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An in vitro comparison of tensile bond strengths of resin- and methacrylate-based sealers to dentin, gutta-percha, and resin-based coated core surfaces

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Purpose: To evaluate the adhesion of AH26 and Epiphany sealers to gutta-percha/Resilon surfaces and dentin in the presence or absence of the smear layer.

Methods: Dentin, gutta-percha, and Resilon surfaces were filled with different combinations of AH26 and RealSeal sealers in eight groups: Group 1- Smear (-) dentin, filled with dentin bonding agent and AH26 sealer; Group 2- Smear (+) dentin, AH26; Group 3- Smear (-) dentin, AH26; Group 4- Smear (-) dentin, RealSeal sealer. In the remaning groups, gutta-percha and Resilon pellets were used as experimental surfaces to test the sealer adhesion. The samples were attached to a universal testing machine and pulled at 1 mm/s to determine the tensile bond strengths.

Results: Statistical analyses revealed that the smear free dentin filled with AH26 had a significantly higher bond strength than those of the other groups ($p < 0.05$). The presence of the smear layer and dentin bonding agent significantly reduced the adhesion of AH26 to dentin ($p < 0.05$). The bonding of AH26 to Resilon and gutta-percha was significantly better than those of the RealSeal sealer ($p < 0.05$).

Conclusion: The tensile bond strength of AH26 sealer to gutta-percha/Resilon and dentin was superior to that of RealSeal sealer.

Keywords: Adhesion, resin-based sealer, smear layer, Resilon.

Introduction

The main objective of root canal treatment is to eliminate the bacteria and their toxins from the root canal by disinfection and obturate the root canal space in three dimensions (1). Various methods and materials have been developed for the obturation of root canals and for this purpose using a core material and sealer is widely accepted. Although a correlation between the sealing ability and adhesion of the sealer has not been established clearly, the sealer's tight adhesion to canal walls and core material is an apparent advantage, as it too much space the stability of the root canal filling during post space preparation and prevent bacterial proliferation by eliminating the spaces (2,3).

Gutta-percha (GP) has been most widely used as a core root canal obturation material in modern endodontics; however, it does not bond to the sealer or the root canal

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dentin (4,5).

To overcome this problem and reduce the intracanal leakage, experientally total etch and self-etch adhesives have been applied to the intraradicular area before the root canals were filled with GP (6). A characteristic feature of dentin bonding agents (DBA) is their ability to penetrate the dentin tubules. Since microleakage in the root canals is caused by the microgaps between the sealer and dentin or the sealer and GP, researchers use the DBA's ability to penetrate into the dentinal walls to create a hybrid layer between the sealer and the dentin wall and claim that this layer can reduce the microleakage (6,7). Studies have focussed on new endodontic adhesive technology and have tried to minimize intracanal leakage by increasing the adhesion between the root canals and the filling material (8).

In 2004, a new adhesive principled root canal obturation system was introduced. This system was commercially presented as Epiphany (Pentron Clinical Technologies, Wallingford, CT, USA) or RealSeal (SybronEndo Corp., Orange, CA, USA).

The RealSeal sealer's matrix comprises a mixture of urethane dimethacrylate, polyethene dimethacrylate, bisphenol-A-glycidyl dimethacrylate (BisGMA), ethoxylated BisGMA, and barium sulphate, silica, calcium hydroxide, bismuth oxide, photoinitiators stabilizers, and pigments (9). This new system includes a self-etch primer that increases the sealer's adhesion, and Resilon cones (Pentron Clinical Technologies, Wallingford, CT, USA) that are used instead of gutta-percha. Resilon, introduced as a reliable alternative to GP, is a polycaprolactone polymer that contains bioactive glass and radiopaque fillers. It is a thermoplastic, synthetic polymer-based, bonded root canal filling material, developed to bond to the adhesive sealer that bonds to dentin, thus creating a monoblock against microorganisms (10).

In endodontics, the smear layer is defined as an amorphous layer covering the root canal dentin walls formed by the friction of the canal instruments used during the shaping of the root canals (11). This layer should be removed completely since the smear layer contains necrotic/infected tissue residues and microorganisms, that prevent the irrigation solutions and intra-canal medicaments from penetrating into the dentinal tubules (12). The smear layer purportedly obstructs the penetration of sealer tags into the dentinal tubules resulting in a reduction in the adhesion of root canal sealers (13).

This study aimed to compare the tensile bond strengths of resin based AH26 and methacrylate-based RealSeal sealers to GP, Resilon surfaces, and dentin in the presence and absence of a smear layer. The null hypothesis was that the RealSeal system would create a monoblock structure.

Materials and Methods

Preperation of dentin discs

Eighty extracted single-rooted human mandibular premolar teeth were used in this study. The teeth were subject to organic tissue elimination by immersion in 5.25% sodium hypochlorite solution followed by the removal of all soft tissues by scaling with a periodontal scaler, and then stored in distilled water until use. Each tooth was molded in an orthodontic acrylic material placed in a standard mold.Using a diamond saw (Isomet, Buehler Ltd, Lake Bluff, IL), the enamel was removed by horizontal sectioning 1 mm below the cementoenamel junction and 7 mm below this point to obtain a section containing exposed dentin surfaces (Figure 1a) in an acrylic block measuring 9 x 10 x 7 mm. The diameter of canal spaces in dentin specimens were measured by a compass (JensenJP-1; Jensen Industries, North Haven, CT, USA), and they were divided in four groups of similar average diameter. The dentin surfaces were sandpapered with a #400 grit SiC paper (Atlas Zımpara Sanayi AŞ, İstanbul, Türkiye) to ensure the formation of a smear layer. In groups with a smear layer, the specimens were placed in bottles containing 5 ml of 5.25% NaOCl (Wizard, Rehber Kimya San, İstanbul, Türkiye) and shaken for 5 min, and then kept in distilled water until use. For smear-free groups , the samples were placed in bottles containing 5 ml of 17% EDTA (Wizard, Rehber Kimya San., İstanbul, Türkiye) and shaken for 5 min, irrigated with 5 ml of 5.25% NaOCl, and then kept in distilled water until use.

Preperation of GP discs

Steel cylinders with a diameter of 9 mm and a height of 3 mm were used as a mold for GP discs. The cylinders were placed on a glass slab and stabilized with boxing wax. The GP pellets (Figure 1b) (Diadent Group International Inc, Seoul Korea) were heated (80°C) in a Soft-Core oven (Soft-Core Dental Production ApS, Copenhagen, Denmark), and after softening they were placed in cylinder and condensed by a cement condenser for 30 secs. Thus GP discs with a height of 3 mm and a diameter of 9 mm with a shiny surface (glass side) were obtained. The GP discs were placed on the prepared acrylic blocks (9 x 10 x 7 mm), with the shiny surfaces facing upwards, and fixed with an cyanoacrylate cement (Henkel KGaA, Duesseldorf, Germany).

Preperation of Resilon (RealSeal Pellet) discs

The protocol described above was repeated with RealSeal pellets (Figure 1c) (SybronEndo, Orange, CA, USA) to form Resilon discs.

Fig. 1. **(a)** Dentin surface, **(b)** GP Pellets, **(c)** Resilon Pellets.

Bonding procedures and groups

A thin silver cylinder with 4.2 mm internal diameter and 6 mm height (0.3 mm wall thickness) was used for the application of materials in the adhesion test (Figure 2). Each cylinder was fixed to the center of experimental surfaces (dentin, gutta-percha, Resilon) with a thin layer of sticky wax to prevent sealer leakage. The materials used for the adhesion test were resin based AH26 sealer (Dentsply DeTrey GmbH, Konstanz, Germany), Scotchbond Multi-Purpose Plus DBA (3M ESPE AG Dental Products, Seefeld, Germany), and RealSeal Sealer (SybronEndo, Orange, CA, USA). The application of materials for bonding procedures and group formation for tensile testing were performed as follows:

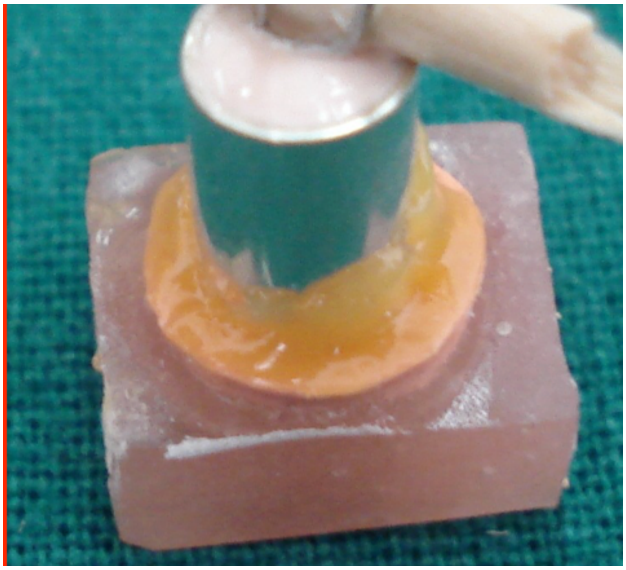


Fig. 2. Application of experimental materials for adhesion test

Group 1 dentin; smear (-), DBA and AH26 sealer: The primer of Scotchbond Multi-Purpose Plus (SMPP) was applied to the dentin surface with a bonding brush and the remnants were removed with an absorbing paper. Equal amounts of (0.5 mm) adhesive and catalyst of SMPP were mixed in a glass godet and applied to the dentin surface with a brush, and the remnants were removed. After the polymerization of DBA, AH26 sealer was mixed according to the manufacturer's suggestion and applied in cylinder with the help of a dental syringe.

Group 2 dentin; smear (+), AH26 sealer: Only AH26 was applied to the dentin surface with a dental syringe as in Group 1.

Group 3 dentin; smear (-), AH26 sealer: AH26 was applied as in Group 2.

Group 4 dentin; smear (-), RealSeal primer and sealer: RealSeal primer was applied to the dentin surface with its own brush and the remnants were removed with an absorbing paper. Subsequently, the RealSeal sealer was applied with its own mixing tip.

Group 5 Resilon, AH26 sealer: Resilon pellets were used as an experiental surface and AH26 was applied as in Group 2 and 3.

Group 6 GP, AH26 sealer: GP pellets were used as an experimental surface and AH26 was applied as in Groups 2, 3 and 5.

Group 7 Resilon, RealSeal sealer: Resilon pellets were used as an experiental surface and the RealSeal sealer was applied as in Group 4 but the primer was not used.

Group 8 GP, RealSeal sealer: GP pellets were used as an experimental surface and RealSeal sealer was applied as in Group 7.

Each experimental group was placed in 100% relative hu-

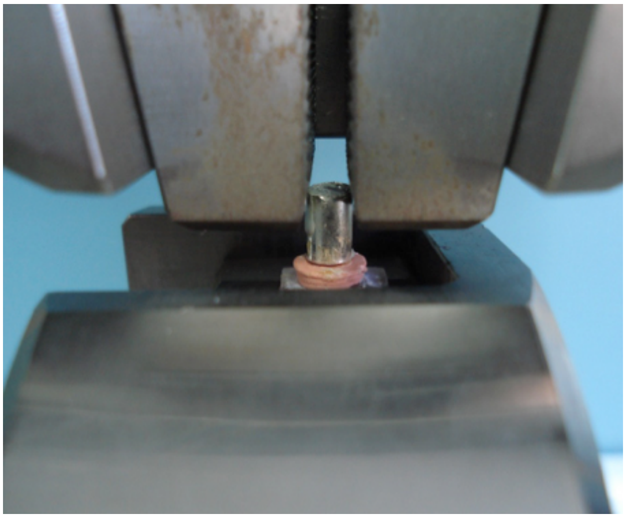


Fig. 3. The fixation of specimens to the immovable frame of the Autograph Universal Test Machine.

midity at 37°C for 7 days and allowed to set.

Tensile Tesing

Each specimen was fixed to the immovable frame of the Autograph Universal Test Machine (Autograph AG-IS; Shimadzu Co, Kyoto, Japan). Then, the tip on the upper arm of the device, which applies force, was lowered in the vertical direction and fixed to the cylinder mold (Figure 3). The tensile strength of the specimens was measured at a cross-head speed of 1 mm/min. The maximum tensile load at failure was recorded in newtons by a computer and divided by the cross-sectional area of the cylinder to express the tensile bond strength in megapascals (MPa).

Stereomicroscope and Field Emission Scanning Electron Microscopy (FESEM) examination

All specimens were examined under 10x magnification with a stereomicroscope (Leica Microsystems, Wetzlat, Germany); adhesive failures were classified as adhesive, cohesive, or mixed.

Five specimens per group were examined under FESEM. The morphologic structure of ruptured surfaces, adhesive failures, and the relation of experimental materials with smear (+) and smear (-) dentin were examined under different magnifications.

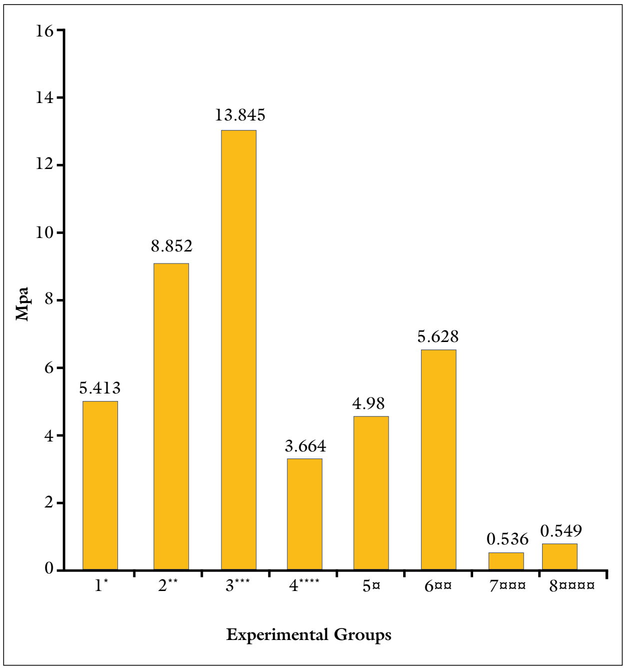
Statistical analysis

The normal distribution of groups were evaluated with the Kolmogorov-Smirnov test and after the confirmation of normal distribution , the statistical analysis were performed with one way ANOVA test with significance set at $p< 0.05$.

Results

Figure 4 presents the average tensile bond strengths of all groups. One-way ANOVA revealed a significant difference among the groups ($p< 0.05$). Among the groups in which we tested the adhesion to dentin surfaces, Group 3, in which the AH26 sealer was applied to the dentin surfaces after the removal of the smear layer, showed significantly higher adhesion values (13.84 ± 4.04 MPa) than those of the other 3 groups ($p< 0.05$). The presence of a smear layer significantly reduced the adhesion of AH26 sealer to dentin ($p< 0.05$). Using the Scotchbond Multi-Purpose Plus DBA on dentin before the application of AH26 sealer showed significantly higher adhesion values (5.41 ± 2.45 MPa) compared to those of RealSeal primer and sealer (3 .66 ± 2.19 MPa, $p< 0.05$).

The analysis of the interaction between AH26 and Realseal to gutta-percha and Resilon also revealed significantly different adhesion values ($p< 0.05$). The adhesion of AH26 to gutta-percha (6.62 ± 1.36 MPa) and Resilon (4.98 ± 1.46 MPa) showed significantly higher values compared to those of the RealSeal sealer's adhesion to gutta-percha



*Dentin surface / Smear (-); DBA+ AH26 sealer
**Dentin surface / Smear (+); AH26 sealer
***Dentin surface / Smear (-); AH26 sealer
****Dentin surface / Smear (-); RealSeal primer + RealSeal sealer
¤Resilon surface / AH26 sealer
¤¤GP surface / AH26 sealer
¤¤¤Resilon surface / RealSeal sealer
¤¤¤¤GP surface / RealSeal sealer

Fig. 4. The average tensile bond strenghts (MPa) of the groups.

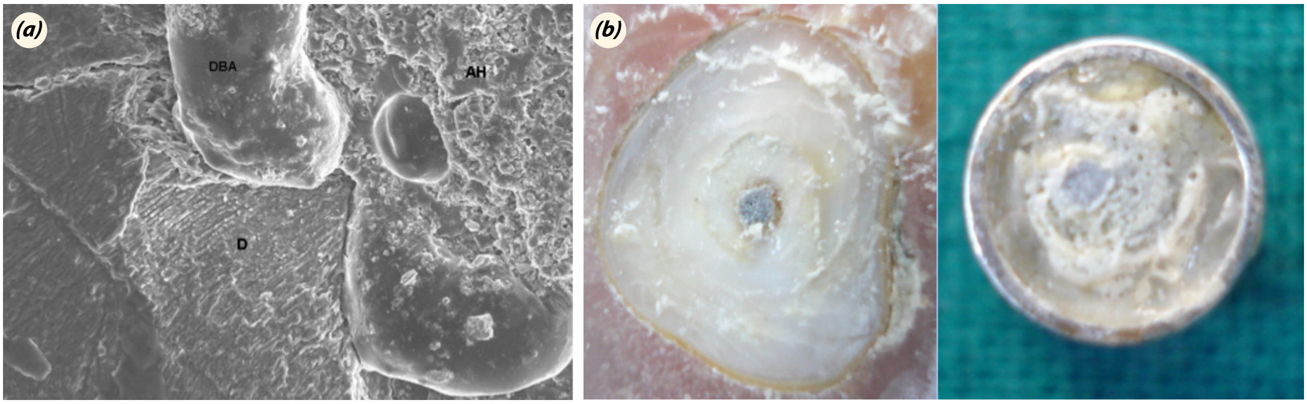


Fig. 5. SEM And Stereomicroscope images for mixed failure in Group1 **(a)** SEM image (D: dentin surface, DBA: dentin bonding agent, AH: AH26 sealer) **(b)** Stereomicroscope image.

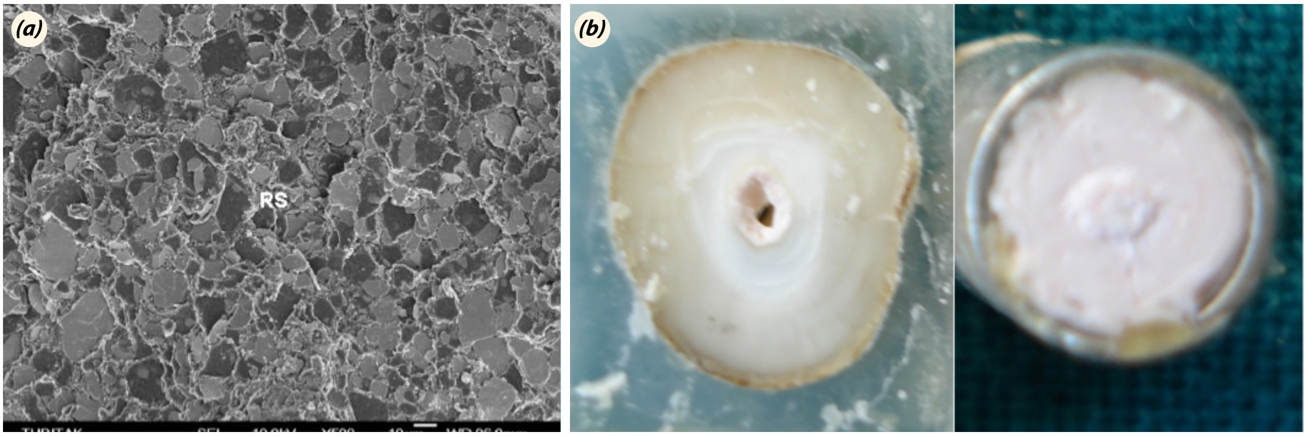


Fig. 6. SEM And Stereomicroscope images of adhesive failure in RealSeal sealer group **(a)** SEM image RS: Real Seal sealer, **(b)** Stereomicroscope image.

(0.54 ± 0.37 MPa) and Resilon (0.53 ± 0.29 Mpa, $p< 0.05$). AH26 bonded to gutta-percha better than Resilon did ($p< 0.05$); however the difference was not significant for the RealSeal sealer ($p> 0.05$).

Regarding the failure mode, AH26 sealer showed a predominance of mixed failures in nearly all groups (Figure 5a, b). The bonds between the RealSeal sealer and smear (-) dentin mostly (66.66%) failed adhesively (Figure 6 a, b), whereas failures between the RealSeal sealer and Resilon were predominantly mixed (93.33%) (Figure 7a1-2) and complete (100%) in the gutta-percha groups (Figure 7b1-2).

SEM examination revealed clear penetration of sealers and DBA into the dentin tubules when the smear layer was removed.

Discussion

Our results rejected the null hypothesis that the use of RealSeal sealer with primer and Resilon is capable of forming a monoblock in the root canals. AH26 displayed a significantly enhanced bonding to dentin, gutta-percha, and Resilon when compared to those of the RealSeal sealer. In addition, the adhesion between the RealSeal sealer and Resilon was lower than that between the RealSeal sealer and GP; however, the difference was not significant. The alleged monoblock system could be achieved using the Realseal system. (14). According to our study data, the use of DBA with the combination of AH26 sealer on smear-free dentin indicated that the penetration of adhesives into the dentinal tubules does not increase the sealer's bond strength as Mannocci and Ferrari found (6); conversely, it may result in an apparent reduction.Therefore the adhesives seem to be too distant to strongly adhere with dentin and core materials.

Adhesion is a desired characteristic for materials used in root canals. In endodontic literature, the general tests used for measuring the adhesion of endodontic sealers are tensile strength, shear, and push-out tests, however, none of these has been accepted as a standard among researchers. Tensile tests measure the perpendicular force to

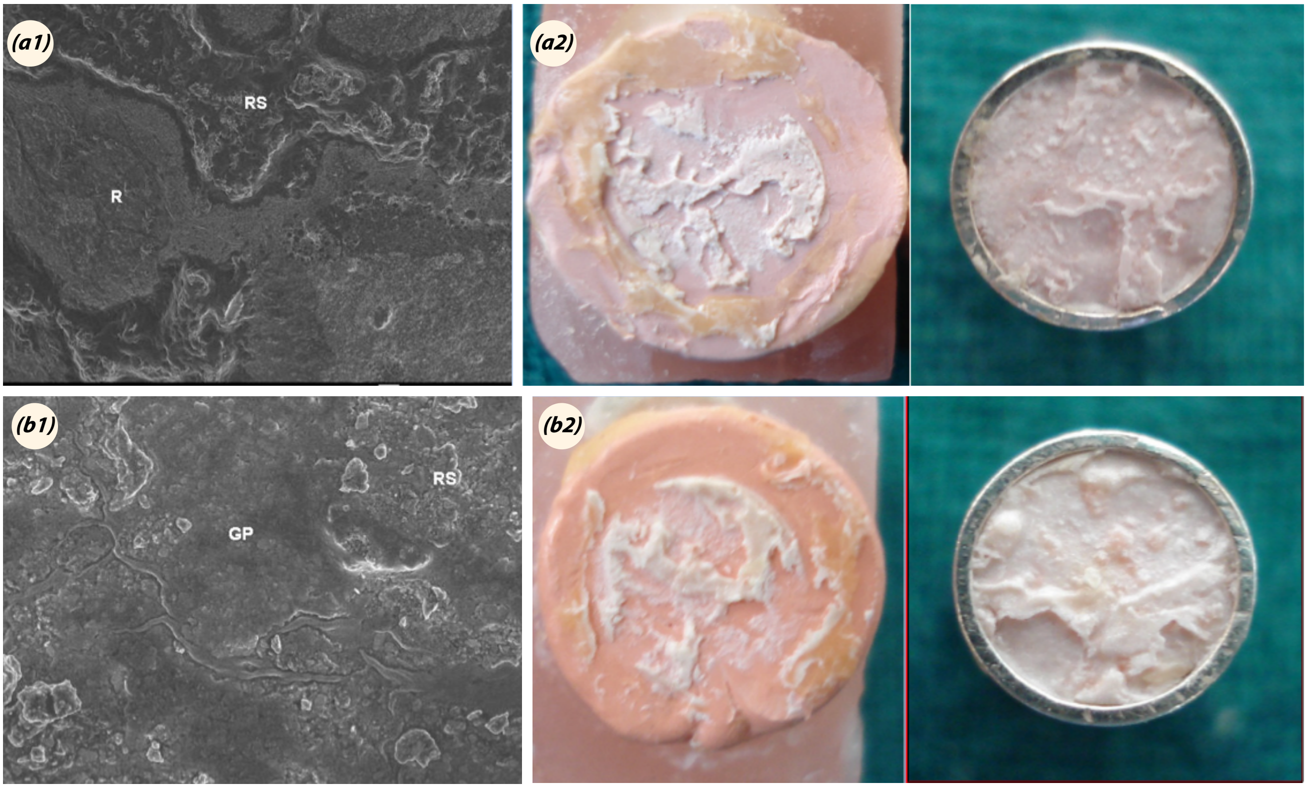


Fig. 7. SEM And Stereomicroscope images of mixed failures between RealSeal sealer and Resilon (a1,2) and gutta-percha (b1,2) groups, **(a1)** SEM image (R: Resilon surface, RS: RealSeal sealer), **(a2)** Stereomicroscope image, **(b1)** SEM image (GP: Gutta-percha surface, RS: RealSeal sealer), **(b2)** Stereomicroscope image

break the bond between a material and surface; however, in shear tests, the force is parallel to the interface between the material and surface (15). The push-out test is advantageous because its samples are easy to prepare and it can even measure samples with low bond strength (16,17). Although the push-out test is frequently used in adhesion studies, it was not used in this study since it could not separately evaluate the bond strength of the sealers to the dentin surfaces and/or gutta-percha/Resilon surfaces. Considering the difficulties in the application of force to the binding interface in the shear test, we used the tensile test, which requires more bulk but produces more uniform stressing than those provided by the shear test (18).

Although the correlation between sealer bond strength and clinical success remains debated, low bond strength obturation materials may show more defects between the sealing material and dentin surface because of polymerization stress (14). Nevertheless, the increased dislocation resistance of sealers to radicular dentin and gutta-percha/Resilon surfaces may be advantageous in maintaining the integrity of the root canal filling during the preparation of post space within filled canal spaces (19). The sealers fill the irregularities, lateral canals, and voids in the root canal because of their flowability and bond solid core materials such as GP to the canal wall. In endodontic literature, the root canal sealer plays an important role in three-dimensional obturation (20). AH26 sealer can penetrate into dentin tubules and canal irragularities because it has a strong wetting effect, flowability, and long setting time (21). Some studies (6,22) applied combinations of AH26 sealer and DBAs on root canal to evalute the adhesive material's effects on apical leakage, quality of the resin-dentin interface, and sealer adhesion, owing to the DBA's ability to penetrate into demineralized dentin to create a gap-free resin collegen hybrid layer. In this study, the Scotchbond Multi Purpose Plus DBA significantly reduced the bond strength of the AH26 sealer to the dentin. This might have been due to the different DBAs and testing methods used (shear test instead of tensile test).

Gogos et al. (22) evaluated the bond strength of AH26 and a combination of AH26 with Single Bond, Bond 1, and Clearfill SE Bond DBAs by shear test, and reported that the DBAs significantly increased the bond strength of AH26 to dentin. Conversely, we found that the Scotchbond Multi Purpose Plus DBA significantly reduced the bond strength of AH26 sealer to the dentin. Differences in the testing methods (shear test instead of tensile test), types of DBAs, and the method of application of DBAs might be the reasons for these discrepancies. Gogos et al. (22) polymerized the DBAs by light-cure after applying them to the dentin surfaces. A study where DBA was applied into the root canals reported difficulties in polymerization with the light sources, since these were not developed for endodontic use (23).

The configuration factor (C-factor) is also mentioned as a major problem for the polymerization of resin-based materials throughout the root canal (8,24). It is defined as the ratio of bonded to unbonded surface areas of cavities. A high C-factor that maximizes the polymerization shrinkage stresses may act as the main factor for the unsuccessful results of the usage of adhesive systems in long and narrow root canals (8,24). In this regard, to provide similarity with clinical conditions and to establish the standardization in the application of testing materials in our study, no light source was used and a dual-cure DBA was selected; thus, polymerization was achieved by adding a catalyst.

The adhesives (RealSeal and DBA) showed a significantly low bond strength to dentin when compared to AH26 sealer. In the DBA group, DBA partially penetrated the open dentin tubules and some remained on the dentin surface, whereas some remained on the AH26 sealer surface, showing a cohesive failure. In the RealSeal group, the RealSeal primer partially penetrated the exposed dentin surfaces and showed primary extensions towards the RealSeal sealer in the part remaining on the RealSeal surface. The failures in this group occurred cohesively within the RealSeal sealer. The reason for the low bond strengths in adhesive groups seems to be the weak bonding between their own molecules. Polymerization shrinkage of the resin sealer may be another reason for the low bond strengths that was observed in the RealSeal group. The size, type, and content of filler particles have an effect on the amount of polymerization shrinkage as well as the type of matrix used. This shrinkage may form some stresses that separate the resin-based sealer from the canal walls, resulting low bong strengths (25).

Some studies suggest that the low concentration of dimethacrylate, which is present in the matrix component of Resilon, may be a reason for the absence of free radicals within the polymerized Resilon material resulting in low bonding values between Resilon and RealSeal sealer (17,26). However, we noticed that the RealSeal sealer also showed low bonding values with gutta-percha.

In this study, in samples where the rupture surfaces were examined with SEM, the AH26 sealer spread perfectly into the dentin and almost formed a replica of it. When all groups were analyzed, AH26 predominantly performed mixed failures; however, cohesive failures were also observed, which indicates strong adhesion between surfaces, especially in dentin groups. The possible reason is that AH26 bonds strongly to dentin when compared to gutta-percha and Resilon, and it also exhibits high cohesion between its molecules (27).

Sly et al. (28) examined the bond strength of AH26/gutta-percha or Epiphany/Resilon in root canals that they filled using the System B and vertical condensation method with push-out method. They reported significantly higher bond strength (1.70 MPa) in root canals filled with AH26/gutta-percha than those filled with Epiphany/Resilon (0.51 MPa), which is parallel with our results.

Jainaen et al. (16), evaluated the bond strength of three different sealers (AH Plus, EndoREZ, RealSeal sealer) using either sealer only or sealer with tapered single cone in the root canals by push-out test. They stated that the bond strength in root canals filled with AH Plus/gutta-percha is significantly stronger than the RealSeal/Resilon group. Researchers reported that if only the sealer was used without using a master cone, the bond strength was higher in all sealer groups. Researchers found that the RealSeal sealer attached better to dentin than to Resilon, and stated that the monoblock concept should be revised due to the weak binding to Resilon.

Ungor et al. (17) examined the adhesion of AH Plus/gutta-percha, AH Plus/Resilon, Epiphany sealer/gutta-percha, and Epiphany sealer/Resilon on root canals filled by lateral condensation method. They reported that the dentin bond strength was significantly highest in the Epiphany/gutta-percha group, whereas the weakest attachment was seen in the AH Plus/Resilon group, and that the bond strength in the Epiphany/Resilon group was lower than the Epiphany/gutta-percha group. Although the results of this study, which reveal that the highest bond strength is in the Epiphany/gutta-percha group contradicts with our study, the fact that the RealSeal sealer has a higher bond strength to gutta-percha than Resilon is in line with our study.

According to our study, the AH26 sealer bonded significantly better to dentin than the RealSeal sealer. Our results conform with the results of most studies comparing the bonding strengths of AH26 and AH Plus sealers with Epiphany sealer (16,17,26,28,29). These results contradict the idea that there is a chemical bond between the RealSeal sealer and Resilon, as the manufacturer claims, and that the RealSeal sealer must be used with Resilon. The results also suggest that the monoblock structure should be reconsidered.

During root canal instrumentation, a smear layer is formed. A smear layer may act as potential reservoir for bacteria and can also prevent the extension of sealer tags into the dentinal tubules, thereby reducing the adhesion for sealers, and should be removed (30). Gettleman et al. (31) examined the effects of the presence or absence of smear layer on dentin bonding strength of 3 different sealer types, and reported that the removal of the smear layer significantly increased the bond strength of AH26 to dentin, while the increase in the bond strengths of Sultan and Sealapex sealers was not significant. Our findings concur with those of this study that the presence of the smear layer reduces the bond strength of AH26 sealer to dentin by approximately 40%. The possible reason for the increased bonding strength of AH26 sealer on smear free dentin is the penetration of the sealer into the dentinal tubules and spreading of it on the irregular dentin surfaces that is formed after the removal of smear layer.

In a literature review, there was no study using DBA, AH26, and RealSeal system that we used in our study, which examined the bond strength of these materials on dentin surfaces in the presence and absence of smear layer and gutta-percha or Resilon surfaces by the tensile test method.

The role of adhesion of sealers on clinical success is still questionable. Saleh et al. (32) evaluated the bacterial penetration of root canals filled with AH Plus/Apexit sealer and gutta-percha or RealSeal system and stated that increased adhesion did not improve the bacterial resistance; factors such as antibacterial properties and physical barrier may play a role in the success. Moinzadeh at al. (33) investigated the correlation between fluid transport and push-out strength in root canals filled with RealSeal sealer and declared that no significant correlation was found between the adhesion properties and fluid transport. As Saleh et al. (32) stated that adhesion is not the only variable in the success of the root canal therapy. However, considering that post spaces are created in most of the root canal-treated teeth, it is one of major factors. As with a loose adhesion, during the preparation of post spaces, the root canal filling may dislocate, thus ruining the integrity of the root canal filling.

In a recent long-term clinical study, Barborka et al. (34) compared the outcome of teeth obturated with AH Plus and the RealSeal system and reported that the RealSeal system had nearly six times greater chances of failure when compared with those of AH Plus. This study is important because, to our knowledge, it is the first clinical study to report the long-term outcomes of the RealSeal system.

Conclusion

The tensile bond strength of the RealSeal sealer to gutta-percha/Resilon and dentin was not superior to that of AH 26 to gutta-percha/Resilon and dentin. The so-called monoblock of RealSeal and Resilon seems questionable. Resin-based sealers such as AH26 have higher adhesion properties on Resilon, gutta-percha, and especially on smear-free dentin when compared to those of the RealSeal sealer. Considering the polymerization problems in the root canal, many developments are needed for adhesive systems to ensure their application in routine endodontic use.

Authorship Contributions: Concept: I.K.; Design: I.K., S.G.; Supervision: I.K.; Materials: S.G.; Data: S.G.; Analysis: S.G.; Literature search: S.G.; Writing: S.G.; Critical revision: S.G.;

Conflict of Interest: None declared.

Ethical Approval: This study was rewieved and approved as a doctoral thesis (no. 224272) by an advisory board appointed by Health Sciences Institute, Istanbul University, as an adequate ethical requirement at the time of its inception (2008).

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