively [2].

The contact angle is an indicator of the flow of liquid on a solid surface. A lower contact angle indicates better wetting of the tooth/restoration surfaces by the luting agent that improves the adaptation. Thus, it enhances the bond strength and retention of the indirect restorations [2]. After peeling off the Fit-checker, a thin layer of residual silicone film will be leftover on the restoration's internal surfaces. It acts as a barrier and prevents bonding between restoration-cement interface. Numerous researches reported that there would be a decrease in retention efficacy and tensile bond strength after Fit-checker application [6-8]. Studies have shown that the residual silicone film on the metal surface can be treated by various mechanical and chemical means. These surface treatments remove the silicone film and increase the tensile bond strength [6-8]. During the silicone disclosing procedure, apart from the restoration's internal surface, Fit-checker also makes contact with the prepared tooth. However, the literature does not provide any evidence that Fit-checker residue on the tooth. Hence, it was hypothesized that Fit-checker tends to leave a residue on the tooth also.

The present in-vitro study was conducted to measure and compare the contact angle of GIC liquid on dentin surfaces treated with the application of Fit-checker and various surface treatments to eliminate the residual silicone layer of Fit-checker. The study's null hypothesis was that all the surface treatments used in this study could not effectively remove the residual film.

2. Materials and methods

A total of 75 extracted molar teeth with adequate occlusal table width and without dental caries were collected and stored in 10% neutral buffered formalin.

2.1 Sample preparation

Each tooth was mounted into an acrylic block to support and position the occlusal surface parallel to the floor. The occlusal surface of the tooth was ground with a diamond disk to remove the enamel and expose the dentin. Dentin surface was considered for the study as most tooth preparations for crown fabrication clinically exposes the tooth dentin surface. Care was taken to make the prepared occlusal surface flat and parallel to the platform. This was verified using a bubble gauge placed over the flat occlusal dentin surface. All the specimens (Figure 1) were prepared similarly. Randomization of samples was done. Specimens were divided into five groups.

Group 1: Without the application of Fit-checker (n=15) (Control group).

All the remaining specimens were applied with a layer Fit-checker (GC Corporation, Tokyo, Japan), and it was peeled off after two minutes of setting. Thus, prepared specimens were randomly divided into four groups.

Group 2: No surface treatment after peeling off Fit-checker (n=15)

Group 3: Surface treatment using Wet Pumice and polishing brush for 10 sec (n=15)

Group 4: Surface treatment using 37% Phosphoric acid for 15 sec (n=15)

Group 5: Surface treatment using 10% Polyacrylic acid for 20 sec (n=15).

2.2 Surface treatments

In group 3, the Surface was treated with pumice slurry (Pumice powder, Vishal Dentocare Pvt. Ltd, Ahmedabad, Gujarat, India) and polishing brush running at low speed for 10 seconds. In group 4, a standard etchant gel of 37% Phosphoric acid (Acid Etching gel, Nimai Dento, Muzaffarnagar, India) was applied on the dentin surface and washed off after 15 seconds. In group 5, three drops of 10% Polyacrylic acid (Dentin conditioner, GC Corporation, Tokyo, Japan) was applied to the whole surface and washed after 20 seconds. All the surface treated samples were subjected to water wash using a two-way syringe for 5 seconds after their respective treatment and dried with blotting paper.

An acrylic device was custom fabricated to standardize the distance of the liquid drop. This device was connected to the analyzing rod of the surveyor. The

acrylic block has a hole in its center with a diameter equal to the GIC type I liquid bottle. A 21-gauge needle was attached to the GIC liquid bottle's nozzle to reduce the liquid drop size. The GIC liquid bottle was inverted and placed in the central hole of the acrylic resin block and was held parallel to the analyzing rod of the surveyor. This entire assembly resulted in providing the dentin surface perpendicular and 10mm away to the position of the liquid bottle. The bottle was gently squeezed to release a drop of liquid in the center of the specimen (Figure 2). Only luting GIC liquid was used in the study as it was technique sensitive to handle the mixed cement to dispense the drop.

2.3 Measurement of contact angle:

A horizontal view of the specimens with the liquid drop was captured using a DSLR camera (Nikon D 3200) and a 90mm macro lens (NIKON Corp., Japan) (Figure 3). The camera was held at the level of the liquid drop and tooth occlusal interface using a tripod. The distance between the specimen and the lens was standardized to 10 cm. The captured images were saved as JPEG images. The images were opened in AUTOCAD software (2010 Autodesk, Inc., Nasdaq: ADSK). A line was digitally drawn on the image along the base of the liquid drop on the tooth occlusal interface extending up to the three-phase (liquid, solid, air) boundary. One more line was digitally traced on the image along the outer surface of the liquid drop extending up to the three-phase boundary. The contact angle was measured at the intersection of the two lines in all the groups (Figure 4-8). A flat dentin surface without irregularities was considered to measure the contact angle as it is difficult to measure the contact angle on a regular clinical tooth preparation design. The obtained data were statistically analyzed using the Statistical Package for Social Sciences 21.0, USA. ANOVA test and Tukey HSD were used to compare the significant difference in mean contact angle values between the groups. The correlation was considered significant at $P<0.05$.

2.4 Scanning Electron Microscopic (SEM) analysis

One sample from each group was randomly chosen for SEM analysis. The samples were subjected to gold sputtering and observed under Scanning Electron Microscope (S 3700N, Hitachi, USA). Images were captured at 200X and 2000X magnification (Figures 9-18).

3. Results

The mean contact angles of all the groups are given in table 1. Group 2 exhibited the maximum mean contact angle followed by group 4, 5 and 3. Group 1 showed less contact angles. The specimens with no application of Fit-checker showed the least contact angles. The specimens treated with Fit-checker and no subsequent surface treatments resulted in increased contact angle. The samples treated with various agents decreased the contact angle compared to the specimens that did not treat after the removing the Fit-checker. However, the specimens with no Fit-checker application displayed the least contact angles compared to the surface-treated specimen groups. One-way ANOVA showed a statistically significant difference ($p=0.000$) among the groups (Table 1). Intergroup comparison using Tukey HSD showed a statistically significant difference ($p=0.000$) between all the groups except between Group 2 and Group 4 ($p=0.639$) (Table 2).

SEM images of specimens before (Group1) and after the Fit-checker application (Group 2) showed a marked difference in roughness patterns. SEM evaluation of Group 3 exhibited less roughness pattern compared to Group 1, but it is more compared to Group 2. SEM evaluation of Group 4 showed no appreciable difference in the roughness of dentin surface compared to Group 1. SEM evaluation of the samples of Group 5 showed reduced roughness of dentin compared to Group 2 and increased roughness compared to Group 1.

4. Discussion

Fit-checking material (GC Fit-checker) is a modified polyvinyl siloxane material exclusively used for checking the internal fit of the indirect restorations. Silicone disclosing material is popularly used to identify and correct the seating interferences as it significantly reduce the marginal opening from 387µm to 97µm and provided 70% improvement in marginal seating [1,6,9]. Verification of the internal fit of indirect restorations using Fit-checker application includes coating the silicon material to the internal surface of the restoration followed by seating on dies or intraorally on the abutment teeth [1]. Once the Fit-checker sets, the restoration's internal surface should be inspected for exposed areas of the restoration. These areas can be subjected to selective grinding to eliminate interferences [1,4]. Assessment of cement space can also be done by measuring the thickness of set silicone film.

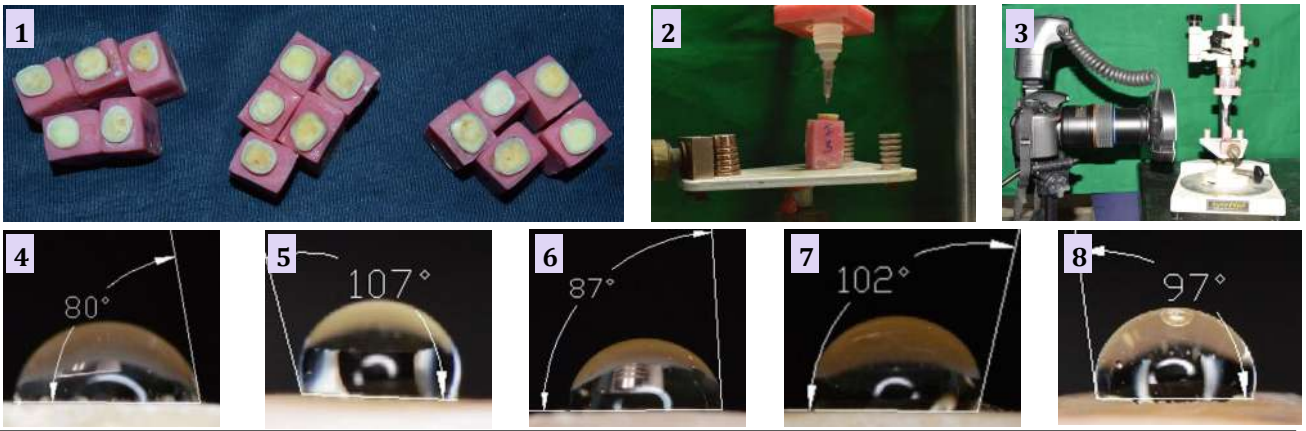
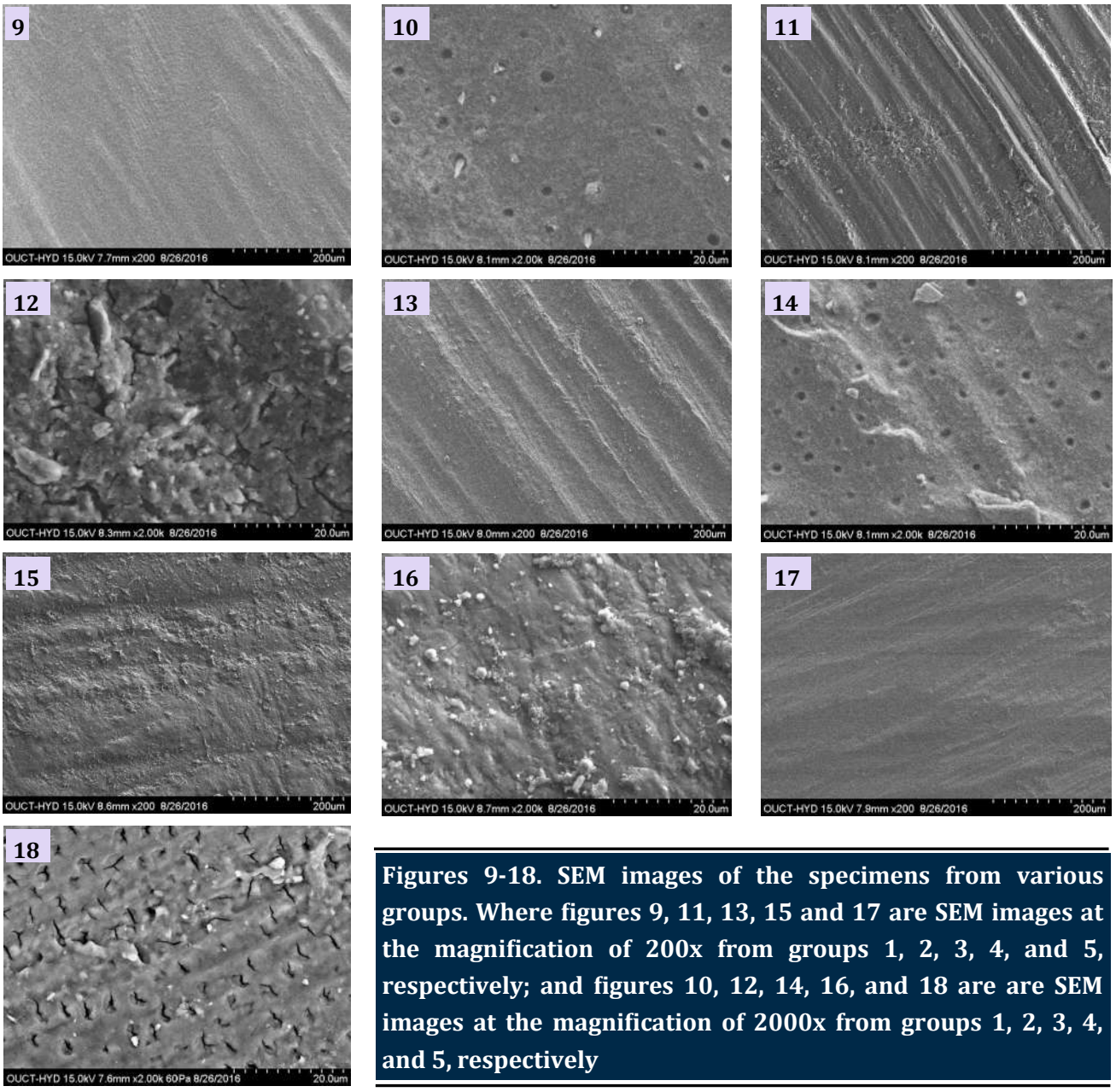


Figure1. Natural teeth embedded in acrylic blocks.
Figure 2. Assembly for placing a liquid drop on specimen from 10mm height.
Figure 3. Position of camera from 10 cm from specimen for making photographs of drop.
Figures 4-8. Contact angle measured using AUTOCAD software. Where figures 4, 5, 6, 7 and 8 are contact angles measured in group 1, 2, 3, 4, and 5, respectively.



Figures 9-18. SEM images of the specimens from various groups. Where figures 9, 11, 13, 15 and 17 are SEM images at the magnification of 200x from groups 1, 2, 3, 4, and 5, respectively; and figures 10, 12, 14, 16, and 18 are are SEM images at the magnification of 2000x from groups 1, 2, 3, 4, and 5, respectively

Table 1. Comparison of contact angle (mean and standard deviation) of the groups using One-Way ANOVA.

Groups	Mean	Standard Deviation	F-value	Significance (p – value)
Group 1	82.1667	2.66369		
Group 2	107.3667	4.09035		
Group 3	90.5667	4.83982	98.525	0.000
Group 4	105.3000	3.98121		
Group 5	98.1333	4.51374		

Table 2. Inter-group comparison of contact angles of the groups using Tukey HSD

Groups		Mean Difference	Standard Error	Significance
Group 1	Group 2	25.200*	1.492	0.000
	Group 3	8.400*	1.492	0.000
	Group 4	23.133*	1.492	0.000
	Group 5	15.966*	1.492	0.000
Group 2	Group 3	16.800*	1.492	0.000
	Group 4	2.066	1.492	0.639
	Group 5	9.233*	1.492	0.000
Group 3	Group 4	14.733*	1.492	0.000
	Group 5	7.566*	1.492	0.000
Group 4	Group 5	7.166*	1.492	0.000

Fit-Checker (GC Co., Tokyo, Japan) is a chemically stable silicone. Hence, it is claimed by the manufacturer that no residue will be left on the internal surface of the restoration. However, X-ray photoelectric spectroscopy (XPS) observation on Zirconia specimens after using Fit-checker has revealed the presence of Si, C, and O2 on the surface [5]. This unpolymerized organic film has shown to interfere in the bonding of cement to the internal surface of restoration [10-12]. Some investigators presumed that chemical reactions and covalent bonds might occur between silicone indicator films and restorations, leading to a stable adherence of silicone to bonding substrate and alter the surface characteristics of metal upon removal [5].

The mode of adhesion of glass ionomer cement (GIC) to the tooth surface is chemico-mechanical. The wettability of the liquid cement to the tooth/ restoration surfaces enhances the flow of the cement into the irregularities on both surfaces and ensures adhesion. The residual silicone film being hydrophobic found to alter the wettability of the cement to the metal surface [2]. A lower contact angle is desired for luting agents to ensure uniform flow and better bonding. Many techniques have been tried to clean the metal and ceramic surfaces contaminated with the silicone residues [7,8,12-14].

Various authors had found a considerable reduction of retention of 10.02 MPa to 4.85 MPa when the restorations were treated with disclosing agents like wax and silicone [7,12]. Methods used to clean ceramic surface post-Fit-checker removal have significantly influenced the resin-ceramic bond strength. The most effective method was air abrasion compared to cleaning procedures using steam cleaning, phosphoric acid and isopropanol [7,12].

Numerous studies have discussed cleaning procedures for silicon residue on metal and ceramic surfaces [4-12]. Since there is no evidence of residue on the tooth surface in the literature, the present study was conducted

on dentin surface to find out the contact angle of GIC cement liquid prior to Fit-checker application and after the application.

In the present study, a significant increase in the mean contact angle was observed in Group 2 compared to Group 1 (Table 2). This can be attributed to the residual silicone film, which was leftover on the dentin surface by the Fit-checker. This is in accordance with the previous studies, which have demonstrated an increase in contact angle, and the residue was also leftover on the restorations [4,5,7,10,14]. SEM images of specimens before (Figures 9,10) and after the Fit-checker application (Figures 11,12) showed a marked difference in roughness patterns. This indicates that Fit-checker has left a residual film on the dentin surface.

In the literature, various agents have been used to remove the smear layer from the dentin surface to enhance the chemical-mechanical bonding between adhesive luting cement and the dentinal matrix. In the present study, some of the most commonly used agents like wet pumice, 37% phosphoric acid, 10% polyacrylic acid were used. These agents have proven their effectiveness in removing the smear layer containing both organic and inorganic contents [15]. Hence, these acids were used in the present study to evaluate their capability of removing the Fit-checker residue from the tooth surface.

The dentin surface treated with wet pumice (Group 3) showed a statistically significant (p=0.000) decrease in mean contact angle (Table 2) compared to treating with the other agents. The pumice treatment improved the wettability denoting the removal of silicone contaminants. However, compared to the control group, the mean contact angle was significantly increased, proving the limitation of the cleaning procedure in eliminating the contaminant. SEM evaluation of Group 3 (Figures 13,14) suggested less roughness pattern compared to Group 1 (Figures 9,10). But it is more compared to Group 2 (Figures11,12). This indicates that the surface treatment using wet pumice and brush for 10 seconds might have mechanically abraded and removed silicone film to a greater extent; however, the silicon residue may not have completely eliminated.

No statistically significant increase in the mean contact angle (p=0.639) was observed in Group 4 compared to Group 2 (Table 2). SEM evaluation of Group 4 (Figures 15,16) showed no appreciable difference in dentin surface roughness. This indicates that cleaning with 37% phosphoric acid did not effectively remove residual silicone film. Although phosphoric acid effectively cleaned the smear layer [16-24], the silicone residue was not completely removed from the restoration surfaces.

In the present study, a statistically significant decrease in the mean contact angle was observed in Group 5 compared to Group 2 (Table 2). This indicates that 10% polyacrylic acid (GC Dentin conditioner) is partially effective in cleaning the silicone residue on the specimens. A statistically significant increase in the mean contact angle (p=0.000) was observed in Group 5, compared to Group 1 (Table 2). SEM evaluation of the samples of Group 5 (Figures 17,18) showed reduced roughness of dentin compared to Group 2 (Figures 11,12) and increased roughness compared to Group 1 (Figures 9,10). Although polyacrylic acid can effectively clean the smear layer [18-22,25-28], the silicone residue was not removed completely from the restoration surfaces.

In this present study, a statistically significant decrease in the mean contact angle (p=0.000) was observed in Group 3, compared to Group 5 (Table 2). This was also supported by SEM (Figures 13,14 & 17,18). It can be observed that mechanical cleaning with pumice and brush was more effective than chemical cleaning with 10% polyacrylic acid. Polyacrylic acid seems to be more effective with the removal of the smear layer than silicone residue. In the present study, 10% polyacrylic acid (GC dentin conditioner) was used and possibly the low concentrations can be attributed to the low effectiveness.

Statistically significant decrease in mean contact angle (p=0.001) in Group 4 compared to Group 5 (Table 2) shows that chemical cleaning with 10% polyacrylic acid for 20 seconds was more effective than 37% phosphoric acid for 15 seconds. However, in the literature, 37% phosphoric acid was more effective in removing the smear layer [21,22]. Even though phosphoric acid and polyacrylic acids were effective in removing the smear layer, which is a combination of inorganic and organic matter, they were not effective enough to remove the residual silicone film organic in nature. In the present study, 10% Polyacrylic acid removed the silicone residue to a lesser extent, whereas 37% phosphoric acid was not effective in removing the residue. This may be due to the nature and strongness

of acid and the time for which it was applied. The null hypothesis of the study was partially accepted.

Clinical significance: Whenever silicone disclosing procedure is used for checking the fit of the indirect restorations, wet pumice cleaning with a polishing brush can be used as a cleaning procedure on the dentin prior to cementation of the prosthesis to enhance the retention of the prosthesis.

Though SEM evaluation was considered an effective adjunct, quantification of surface roughness could have brought more insight. Further studies can be done to evaluate the effect of other cleaning agents in removing the residual silicone film and also to evaluate the effect of residual silicone film on the shear bond strengths between luting cement and the dentin surface.

5. Conclusion

From this study, the following conclusions can be drawn;

a. The residue of the Fit-checker significantly reduced the wettability of the tooth dentin surface to GIC type 1 liquid.
b. Among the three surface treatments, mechanical surface treatment using wet pumice and the rotary brush was more effective in removing residual silicone film followed by polyacrylic acid (10%). Phosphoric acid (37%) was the least effective.

Conflicts of interest: The declared no conflicts of interests.

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