

SEM EVALUATION OF SURFACE PRETREATMENT PROTOCOLS ON ADHESIVE PENETRATION AND BOND STRENGTH IN SANDWICH RESTORATIONS: AN IN VITRO STUDY

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Abstract

Aim: To compare the effect of three surface pretreatment protocols on adhesive penetration and bond strength in sandwich restorations.

Materials and Methods: Thirty extracted human maxillary premolars with standardized Class I cavity preparations were restored using RMGIC and randomly divided into three groups (n = 10) based on the surface pretreatment protocol: Group I – no surface pretreatment (control), Group II – 37% phosphoric acid etching for 15 seconds, and Group III – air abrasion with 50 μ m aluminum oxide particles. Rhodamine B dye-labeled adhesive resin was applied over the treated RMGIC surfaces, followed by composite resin restoration. Each group was further subdivided into two subgroups for SEM evaluation of adhesive penetration depth and shear bond strength analysis. Samples for bond strength testing were subjected to thermocycling and artificial saliva aging before testing. Statistical analysis was performed using one-way ANOVA and Bonferroni post hoc test with significance set at $p < 0.05$.

Results: Air abrasion demonstrated the highest mean adhesive penetration depth and bond strength values among all groups, followed by phosphoric acid etching and the control group. Statistically significant differences were observed between the groups ($p < 0.05$). A positive correlation was observed between adhesive penetration depth and bond strength.

Conclusion: Surface pretreatment of RMGIC significantly enhances adhesive resin penetration and bond strength at the RMGIC–composite interface. Among the tested protocols, air abrasion produced superior interfacial adaptation and bonding performance compared with phosphoric acid etching and no surface pretreatment.

Clinical Significance: Appropriate surface pretreatment of RMGIC can improve the interfacial integrity and durability of sandwich restorations. Air abrasion may be considered an effective pretreatment method to enhance adhesive penetration and bonding between RMGIC and composite resin.

Key words : Adhesive penetration; Air abrasion; Bond strength; Composite resin; Resin-modified glass ionomer cement; Sandwich technique; Scanning electron microscopy; Surface pretreatment

Introduction

Restorative dental development has been fueled by the continuous efforts of finding materials that can emulate the biological and functional characteristics of teeth. Ideally, a good restorative material must be able to form a stable adhesion with the teeth's enamel and dentine, have sufficient mechanical strength, resist degradation in the oral cavity, and be stable for an extended period of time. One of the revolutionary restorative materials is Glass Ionomer Cement (GIC), discovered by Wilson and Kent; GIC is known for its strong chemical bonding with teeth and excellent biocompatibility¹.

Even though conventional GICs have some benefits, they still have drawbacks, such as low strength, wear resistance, and poor aesthetics, which limit their applications to non-stress-bearing regions. However, to improve on this drawback, Resin-Modified Glass Ionomer Cement (RMGIC) was introduced through the addition of resin constituents to conventional GIC, leading to improved physical characteristics of RMGIC, such as moisture and aesthetic resistance².

RMGIC has been found to be an integral part of the sandwich restoration approach, where it serves as a liner or a base layer beneath the composite resin restoration. The sandwich restoration process takes advantage of both the restorative materials, employing the fluoride release property and dentinal bonding ability of RMGIC along with the aesthetics, durability, and mechanical properties of composite resins^{2,3}. The effectiveness and lifespan of sandwich restorations are highly reliant upon the quality of the interface between RMGIC and composite resin.

Bonding between RMGIC and composite resins is a combination of chemical bonding and mechanical retention. Nevertheless, because of the smooth and hydrophilic nature of the RMGIC after maturation, it is difficult for the resin to infiltrate the surface

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and form strong interfacial bonds. Various surface pretreatment techniques have been suggested to enhance the adhesion of resin, such as phosphoric acid etching, air abrasion, and laser irradiation⁴. The use of phosphoric acid makes the surface more roughened by removing the superficial layer of the RMGIC^{4,5}. Moreover, the durability of the restorative interface is affected by the stresses related to temperature and moisture encountered inside the oral cavity. For this reason, it is common practice in laboratory tests to use techniques such as thermocycling and artificial saliva aging to mimic conditions found in vivo and assess the long-term stability of restorative materials^{6,7}.

Several techniques such as dye penetration techniques, radioisotope technique, confocal laser scanning microscopy (CLSM), and scanning electron microscopy (SEM) have been applied to determine the nature of adhesion bonding and penetration of the adhesives. SEM technique among all these methods is considered to be a good technique due to its high-resolution visualization capability⁸.

Previous studies have explored the bond strength of surface treated RMGIC; however, there is scant literature describing the association between penetration depth and bonding longevity after subjecting specimens to thermal cycling and aging under artificial saliva conditions. In

addition, few investigations have simultaneously compared adhesive penetration depth, bond strength after thermocycling, and adaptation at the RMGIC-composite interface under SEM analysis. Thus, this in vitro study aimed at evaluating the effect of three surface pretreatments on adhesive resin penetration and bond strength between RMGIC and composite resin using SEM evaluation. This study assumed that the application of surface treatments had no significant impact on both penetration depth and bond strength at the interface.

Materials And Methods

This in vitro study was conducted after obtaining approval from the Institutional Ethical Committee of Sri Siddhartha Dental College (SSDCHIEC/2025/41).

Sample Collection

Thirty freshly extracted human permanent maxillary premolars free from caries, restorations, cracks, or structural defects were selected for the study. Soft tissue remnants and debris were removed using an ultrasonic scaler. The extracted teeth were disinfected by autoclaving and stored in 0.1% thymol solution at room temperature until use to prevent dehydration and microbial contamination.

Cavity Preparation and Restoration Procedure

Standardized Class I cavity preparations were prepared on all specimens using a No. 245 carbide bur mounted on a high-speed handpiece under water coolant. The cavity dimensions were standardized to 3 mm in depth and 3 mm in width. The bur was replaced after every five preparations to ensure cutting efficiency and uniformity.

Resin-modified glass ionomer cement (RMGIC) was manipulated according to the manufacturer's instructions and placed incrementally in 1-mm

layers within the prepared cavities. Each increment was light cured for 20 seconds using an LED curing unit operating at an intensity of 1000 mW/cm².

Surface Pretreatment Protocol

The specimens were randomly allocated into three experimental groups (n = 10) according to the surface pretreatment protocol employed:

- **Group I (Control):** No surface pretreatment
- **Group II (Acid Etching):** Surface etching with 37% phosphoric acid (Ivoclar Vivadent) for 15 seconds
- **Group III (Air Abrasion):** Air abrasion using 50 µm aluminum oxide particles (Coxo, China) for 15 seconds at 60–80 psi pressure, applied at a 45° angle from a distance of 2 mm

Following surface pretreatment, all specimens were rinsed thoroughly with water for 30 seconds and gently air dried using oil-free compressed air. The Econom Plus adhesive system (Ivoclar Vivadent) incorporated with 0.1% Rhodamine B dye (TM Media, Delhi, India) was applied over the treated RMGIC surface using a microbrush and light cured for 20 seconds.

Subsequently, the cavities were restored using Te Econom Plus composite resin (Ivoclar Vivadent) with incremental placement technique. Each composite increment was light cured for 20 seconds.

Each experimental group was further subdivided into two subgroups (n = 5):

- **Subgroup I:** Scanning Electron Microscopy (SEM) evaluation
- **Subgroup II:** Shear bond strength analysis

Scanning Electron Microscopy Evaluation

Specimens allocated for SEM evaluation were sectioned buccolingually through the center of the

restoration using a diamond disc under water coolant. The sectioned samples were examined under a scanning electron microscope (JEOL JCM-6000 Plus, Japan) to evaluate the depth of adhesive resin penetration at the RMGIC–composite interface. Measurements were recorded in micrometers (μm) at standardized magnifications.

Three measurements of adhesive penetration depth were obtained from each specimen at standardized locations using ImageJ image analysis software (National Institutes of Health, Bethesda, MD, USA), and the mean value was considered for statistical analysis. All measurements were performed by a single calibrated examiner blinded to the study groups to minimize observer bias. SEM evaluation was performed at a standardized magnification of $\times 500$.

Shear Bond Strength Analysis

Specimens designated for shear bond strength testing were stored in artificial saliva (Entod Pharmaceuticals Ltd., India) and subjected to thermocycling for 7 days to simulate oral conditions. Thermocycling was performed between 5°C and 55°C with a dwell time of 30 seconds. Following aging procedures, shear bond strength testing was performed using a universal testing machine (Micro Mach Technologies S1300). Each specimen was positioned under a shear knife-edge loading apparatus, and load was applied at a crosshead speed of 0.5 mm/min until bond failure occurred. Bond strength values were recorded in megapascals (Mpa).

Bond strength (MPa) = Bonded area (mm^2) / Force (N)

Statistical Analysis

All statistical analyses were performed using SPSS software version 26.0 (IBM Corp., Armonk, NY, USA). Descriptive statistics, including mean,

standard deviation, median, minimum, and maximum values, were calculated for adhesive penetration depth and shear bond strength across all experimental groups. The distribution of data was assessed for normality using the Shapiro–Wilk test, while homogeneity of variance was evaluated using Levene's test. Since the data demonstrated normal distribution and homogeneity, intergroup comparisons were carried out using one-way analysis of variance (ANOVA). Multiple pairwise comparisons between groups were subsequently performed using Bonferroni post hoc analysis to identify statistically significant differences. For variables that did not satisfy parametric assumptions, nonparametric analysis was performed using the Kruskal–Wallis test followed by Dunn's post hoc comparison test. Pearson's correlation analysis was additionally employed to evaluate the relationship between adhesive penetration depth and shear bond strength values. The level of statistical significance was established at $p < 0.05$ with a 95% confidence interval. Sample size estimation and power analysis were performed using G*Power software version 3.0.1 (Heinrich Heine University Düsseldorf, Düsseldorf, Germany). The minimum sample size was calculated based on an anticipated effect size derived from previous similar studies, with a study power of 80% and alpha error probability of 5%.

Results

Adhesive Penetration Analysis

Scanning electron microscopic evaluation demonstrated significant differences in adhesive resin penetration depth among the three surface pretreatment groups. The air abrasion group exhibited the greatest mean adhesive penetration depth ($176.67 \pm 24.33\ \mu\text{m}$), followed by the acid etching group ($116.35 \pm 5.85\ \mu\text{m}$). The control group without surface pretreatment demonstrated the least adhesive penetration depth (34.19 ± 8.56

μm) (Table 1).

One-way ANOVA revealed a statistically significant difference among the groups ($p < 0.05$). Post hoc Bonferroni analysis demonstrated statistically significant pairwise differences between all experimental groups. SEM observations revealed deeper and more uniform adhesive infiltration in the air abrasion group when compared with acid etching and untreated specimens (Graph 1).

Shear Bond Strength Analysis

Significant differences in shear bond strength values were observed among the experimental groups following thermocycling and artificial saliva aging. The air abrasion group demonstrated the highest mean bond strength value (127.28 ± 7.48 MPa), followed by the acid etching group (64.57 ± 2.87 MPa). The control group exhibited the lowest bond strength value (25.76 ± 1.25 MPa) (Table 2).

Statistical analysis using one-way ANOVA demonstrated significant intergroup differences ($p < 0.05$). Bonferroni post hoc analysis revealed statistically significant differences between all groups. Surface pretreatment with air abrasion resulted in superior bond strength compared with acid etching and untreated specimens (Graph 2).

Correlation Analysis

Correlation analysis demonstrated a positive association between adhesive penetration depth and shear bond strength values. Groups exhibiting greater adhesive infiltration into the RMGIC surface also demonstrated improved bond strength values, indicating that enhanced resin penetration contributed to superior interfacial bonding (Table 3).

Table 1: Comparison of Mean Adhesive Penetration Depth among Experimental Groups

Group	Mean (μm)	SD	SE
Air Abrasion	176.67	24.33	10.88
Acid Etching	116.35	5.85	2.62
No Treatment	34.19	8.56	3.83

One-way ANOVA showed statistically significant differences among groups ($p < 0.05$).
SD: Standard deviation; SE: Standard error

Table 2: Comparison of Mean Shear Bond Strength among Experimental Groups

Group	Mean (MPa)	SD	SE
Air Abrasion	127.28	7.48	3.34
Acid Etching	64.57	2.87	1.28
No Treatment	25.76	1.25	0.56

One-way ANOVA showed statistically significant differences among groups ($p < 0.05$).
SD: Standard deviation; SE: Standard error;
MPa: Megapascal

Table 3: Correlation between Adhesive Penetration Depth and Bond Strength

Variable	Highest Value	Moderate Value	Lowest Value	Interpretation
Adhesive Penetration Depth	Air Abrasion (176.67 μm)	Acid Etching (116.35 μm)	No Treatment (34.19 μm)	Greater surface modification enhanced adhesive infiltration
Bond Strength	Air Abrasion (127.28 MPa)	Acid Etching (64.57 MPa)	No Treatment (25.76 MPa)	Increased adhesive penetration improved bond strength

Discussion

The longevity and clinical success of sandwich restorations depend largely on the integrity of the bond formed between resin-modified glass ionomer cement (RMGIC) and composite resin. Adequate adhesive penetration into the RMGIC surface plays a critical role in establishing micromechanical interlocking and improving interfacial stability. Therefore, the present study evaluated the effect of different surface pretreatment protocols on adhesive resin penetration and bond strength at the RMGIC–composite interface using scanning electron microscopy (SEM).

The findings of the present study demonstrated that surface pretreatment significantly influenced both adhesive penetration depth and shear bond strength. Among the tested groups, air abrasion produced the greatest adhesive penetration depth and highest bond strength values, followed by phosphoric acid etching, while the untreated control group demonstrated the least favorable results. A positive correlation was observed between adhesive penetration depth and bond strength, suggesting that enhanced adhesive infiltration contributes to improved interfacial bonding.

The superior performance observed with air abrasion may be attributed to the formation of a uniformly micro-retentive surface topography following abrasion with aluminum oxide particles. Air abrasion increases surface roughness and surface energy by creating microporosities and partially removing the superficial resin-rich layer of RMGIC. This altered surface morphology facilitates deeper penetration of adhesive resin and promotes stronger micromechanical interlocking between RMGIC and composite resin. SEM evaluation in the present study revealed more uniform and deeper adhesive infiltration in the air abrasion group when compared with the other

experimental groups (Fig 1). Similar findings have been reported by Nihalani et al., Otsuka et al., and Zakavi et al., who demonstrated improved interfacial adaptation and enhanced bond strength following airborne-particle abrasion of RMGIC surfaces^{4,12,24}.

The air abrasion group also demonstrated the highest shear bond strength values following thermocycling and artificial saliva aging. The improved bond strength observed in this group may be related to the enhanced adhesive penetration and increased surface area available for bonding. Greater micromechanical retention at the interface may improve stress distribution and reduce the possibility of interfacial failure during thermal and mechanical loading. Similar observations have been reported by Sauro et al. and Lima et al., who concluded that airborne-particle abrasion significantly improves the bonding performance of restorative materials by enhancing surface microroughness and adhesive interaction^{17,20}.

In the present study, phosphoric acid etching also improved adhesive penetration depth and bond strength when compared with the untreated control group. Acid etching partially dissolves the superficial matrix of RMGIC, thereby increasing surface roughness and facilitating adhesive infiltration. Although the values obtained with acid etching were lower than those observed with air abrasion, the improvement was statistically significant (Fig. 2). These findings are in agreement with previous studies by Farshidfar et al. and Otsuka et al., who reported enhanced bond strength following phosphoric acid pretreatment of glass ionomer-based restorative materials^{1,12}.

The untreated control group demonstrated the lowest adhesive penetration depth (Fig. 3) and bond strength values among all groups. The relatively smooth and hydrophilic surface of

untreated RMGIC may limit effective adhesive infiltration and reduce micromechanical retention at the interface. Insufficient resin penetration may adversely affect interfacial adaptation and compromise long-term restoration durability. Similar findings have been reported in earlier studies evaluating the bonding behavior of untreated RMGIC surfaces^{3,21}.

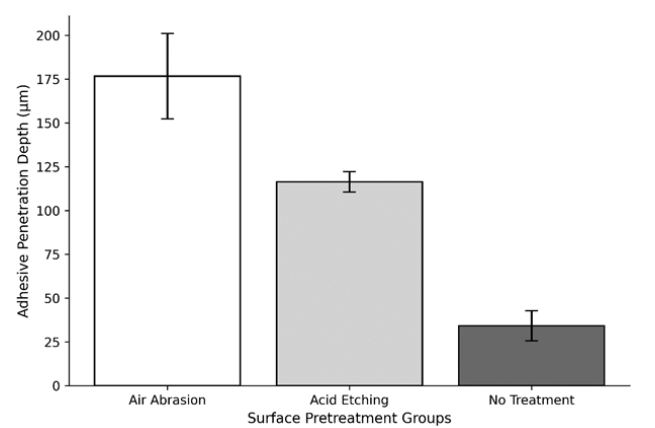
Thermocycling and artificial saliva aging were incorporated in the present study to simulate oral environmental conditions and evaluate the durability of the bonded interface. Thermal fluctuations and moisture exposure may contribute to interfacial degradation through hydrolytic breakdown, water sorption, and stresses generated by differences in thermal expansion coefficients between restorative materials. Despite these aging procedures, the air abrasion group maintained superior bond strength values, indicating improved interfacial stability and resistance to degradation.

The positive correlation observed between adhesive penetration depth and bond strength highlights the importance of effective surface pretreatment in improving the performance of sandwich restorations (Graph 3). Increased adhesive infiltration may enhance the formation of resin tags and improve micromechanical interlocking, thereby contributing to greater interfacial integrity and bond durability. The higher bond strength values observed in the present study may be attributed to differences in specimen configuration, bonded surface area, testing methodology, and enhanced micromechanical interlocking produced by aggressive surface pretreatment.

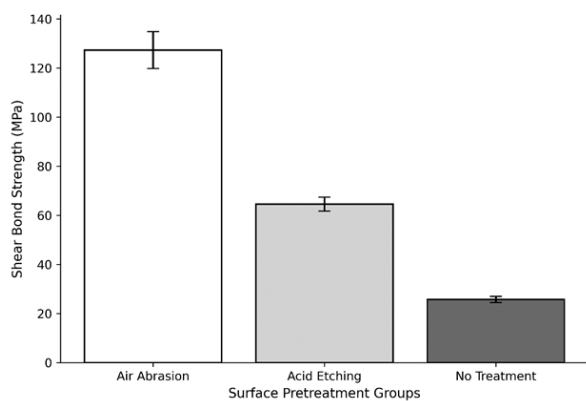
Certain limitations of the present study should be considered while interpreting the results. The study was conducted under in vitro conditions with a relatively small sample size, which may not completely replicate the complex oral

environment. Furthermore, only one adhesive system and limited aging duration were evaluated. Long-term clinical studies incorporating different adhesive systems, aging protocols, and oral simulation methods are recommended to further validate the present findings.

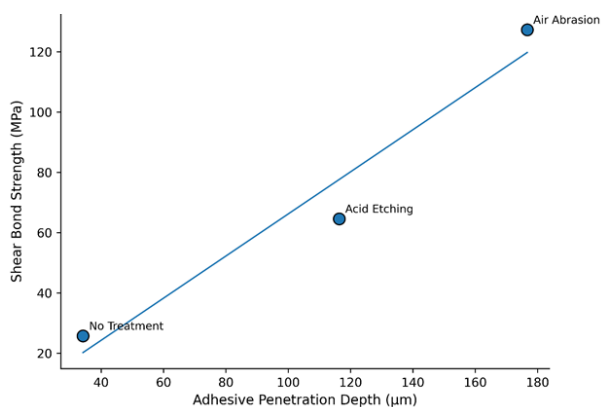
Within the limitations of the present study, air abrasion was found to be the most effective surface pretreatment protocol for improving adhesive penetration and bond strength at the RMGIC–composite interface. These findings may have important clinical implications in enhancing the longevity and performance of sandwich restorations.



Graph 1:- Mean Comparison of Adhesive Penetration Depth among Experimental Groups. The graph illustrates the mean adhesive resin penetration depth observed following different surface pretreatment protocols. The air abrasion group demonstrated the highest penetration depth, followed by the acid etching group, whereas the control group without surface pretreatment exhibited the lowest values. Error bars represent standard deviation.



Graph 2:- Mean comparison of shear bond strength among experimental groups. The graph represents the mean shear bond strength values obtained following different surface pretreatment protocols. The air abrasion group demonstrated the highest bond strength, followed by the acid etching group, while the untreated control group exhibited the lowest values. Error bars indicate standard deviation.



Graph 3. Correlation between adhesive penetration depth and shear bond strength. The graph demonstrates a positive correlation between adhesive penetration depth and shear bond strength among the experimental groups. Increased adhesive infiltration into the RMGIC surface was associated with improved interfacial bond strength.

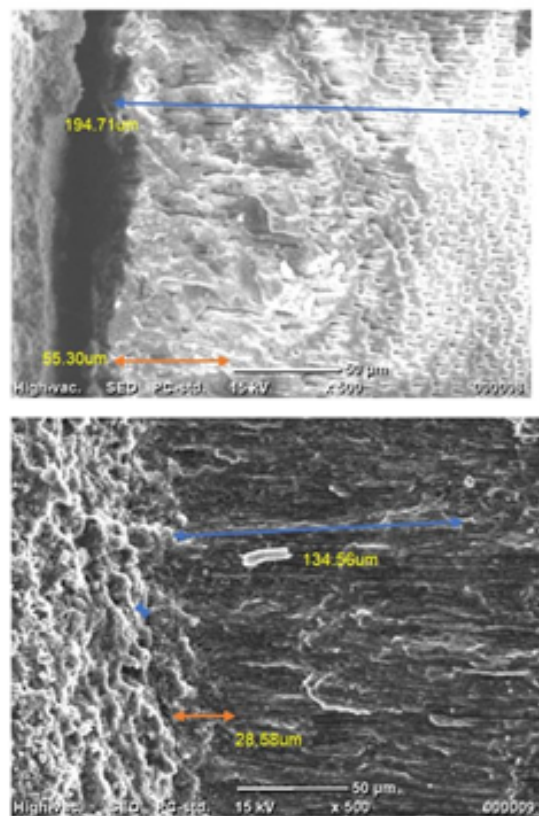


Figure 1:- SEM analysis depicting the depth of adhesive penetration following air abrasion pretreatment protocol.

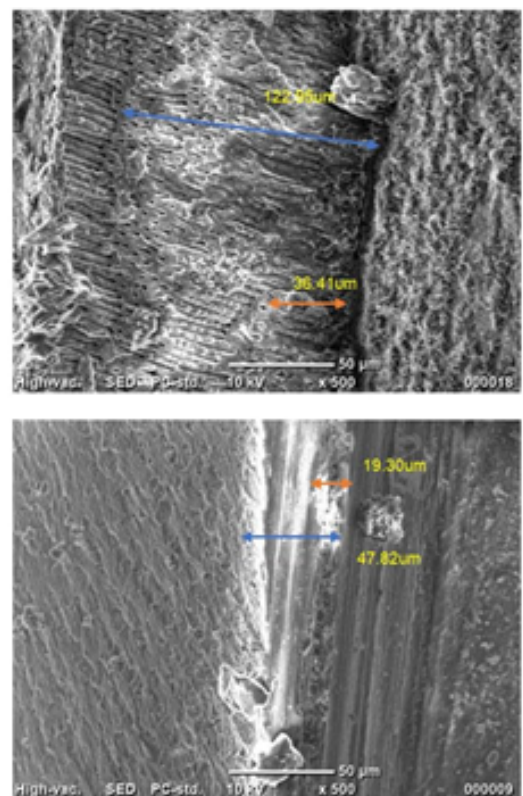


Figure 2:- SEM analysis depicting the depth of adhesive penetration following acid etching pretreatment protocol.

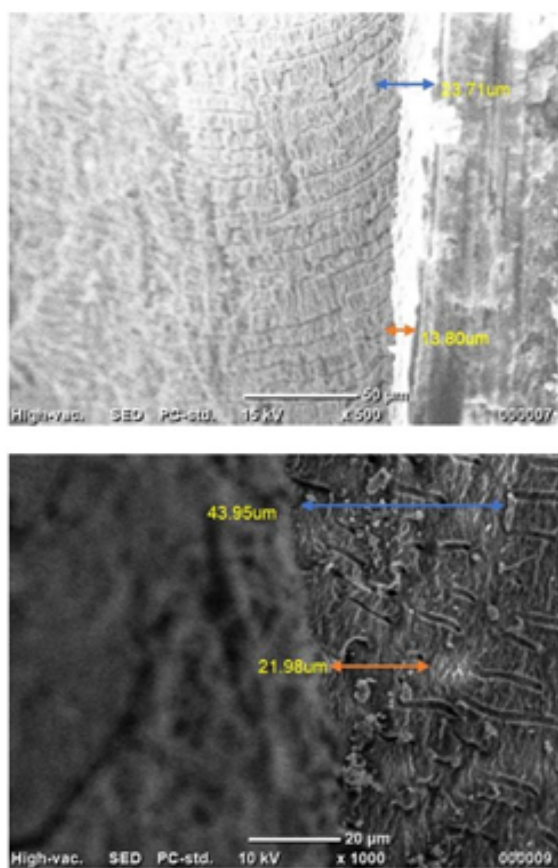


Figure 3 :- SEM analysis depicting the depth of adhesive penetration following no surface pretreatment protocol.

Conclusion

Surface pretreatment of RMGIC significantly improved adhesive resin penetration and shear bond strength at the RMGIC–composite interface. Among the evaluated pretreatment protocols, air abrasion demonstrated the greatest adhesive penetration depth and highest bond strength values, followed by phosphoric acid etching, whereas the untreated control group exhibited the least favorable performance. The improved interfacial characteristics observed following thermocycling and artificial saliva aging indicate that enhanced adhesive infiltration contributes to superior bond durability and interfacial stability in sandwich restorations.

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