Shear Bond Strength Measurement of Three Different Adhesive Sealers to Dentin & Gutta-percha (In Vitro Study)

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ABSTRACT

Background and Objective: Bond strength of the adhesive sealers to both human dentin and gutta-percha is critical property for success of the root canal sealing. This in vitro study aimed to measure the shear bond strength (SBS) of three different adhesive sealers (glass -ionomer based sealers (Ketac-Endo) and (Diaket) and epoxy resin-based sealer (AH 26) to both the gutta-percha and human dentin.

Methods: Part I: The dentin substrate was obtained from single rooted human teeth. The dentin specimens were treated either with EDTA 15% or phosphoric acid 37% to achieve the removal of smear layer. Three adhesive sealers (glass-ionomer based sealers (Ketac-Endo) and (Diaket) and epoxy resin-based sealer (AH 26) were placed on the dentin surfaces both with and without the use of the bonding agent. Bond strength was tested using a single plane shear test assembly. Part II: Discs of gutta-percha with a diameter of 8 mm and thickness of 4mm were made by softening of the gutta-percha cones and fixed with plaster in rings. Four millimeter long sections of polyethylene tube filled with freshly mixed sealer were placed on the gutta-percha and tested using a single plane shear test assembly.

Results: By using t-test, the results revealed that there was highly significant difference between epoxy resin-based sealer and the two other glass-ionomer based sealers at $p< 0.01$ with in favor of the AH26 sealer.

Conclusions: The bonding system & the dentin pretreatment increased the adhesive potential of the AH26 sealer which had higher shear bond strength than the two glass -ionomer based sealers

Key words: Shear Bond Strength, Adhesive Sealers

INTRODUCTION:

Microleakage whether from an apical or a coronal direction remains a clinical problem and a possible cause of failure of endodontic therapy[1,2]. A desirable property of a root canal sealer, therefore, is to have good sealing ability[3]. In addition an endodontic sealer should adhere firmly both to dentine and to gutta-percha. Adhesion of the root canal filling to the dentinal walls is important both in static and dynamic situations since it eliminates any space allowing penetration of fluids between the filling and the wall and resists any tendency towards dislodgment of the filling during subsequent manipulation (e.g. post space preparation)[4] It seems that the bond of the root canal filling to the walls should be a major factor of interest. The adhesion of endodontic sealers to dentin has been the subject of several studies[5-7]. The stability of the filling also depends upon the bond of endodontic sealer to gutta-percha. The adhesion depends on a multitude of interacting factors including the surface energy of the adherent (dentin or gutta-percha); the surface tension of the adhesive (sealer); its ability to wet the surfaces & cleanliness of the adherent surface. The sealer must also have cohesive strength to hold the obturation together[8, 9]. Wennberg and Orstavik[10] studied the adhesion of eight

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endodontic sealers placed between bovine dentine and gutta-percha. AH-26 (an epoxy resin based sealer) showed the highest bond strength, followed by Diaket (ESPE, Seefeld, Germany) and CRCS (Hygenic, Akron, OH, USA). Similar findings for the adhesive properties of AH-26 have been reported in other studies[11-13]. During chemo -mechanical preparation, a layer of debris, the smear layer, is formed. Some studies have shown that removal of the smear layer enhances the adhesion of sealers to the root canal wall[12,14]. Timpawatt *et al*[15] supported the view that pretreatment of root canal with phosphoric acid or citric acid should be used in association with glass-ionomer sealer to achieve the most effective removal of the smear layer and enhance adhesion. The use of dentin bonding agents it is known from restorative dentistry that improves the adhesion of composite resins. The bonding agents create a micromechanical interlock between the dentin collagen and resin by forming a hybrid layer[16]. Only a few studies have attempted to evaluate the use of bonding agents in root canal obturation. Mannocci and Ferrari[17] evaluated the sealing ability of AH-26 sealer with and without dentin bonding agents. They found that the use of bonding agents significantly reduced the microleakage in specimens when the pretreatment of root canal walls was performed with phosphoric acid 37%. It is of interest, whether the improved control of the microleakage can be attributed to an increase in adhesion of AH-26 to dentin in the presence of bonding agent. There might be a relation between the sealer function & the bonding agent. This study aimed to measure the shear bond strength of three different resin sealers to both dentin and gutta-percha with and without use of bonding agent.

MATERIALS & METHODS:

The samples were obtained from one hundred twenty freshly extracted teeth. The external tissue debris, calculus, soft tissue and the clotted blood were removed with scalar and tooth brush under running tab water and collected in a special container containing distilled water to preserve the teeth from dehydration.

Part I

1. Sample preparation

The teeth (posterior teeth) were embedded in auto-polymerized methy1 methacrylate. The crowns and 1.0 mm of the roots below the cemento-enamel junctions were left exposed. The occlusal enamel surfaces were ground with diamond cutting discs using low-speed hand piece to expose flat peripheral dentin surfaces and each disc was changed after being used for 15 specimens. The occlusal surfaces, then, were grounds with fine stone disc using lathe with water-cooling system to remove any remnant enamel until exposing the peripheral dentin surfaces. The teeth were wet-polished with 600-grit silicone carbide abrasive papers manually to create a uniform smear layer. Specimens were stored in distilled water at room temperature for 48 hours before bonding prevent dentin dehydration. After that, a measuring ribbon measured the coronal diameter of each specimen, and the radius of each specimen was calculated to find the interfaced surface area in square millimeters (mm^2).

2. Sample Grouping

The dentin bonding system was one-bottle bonding agent; a light-cure syntac “single-component, multi-use” system “5th generation”:

All specimens were randomly assigned to three groups of forty each to receive one of the following sealers:

Group 1) glass-ionomer based sealer (Ketac-Endo);

Group 2) epoxy resin-based sealer (AH 26);

Group3) glass-ionomer based sealer (Diaket).

Each group subdivided into 4 groups of ten each to receive one of the following treatment combinations:

A- EDTA 15%, without syntac.

B- EDTA 15%, with syntac.

C- Phosphoric acid 37%, without syntac.
D- Phosphoric acid 37%, with syntac.

3. The treatment combinations application

Before dentin conditioning, the dentin surface was re-polished with 600-grit SiC paper to produce a fresh smear layer, rinsed and gently air-dried. Dentin bonding agents were used according to the manufacture's instructions. For groups A & B, the dentin specimens were etched with 3 ml 15% EDTA (Prevest Denpro Ltd., India) for 2 min. and then rinsed with 3 ml 2.5% NaOCl and distilled water. Finally the dentin surfaces were dried with paper points. For groups C & D where the dentin was etched with 35% phosphoric acid (3M, St Paul, USA) for 15 sec. The etchant was rinsed for 15 sec. with distilled water, and the dentin was gently air dried for 5 sec. For groups B & D, the Syntac "single-component" (primer and adhesive in one bottle) was applied to all etched surfaces as first layer and air blown to a thin layer for 2 seconds and light-cured for 10 seconds. A second layer of syntac was then applied with the brush and again air blown to a thin layer for 2 seconds and light -cured for 10 seconds. Four millimeter long sections of polyethylene tube filled with freshly mixed sealer were placed on the conditioned dentin. These tubes were of different sizes according to the different diameters of the specimens were then attached to each specimen and tightly fitted to the outer surfaces of the teeth without any displacement. The AH-26 sealer (Densply, Germany, batch # 8760), Ketac-Endo (Fuji, Japan, batch # 34623) & the Diaket (ESPE, USA, batch # 5690) were mixed according to manufacturer instructions (powder/liquid ratio: 2:1) and placed on the dentin surfaces. After placement of the sealer, the entire assemblies (Plate, dentin specimens and bonded materials) were left for 12 hrs. at room temperature. After their initial set they were transferred to an incubator (37°C, 100% relative humidity) for period of 1 week.

4. Samples Testing

The dentin samples were mounted into a single plane shear test assembly, as described by Watanabe *et al*[18,19]. Each specimen was loaded parallel to the adhesive interface formed between the dentin and test material. Bond strength was tested using a Zwick universal test machine by subjecting samples to a shear load at a cross-head speed of 0.5 mm/min. until separation from the tooth. The failure loads were in kilogram and transferred into Newton's by multiplying the value by 9.8 and divided by the corresponding interface surface area in (mm^2) to get the shear bond strengths in Mega Pascal (MPa).

Part II

1. Sample preparation

Disks of gutta-percha were prepared by compaction a number of standardized cones of size # 70 (Gapadent, Germany, batch no. 010907). The cones were softened by short immersion in a thermostat controlled water bath (45+3°C) & compacted with a large plugger into a split ring mold 8 mm in diameter & 4 mm height. Proper control of the heating of gutta-percha is necessary to avoid physio-chemical & structural-molecular changes[20]. After cooling, the gutta-percha disks were embedded in plaster of Paris at the surface of a 1 inch phenolic ring. After the plaster had set, the surface of the pellet was lightly polished on wet waterproof polishing paper to standardize the surface preparation of each pellet to get intimate contact between the sealer material & the gutta-percha substrate.

2. The sealers application

The sealers were mixed according to manufacturer instructions. Four millimeters long sections of polyethylene tubing with 6 mm internal diameter were filled with freshly mixed sealer & carefully placed with one open side contacting the gutta-percha, perpendicular to its surface. The more fluid sealers were poured into the tubes that were held in contact with the gutta-percha disks, taking care to let the material flow to the substrate & avoid air bubbles entrapment. Due to the great variety of types of sealers, different methods & times were used to ensure complete setting. All sealer cylinders were allowed to bench set for 12 hrs. to ensure that initial setting reaction had taken place. Specimens were then stored at 100% humidity and 37°C for period of 1 week. The shearing bond strength of the sealers was tested after they had set completely.

3. Sample Grouping

All specimens were randomly assigned to three groups of ten each to receive one of the following sealers:

Group 1) glass-ionomer based sealer (Ketac-Endo);

Group 2) epoxy resin-based sealer (AH 26);

Group 3) glass-ionomer based sealer (Diaket).

4. Samples Testing

The phenolic rings were inserted into a special holder, & locked with a screw in such a way that the flat surface of the substrate was on a perpendicular plane on which a blunt chisel, fixed to the moving upper jaw of the device, could slide. The samples were subjected to shear loading until failure using shear punch test with Zwick universal test machine (Model 1454, Germany). A stainless steel chisel-shaped rod was directed toward the interface between the two substrates, allowing the sealer to be sheared from the gutta-percha substrate in displacement speed of 0.5 mm/min. The failure loads were in kilogram and transferred into Newton's by multiplying the value by 9.8 and divided by the corresponding interface surface area in (mm^2) to get the shear bond strengths in Mega Pascal (MPa).

RESULTS:

Part I By using t-test, the results revealed that there was highly significant difference between epoxy resin-based sealer & the two other glass-ionomer based sealers at $p < 0.01$ with in favor of the AH26 sealer that recorded 13.58 mega pascal (MPa) SBS mean value. However, the Diaket sealer had better shear bond strength than that of the Ketac-Endo sealer but the difference was non-significant at $p > 0.05$. Using of the bonding system gave higher shear bond strength than the samples that were not using it & the difference was highly significant at $p < 0.01$. The uses of 15% EDTA had highly significant difference than the use of Phosphoric acid 37% in removing the smear layer & in turn give higher SBS mean values at $p < 0.01$, see (Table 1,2 & 3) & (Figure 1).

Part II By using t-test, the results revealed that there was highly significant difference between epoxy resin-based sealer & the two other glass-ionomer based sealers at $p < 0.01$ with in favor of the AH26 sealer that recorded 7.8234 mega pascal (MPa) shear bond strength mean value. However, the Diaket sealer had better shear bond strength than that of the Ketac-Endo sealer & the difference was highly significant at $p < 0.01$, see (Table 4, 5 & 6) & (Figure 2).

Table 1: Part I-Descriptive Statistics for the differences in the shear bond strength values between sealers & teeth surfaces.

Groups	N	Min of SBS	Max of SBS	Mean	SD
1B	10	7.368	11.643	9.3627	± 1.01043
1C	10	6.874	10.636	8.3806	± .90840
1D	10	7.853	11.34	9.2529	± .84161
2A	10	7.457	12.565	10.2638	± 1.21132
2B	10	11.986	14.647	13.5897	± .64352
2C	10	8.975	12.098	10.3241	± .74456
2D	10	10.964	14.496	12.5844	± .83603
3A	10	6.437	10.765	8.5578	± 1.02037
3B	10	7.329	11.665	9.4482	± 1.02233
3C	10	6.532	9.543	8.3763	± .73182
3D	10	7.964	11.654	9.3738	± .89948

Table 2: Part I-Paired sample comparison for the differences in the shear bond strength values between sealers & teeth surfaces .

Pairs	Groups	Mean	N	Std. Deviation	Std. Error Mean
I	**1**	8.8721	40	± 1.00175	.15839
	2	11.6905	40	± 1.68932	.26710
	3	8.9390	40	± 1.01250	.16009
II	**A+C**	9.0658	60	± 1.26053	.16273
	B+D	10.6019	60	± 1.98634	.25644
III	**A+B**	9.9524	60	± 1.98890	.25677
	C+D	9.7154	60	± 1.65834	.21409

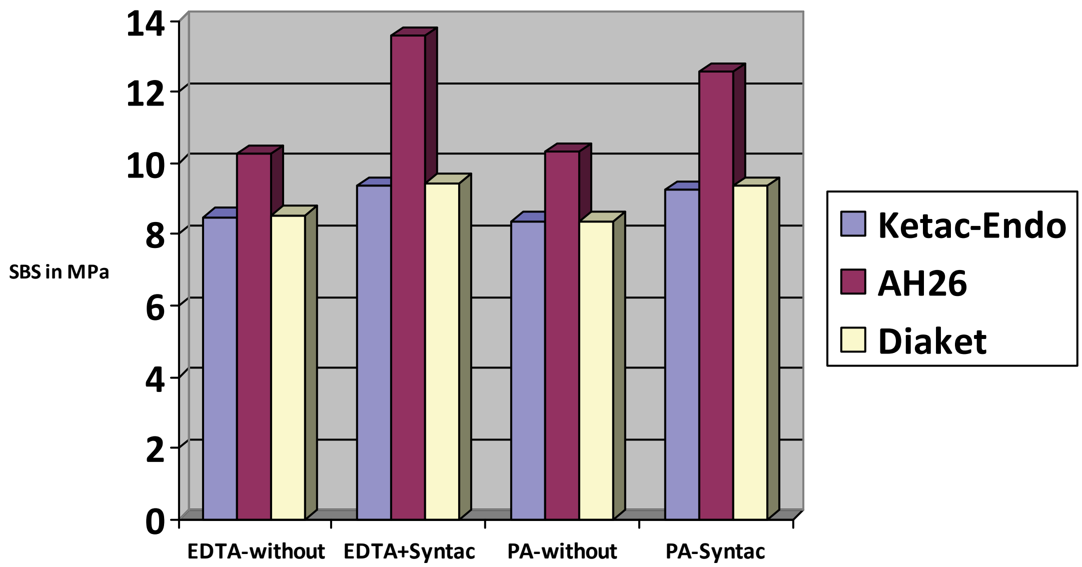

Figure 1: Part I for the differences in the shear bond strength values between sealers & teeth surfaces

Table 3: Part I-t-test for the differences in the shear bond strength values between sealers & teeth surfaces

Pair of differences	Paired Differences					t	df	Sig. (2-tailed)
	Mean	Std. Deviation	Std. Error Mean	95% Confidence Interval of the Difference				
				Lower	Upper			
1-2	-2.81840	1.06231	.16797	-3.15814	-2.47866	-16.780	39	.000
2-3	2.75147	1.03765	.16407	2.41962	3.08333	16.770	39	.000
1-3	-.06693	.21103	.03337	-.13442	.00057	-2.006	39	.052
A+C - B+D	-1.53615	.99203	.12807	-1.79242	-1.27988	-11.995	59	.000
A+B – C+D	.23705	.47299	.06106	.11486	.35924	3.882	59	.000

Table 4: Part II-Descriptive Statistics for the differences in the shear bond strength values between sealers & gutta-percha.

Groups	N	Min of SBS	Max of SBS	Mean	SD
1	10	1.264	3.653	2.5237	± .56414
2	10	6.478	8.956	7.8234	± .58676
3	10	2.483	4.326	3.8489	± .49352

Table 5: Part II-Paired sample comparison for the differences in the shear bond strength values between sealers & gutta-percha.

Pairs	Groups	Mean	N	Std. Deviation	Std. Error Mean
I	1	2.5237	10	± .56414	.17840
	2	7.8234	10	± .58676	.18555
	3	3.8489	10	± .49352	.15606

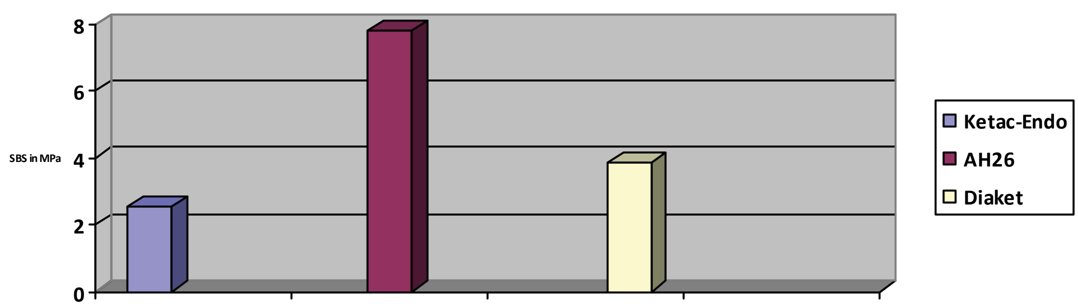

Figure 2: Part II for the differences in the shear bond strength values between sealers & gutta-percha .

Table 6: Part II-t-test for the differences in the shear bond strength values between sealers & gutta-percha.

Pair of differences	Paired Differences					t	df	Sig. (2-tailed)
	Mean	Std. Deviation	Std. Error Mean	95% Confidence Interval of the Difference				
				Lower	Upper			
1-2	-5.29970	.03019	.00955	-5.32130	-5.27810	-555.082	9	.000
2-3	3.97450	.23267	.07358	3.80806	4.14094	54.018	9	.000
1-3	-1.32520	.23771	.07517	-1.49525	-1.15515	-17.630	9	.000

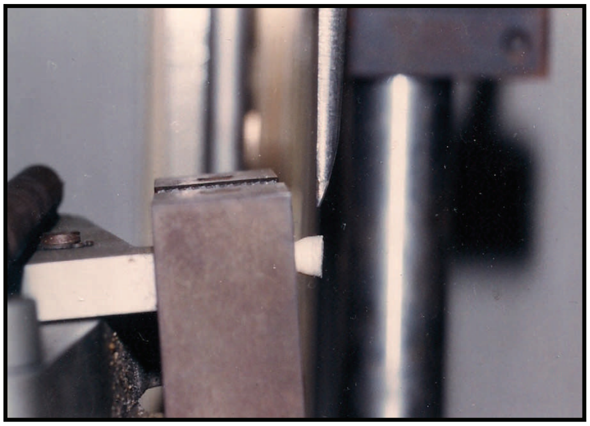

Figure 3: Sample testing

Figure 4: Sample preparation

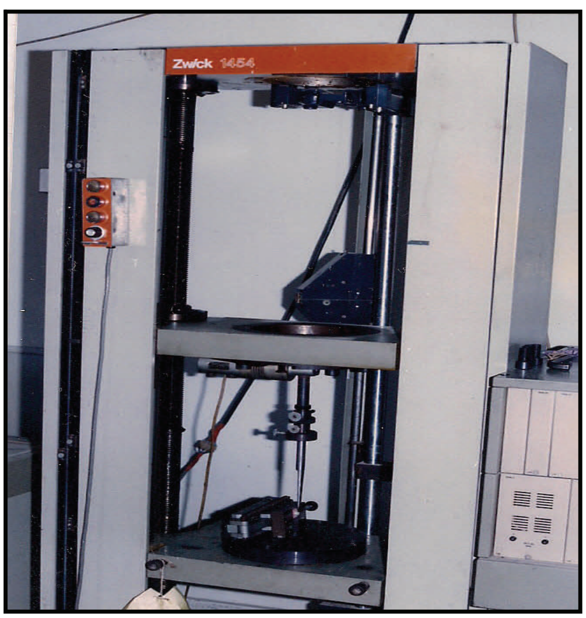

Figure 5: Zwick Universal Testing Machine

DISCUSSION:

This study compared the shear bond strength of AH-26 sealer in human dentin in combination with bonding agent with other two sealers. The dentin was treated either with phosphoric acid or EDTA to achieve the removal of smear layer and to provide better adhesion. Two mechanisms of adhesion may be distinguished: chemical and mechanical. In the case of chemical bonding, a smooth surface generally results in better adhesion. Micromechanical bonding, on the other hand, requires the presence of irregularities on the surface of the dentin matrix into which the material can penetrate[20]. The removal of smear layer permits the penetration of sealers into the dentinal tubules and promotes greater bonding between dentin and sealer, increasing the bond strength values compared to untreated dentin. The results of our study showed that pretreatment, either with phosphoric acid or EDTA did affect the adhesive properties of AH-26. Timpawat *et al*[15] found that pretreatment of dentin with 35% phosphoric acid resulted in a significantly higher bond strength for a glass-ionomer sealer (Ketac-Endo) than treatment with EDTA. Blomlof *et al*[6] compared EDTA (24%) and phosphoric acid (32%) conditioning of dentin in combination with two different dentin bonding systems. They found that use of EDTA in combination with All-Bond 2 resulted in significantly greater bond strength to dentin than did conventional acid etching. Our results showed that EDTA is effective and can be used as an alternative to phosphoric acid which is a strong acid and probably more toxic to the periapical tissues. It should also be stated that EDTA selectively removes mineral from dentin surfaces without compromising the collagenous matrix[19]. This is in contrast to the action of phosphoric acid, which not only dissolves the mineral phase of dentin but also results in a recession of the collagen matrix which may interfere with one-bottle adhesive systems, such as Syntac, have been introduced to simplify the bonding procedures and decrease the time needed for application. Manocci and Ferrari[17] compared the in vitro apical microleakage of fillings performed with AH-26 sealer, used in conjunction with two dentin bonding agents and laterally condensed gutta-percha, with the microleakage of root canal fillings performed with lateral condensation and AH-26 root canal sealer alone. The adhesive systems were All-Bond 2 and Scotchbond Multi Purpose Plus. Their results showed that the use of dentin bonding agents significantly reduced the apical microleakage. Scanning electron microscopic observation revealed a large amount of adhesive in the apical area in the Scotchbond Multi Purpose Plus samples while a thin hybrid-like layer was observed in all specimens. The results of the present study showed that the use of dentin bonding agents improved significantly the adhesion of AH-26 sealer with the root canal dentin. These adhesive systems dissolve the smear layer and partially demineralize the underlying dentin surface[17]. The dissolved smear layer is incorporated in the bonding process. Two types of acidic molecules are present in the current systems: phosphate monomers and polycarboxyl molecules. The actual rationale behind these systems is to superficially demineralize dentin and simultaneously infiltrate the exposed collagen fibril scaffold with resin up to the same depth of demineralization[17].Lee *et al*[22] evaluated the tensile bond strength between five endodontic sealers and either dentin or gutta-percha. AH-26 gave the highest bond strength of all the sealers tested. The epoxy resin-based AH-26 is thought to be able to react with any exposed amino groups in collagen to form covalent bonds between the resin and collagen when an epoxide ring opens. According to the authors, seventy percent of the AH-26-dentin bond failures were adhesive. However, the dentin was not resin (look like glue) in the formulationstrength to gutta-percha was recorded & this may be due to the presence of Biphenol A Epoxy of this sealer. This result suggests that the resin can react with the components of gutta-percha that may induce chemical bonding, especially with smooth surface gutta-percha[23]. Lee *et al*[22] showed that the AH26 sealer bonded more strongly to gutta -percha than the dentin. The slightly acidic pH of AH26 sealer associated with potential chemical bonding, due to ring opening (hydroxyl ring of the chemical structure of the BPAE resin that become available to bond chemically with the gutta-percha), may explain the high values obtained.Both glass ionomer based sealers had lesser values however the polyacrylic acid matrix of GIC contains multiple ionized carboxylate groups than can react with the mineral phase of gutta-percha, as the GIC could chelate with zinc in gutta-percha. A recent SEM study, showed a characteristic etch pattern on gutta-percha points sealed with a GIC, as well as there was associated evidence of chemical or physical bonding. The adhesion of the GIC to gutta-percha may be limited to surface phenomena of gutta -percha[22].

CONCLUSIONS:

The shear bond strength of the AH26 sealer was higher than the other two sealers and the Diaket sealer had higher shear bond strength than the Ketac-Endo. The bonding system & the dentin pretreatment increased the adhesive potential of the AH26 sealer which had higher SBS than the two glass-ionomer based sealers.

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