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Effects of Dentin Bonding Agents on Bonding Durability of a Flowable Composite to Dentin

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The present study evaluated the bonding durability of a flowable composite on bovine dentin using dentin bonding agents with different numbers of application steps: Scotchbond Multipurpose (three steps), Prime & Bond NT and One-Step (two steps), AQ Bond and Prompt L-Pop (one step). Shear bond strength tests were performed, and resin-dentin interface and fracture mode were observed. There were no significant differences in bond strength among the specimens within 37℃ storage group ($p>0.05$) and post-thermocycling group, except between Prompt L-Pop and Scotchbond Multipurpose ($p<0.05$) in the post-thermocycling group. Further, Scotchbond Multipurpose and One-Step showed significantly lower bond strengths after thermocycling ($p<0.05$). It was thus shown that the use of simplified bonding agents did not necessarily improve the bonding strength of flowable composites.

Keywords: Flowable composite, Dentin adhesives, Shear bond strength

INTRODUCTION

Recently, there is an increasing demand for a new class of low-viscosity “flowable resin composites” for tooth-colored restorations. Flowable composites were first introduced in 1995 to restore Class V lesions[1)]. Based on traditional hybrid composites, flowable composites are characterized by a smaller filler concentration, low modulus of elasticity, low viscosity, and remarkable wettability[1-3)]. To date, there are wide-ranging applications for flowable composites: Class V cavities, pits and fissures, base for composite restorations, amalgam margin repairs, and of late as filled adhesives.

Flowable composites were developed principally to provide their own unique brand of handling characteristics, rather than for their physical properties or bonding performance[1-3)]. As a result, little is known about their bonding performance. Some studies showed that traditional composites exhibited superior performance in all the mechanical properties tested−in terms of compressive strength, flexural strength, radiopacity, and toughness[2,3)]. Flowable composites, on the other hand, have been reported to adapt well to the cavity wall, and this optimal adaptation may result in improved adhesive perfomance[1-3)]. Therefore, in terms of clinical applications, it is recommended that flowable composites be directly applied on dentin as an adhesive system. As for bonding to enamel, it is now accepted as clinically reliable; but adhesion to dentin still remains unpredictable because of organic complexity.

Adhesive dentistry, including flowable composite restorations, can be explained as a simple relationship between bond strength and the stress generated by polymerization shrinkage[4)]. For dental materials used in adhesive dentistry, durable bond strength is one of the chiefest and foremost considerations[5)]. This is because a strong and permanent bonding to tooth structure will serve to minimize microleakage and maintain integration at the restoration area in the harsh oral environment.

However, there are only a few studies on the bonding behavior of flowable composites as compared with conventional composites. It should be noted that although flowable composites may be used as a liner together with overlying conventional composites, their bonding properties differ from those of conventional composites by virtue of their differing characteristics. Flowable composites may exhibit the capillary phenomenon−as influenced by viscosity and wettability−to act in the same way as a filled adhesive replacing the bonding agent of three-step systems. However, it has been demonstrated that flowable composites are not capable of hybridizing etched and primed dentin as efficiently as regular bonding agents[6].

Dentin bonding agents (DBAs) are used to improve the marginal seal of resin composite restorations at the composite-tooth interface—and they have been proven to be effective. Currently, many bonding systems are available but their mechanisms differ one from another. Indeed, the mechanism of a dentin bonding system is complex and relies on the component parts of each system. Traditionally, DBAs comprise separate components of etchant, primer, and adhesive—these DBAs are referred to as multi-component systems. With a chief aim and focus on simplicity, subsequent DBAs such as single-bottle and all-in-one systems have been developed. Thus, it could be said that modern approaches to adhesive dentistry are inclined to shortening the application time, simplifying the application procedure, and eliminating steps related with technique senstivity[5]. In practical terms, it would be the most convenient and ideal for the practitioner if restorative procedures could be made easier and simpler.

Against the plethora of DBAs currently available on the market, the hypothesis of the present study was that the bonding durability of a flowable composite subjected to thermocycling was affected by the type of dentin bonding agent used.

MATERIALS AND METHODS

Specimen preparation

One hundred bovine incisors extracted recently within the last six months were used in this study. They were kept frozen in distilled water. After defrosting, all the bovine teeth were cleaned and stored in 4℃ isotonic sodium chloride until use. The roots were sectioned with a low-speed diamond disk (Shofu Inc., Kyoto, Japan), and each crown was embedded in a custom-made silicon mold (diameter 20 mm, height 10 mm) with epoxy resin. The coronal enamel portion was then sectioned to expose the pure dentin surface with 180- to 600-grit silicon carbide papers under running water. After the dentin surface was finished, a microscopic examination (SMZ-U, Nikon, Tokyo, Japan) at ×50 magnification was done to ensure that there was no remaining enamel on the ground surface.

Filtek Flow (3M ESPE, St. Paul, USA) was used as the flowable composite, and specimens were randomly divided into five groups with the five different DBAs used (Table 1): Scotchbond Multipurpose (SBMP; 3M ESPE, St. Paul, USA) as a conventional three-step adhesive system, Prime & Bond NT (P&B; Dentsply, Konstanz, Germany) and One-Step (OS; Bisco, Itasca, USA) as single-bottle systems, AQ Bond (AQ; Sun Medical, Moriyama, Japan) and Prompt L-Pop (PLP; 3M ESPE, St. Paul, USA) as all-in-one, self etching primer systems.

For the bonding procedure of SBMP, dentin was etched for 15 seconds with 37% phosphoric acid gel, rinsed for 10 seconds, and primer was applied to the etched dentin and gently air-dried before adhesive resin application. P&B was carried out by etching the dentin with 34% phosphoric acid for 15 seconds, rinsed for 15 seconds, and blot-drying with moist cotton. After which, two consecutive coats of self-priming adhesive were applied onto etched dentin surface with a five-second gentle air blast. For OS, dentin was etched with 32% phosphoric acid semi-gel for 15 seconds and rinsed with water. After blot-drying of dentin surface, two consecutive coatings of adhesive were made without time lapse between the two coats. For AQ, a double-coating application was done with AQ sponge by stirring the expressed adhesive liquid for 3-5 seconds. PLP was activated in the disposable applicator and brushed on the dentin surface with double coats for 15 seconds each. All

Table 1 Products used in this study

Product	Manufacturer	Composition
Filtek Flow	3M ESPE, St. Paul, USA	Bis-GMA, TEGDMA, 47% filler: zirconia/silica
Scotchbond Multipurpose	3M ESPE, St. Paul, USA	HEMA, polyalkenoic acid, water, Bis-GMA
Prime & Bond NT	Dentsply, Konstanz, Germany	Di-trimethacrylate resin, PENTA, acetone
One-Step	Bisco, Itasca, USA	BPDM, HEMA, acetone
AQ Bond	Sun Medical, Moriyama, Japan	4-META, acetone, water, P-toluene sulfonic acid
Prompt L-Pop	3M ESPE, St. Paul, USA	Liquid 1: Methacrylated phosphoric ester, Bis-GMA, camphorquinone; Liquid 2: Water, HEMA, polyalkenoic acid

Bis-GMA: Bisphenol-A-glycidylmethacrylate; TEGDMA: Triethyleneglycol dimethacrylate; HEMA: 2-hydroxyethyl methacrylate; BPDM: Biphenyl dimethacrylate; PENTA: Dipentaerythritol penta acrylate monophosphate; 4-META: 4-methacryloxyethyl trimellitate anhydride adhesive layers were then light-cured (380-520 nm wavelengths) for 10 seconds with a plasma arc radiation unit (Flippo, Lokki, France) prior to composite resin application.

To ensure standardized and equal bonding of flowable composite specimens tested, a custom-made bonding jig was used. Dentin surface to be bonded was properly isolated with a plastic mold that contained a small cylindrical hole (diameter 3 mm, height 2 mm) within the jig. After preparing the dentin surface of each group according to each manufacturer's instructions, Filtek Flow was applied and light-cured for 10 seconds. To evaluate bonding durability, each group was subdivided into two categories: storage in distilled water at 37℃ (24 hours) and thermocycling (0-55℃, 1000 cycles) with a dwell time of 30 seconds in each bath.

Shear bond strength measurement

Specimens were mounted in a universal testing machine (Model 4200, Instron Inc., Canton, MA, USA) at a crosshead speed of 1.0 mm/min until bonding failure occurred. Shear bond strength was calculated as the peak load of failure divided by the surface area.

Statistical analysis

Differences between groups were determined by using one-way ANOVA with Tukey's HSD multiple comparison test ($p<0.05$).

Failure mode analysis

Fractured surfaces were air-dried and examined at ×20 magnification with a stereomicroscope (SMZ-U, Nikon, Tokyo, Japan) to determine the mode of failure. Failure modes were classified into three categories: adhesive failure between flowable composite and adhesive layer, cohesive failure within dentin or composite, or mixed failure of both adhesive and cohesive types.

Scanning electron microscope (SEM) evaluation

Specimens were fabricated in the same way as for shear bond test and sectioned in a perpendicular direction to the dentin-composite interfacial plane with Isomet. Each specimen was wet-ground with silicon carbide papers in a succeeding order from 600- to 2000-grit sizes. Final polishing was done on polishing cloths with diamond suspensions of 6, 3, and 1 μm grit sizes (Buehler Metadi Diamond Suspension, Buehler, USA). Samples were cleaned and dehydrated by immersing in ethanol solution (95%) and fixed in 2% paraformaldehyde. Then, samples were sputter-coated, mounted on aluminum holders, and observed under a SEM (S-4200, Hitachi, Tokyo, Japan) for the presence and morphological appearance of hybrid layer.

RESULTS

Table 2 shows the means and standard deviations of shear bond strength. Among the groups stored at 37℃ (24 hours), there were no statistically significant differences in bond strength ($p>0.05$). In terms of post-thermocycling comparison, PLP (5.2 MPa) produced a statistically higher value than SBMP (2.4 MPa), but was not significantly different from P&B, OS, and AQ. After thermocycling, it could be seen that the bond strengths of SBMP and OS were statistically lower than before thermocycling ($p<0.05$).

Table 2 Means and standard deviations of shear bond strength (MPa) (n=10)

Group	0 thermocycles	1,000 thermocycles
1 (SBMP)	4.4 ± 1.0^{a}	$2.4 \pm 1.1^{a,b}$
2 (P&B)	5.3 ± 1.1	3.9 ± 1.3
3 (OS)	6.0 ± 1.6^{a}	4.4 ± 1.0^{a}
4 (AQ)	4.9 ± 1.6	4.3 ± 1.7
5 (PLP)	5.4 ± 1.7	5.2 ± 1.0^{b}

Values are represented as mean ± SD.
a: Statistical difference ($p<0.05$) between pre-thermocycling and post-thermocycling subgroups.
b: Statistical difference ($p<0.05$) within the post-thermocycling subgroup.

Table 3 Types of failure observed in dentin-composite specimens

Group	Type 1	Type 2	Type 3
SBMP	7	3	-
SBMP*	8	2	-
P&B	7	3	-
P&B*	7	3	-
OS	5	4	1
OS*	6	4	-
AQ	7	3	-
AQ*	7	3	-
PLP	6	3	1
PLP*	7	3	-

*: After 1,000 thermocycles
Type 1: Adhesive fracture between flowable composite and adhesive layer
Type 2: Mixed fracture of both adhesive and cohesive types
Type 3: Cohesive fracture within dentin or composite

Table 3 shows the failure modes of fractured specimens. Most failures appeared to be of adhesive (67%) and mixed types (31%). Cohesive failures were recorded in OS and PLP prior to thermocycling.

DISCUSSION

A correlation exists between filler content and mechanical properties, particularly for modulus of elasticity: the lower the filler content, the lower it will be for modulus of elasticity and hence an increase in polymerization shrinkage[7,8]. The higher shrinkage of flowable composites over that of hybrids may indicate a potential for higher interfacial stress, but the lower rigidity and elastic modulus of flowable composites may be a counteracting factor[9]. To date, studies have revealed that the bond strength of flowable composites was clinically acceptable when applied on the enamel surface[10-12]. Nonetheless, flowable composites are produced mainly for use on etched dentin surfaces. On this matter, Frankenberg *et al.*[7] demonstrated that flowable composites did not fulfill the requirements to act as filled adhesives because they were not capable of hybridizing etched and primed dentin as efficiently as the commercially available dentin bonding agents. Further, Uysal *et al.*[13] revealed that flowable composites should not be used to replace bonding agents due to their lower bond strength values. Therefore, when a flowable composite is applied to dentin, it is mandatory to use it in combination with a DBA. Fortuitously, with the unique characteristics of flowable composites, a simple bonding procedure with reduced application steps could be achieved—which is especially desirable in pediatric dentistry.

All-in-one systems, which are modified self-etching DBAs, allow reduction in the number of steps required in the bonding process[14-16]. Separate etching, rinsing, and drying steps are no longer necessary—which means that not only time is reduced, but that technique sensitivity on blotting process to obtain maximum performance during the bonding procedure is also reduced[17]. However, PLP and AQ did not exhibit statistically higher bond strengths than OS, P&B, and SBMP prior to thermocycling at 37℃ ($p>0.05$), and only PLP showed statistically higher strength than SBMP after thermocycling ($p<0.05$). On this note, some studies have shown that PLP seemed less reliable than conventional or single-bottle adhesives[18-19]. Frankenberg *et al.*[7] also reported lower bond strength for PLP applied in one layer, but the application of two coats of PLP was successful in increasing the bond strength to sound dentin[20].

With all-in-one systems, the time allowed for the chemical reaction seemed to be extremely shortened[21]. Therefore, by a second application, additional supply of adhesive resin might improve the infiltration of resin monomers into the intertubular demineralized dentin[20] while a thicker, unfilled adhesive layer might provide a better stress-absorbing effect[22]. However, multiple coating with this system might go against its simplicity appeal—although the latest all-in-one systems could still boast of less technique sensitivity due to the reduction of application steps to only one step. In addition, factors such as air-blowing duration and pressure could also influence the bond strength of these all-in-one adhesives[23]—although it is still an ongoing debate whether deviations from the suggested protocols would affect the bonding performance. Some researchers reported that manufacturers' instructions were often not strictly adhered to[23], while Spreafico *et al.*[24] demonstrated that there was no technique sensitivity associated with the air-blowing step.

For the more recently developed all-in-one self-etching adhesives, they are more hydrophilic and hence more permeable to water derived from the underlying bonded dentin. This permeability can lead to a wide variety of seemingly unrelated problems, including incompatibility of chemically or dual-cured composites with simplified adhesives and expedited degradation of resin-dentin bonds[25]. To solve this problem, one proposed solution was to cover these hydrophilic adhesives with a hydrophobic adhesive or a thin layer of flowable composite[26]. In this study, PLP's hydrophilic nature might be compensated with the use of a flowable composite.

Presently, the commonly used solvents are namely water, acetone, or a mixture of these solvents. It should be noted that the solvent type affects the bonding technique: if organic solvent were used, dentin must not be dry but must rather be hydrated; if water were used, then dentin must not be too wet. These considerations have led to the development of water-organic solvent mixtures[27], such as the water-acetone mixed solvent used in AQ in this study.

In terms of irradiation, the manufacturer of AQ recommended the use of short-wavelength light (with the peak at 380 nm) as compared to products using camphorquinone (CQ) as a photoinitiator. For DBAs that contain an alternative photoinitiator (such as AQ) with an absorption spectrum different from that of CQ, the use of a narrow spectrum can result in insufficient polymerization of the bonding resin. This is due to some photoinitiators falling outside the absorption range of CQ, and hence causing a curing problem[28-30].

After 1,000 cycles of thermocycling, three-step SBMP and two-step OS showed statistically lower bond strengths than before thermocycling ($p<0.05$). As for OS, oxygen inhibition caused by an extremely thin resin layer might have prohibited bond establishment – and this phenomenon is particularly relevant for single-bottle adhesives such as One-Step. Therefore, Unterbrink and Liebenberg[4] recommended single-bottle adhesives to be used in combination with a radiopaque flowable composite as a filled adhesive. However, a microleakage study showed that the use of a flowable composite cured simultaneously with an adhesive yielded the worst results[31]. On this note, Frankenberger *et al.*[7] suggested that the single-bottle system could have completed hybridization prior to flowable composite application. The uncured dentin-resin hybrid layer might thus collapse if excessive pressure were applied during composite placement or seating of the restoration[32]. Hence, single-bottle adhesives should be irradiated properly prior to flowable composite application, although the entire adhesive layer might not be completely polymerized due to oxygen inhibition. It seemed that flowable composites of lower elastic moduli might compensate to an extent the polymerization shrinkage stress that occurred within an uncured adhesive layer during setting shrinkage of the resin.

For SBMP and OS used in this study, water occupying the interfibrillar spaces was lost through evaporation during air-drying after etching and rinsing, resulting in a collapse of the proteic network. As such, these DBAs need a careful management of the dentin moisture content, using the so-called “wet bonding” procedure to get optimum bond strength[33]. The self-priming systems OS and P&B differed in their composition but contained the same solvent, acetone, which might be extremely sensitive to the amount of water present on the dentin surface. On wet bonding, Kanca[34] also demonstrated that drying time and air syringe-to-tooth distance had a significant impact on shear bond strength.

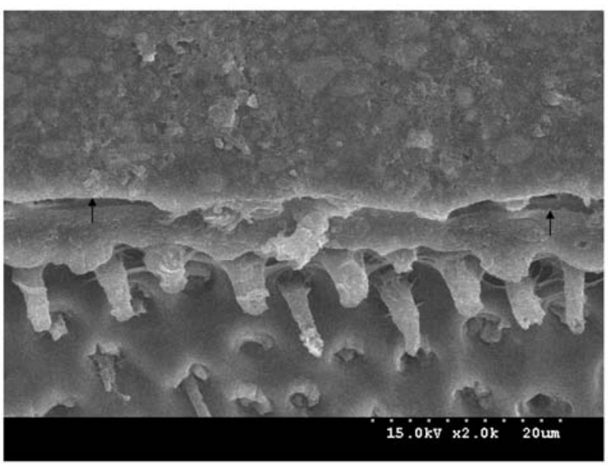

Fig. 1 SEM of interfaces bonded with Scotchbond-Multipurpose (Magnification: 2000×) after 1000 cycles. Well developed resin impregnated zone is seen, however, distinct gap between flowable composite and adhesive layer is detectable (arrow).

Gwinnett[35] showed that the total bond strength of resin-based materials to dentin was due to any or all of the following factors: resin tag formation, hybrid layer formation, and surface adhesion. However, according to Finger and Fritz[36], there was no correlation between the thickness of hybrid layer and bond strength nor was the formation of a hybrid layer the sole determinant of bond strength. In the present study, SEM examination (×2000 magnification) showed that SBMP was able to infiltrate the decalcified dentin layer but revealed gap formation between composite and adhesive layer (Fig. 1). Some reports have described factors that might influence bond strength: risk of collagen collapse during air-drying after etching and rinsing, as well as moisture control to prevent overdried[37] or overwet[38] dentin. In addition, there was an increased risk of making mistakes due to its number of application steps[22]. Therefore in pediatric dentistry, there is especially a need to develop adhesive systems with reduced and easy application steps, *i.e.*, sharing the same motivation that lies behind the development of flowable composites.

However, it must be pointed out that most conventional (three-step) DBAs have shown reliable bonding performance. Bouillaguet *et al.*[21] reported that despite a simplified bonding procedure offered by all-in-one DBAs, the conventional system produced higher bond strengths than most all-in-one self-etching DBAs. Further, a water-containing primer such as SBMP allowed rehydration of the collagen, thereby making resin infiltration easier and causing the DBA to be less sensitive to the moisture content of dentin, despite its disadvantage of a more time-consuming application procedure[22].

For OS, the SEM image showed a well-developed hybrid layer and long resin tags covered by a little irregular adhesive layer (approximately 20-30 μm) (Fig. 2). P&B showed nearly the same adhesive layer thickness but less developed resin tags (Fig. 3). For PLP, nearly uniform close contact with composite at the top of the adhesive layer was seen (Fig. 4). As for AQ, a thicker adhesive layer than PLP was observed, but so were voids and incomplete bonding between composite and adhesive layer (Fig. 5).

The failure modes that occurred were mostly adhesive fracture (67%) and mixed fracture (31%), with adhesive failures being more increased after thermocycling (Table 3). It was reported that at higher bonding strength, there was an increasing pattern of mixed failure[39], whereas Phrukkanon[40] reported that adhesive failure was consistently found with lower bond strengths.

In this study, bovine teeth were selected as substitutes for human teeth because of the latter’s

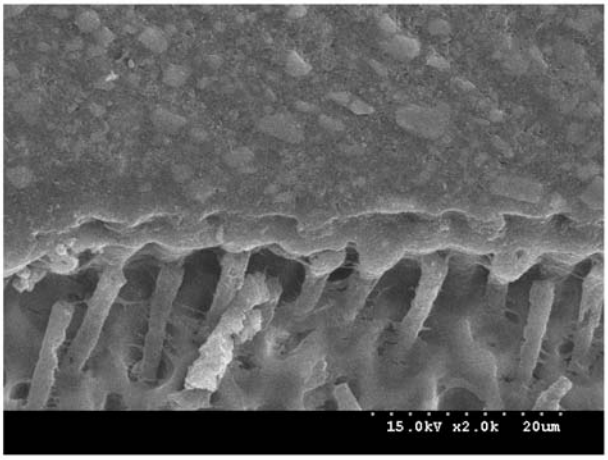

Fig. 2 SEM of interfaces bonded with One-Step (Magnification: 2000×), well-developed resin impregnated zone and approximately 20-30 μm thickness adhesive layers are seen. In some thin adhesive layer, flowable composite had reached to the hybrid layer.

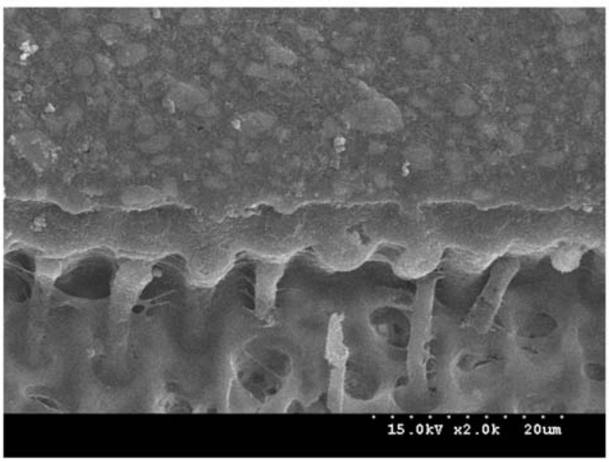

Fig. 3 SEM of interfaces bonded with Prime & Bond NT (Magnification: 2000×). Weak resin impregnated zone and approximately 20-40 μm adhesive layer were examined. Uniform close contact with composite at the top of the adhesive layer is seen.

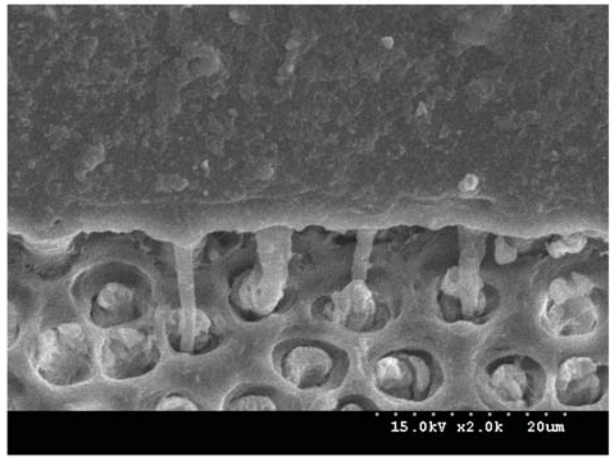

Fig. 4 SEM of interfaces bonded with Prompt L-Pop (Magnification: 2000×), 20-30 μm thickness of adhesive layer and hybrid layer with weakly infiltrated resin is seen. Nearly uniform close contact with composite at the top of the adhesive layer is seen.

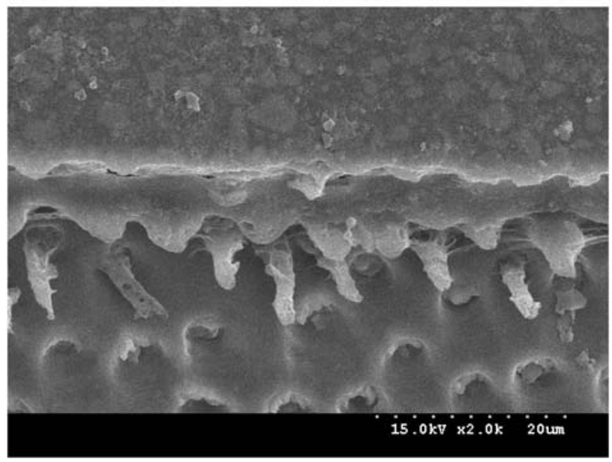

Fig. 5 SEM of interfaces bonded with AQ Bond (Magnification: 2000×) after 1000 thermocycling. Voids and incomplete bonding between composite and adhesive layer is seen, thickness of adhesive layer is nearly 40 μm above due to double coating in this adhesive.

scarcity. Schilke *et al.*[41] reported that when compared to human permanent teeth, significantly lower shear bond strength values for dentin adhesives were obtained when tested on bovine teeth. However, the collagenous matrix of bovine dentin is mainly Type I collagen, the same as human dentin.

In the present study, the flowable composite yielded lower shear bond strength values than expected, as the manufacturer presented shear bond strength of approximately 17 to 18 MPa when used with fourth- or fifth-generation DBAs on dentin surface. This discrepancy could be explained by differences in testing conditions, variable nature of dentin, and operational factors[42]. Furthermore, concerning the effect of operator variability with different types of adhesive systems, it was found that technique sensitivity was one of the most important variables that affected optimal bonding[43].

Results of the present study showed that bonding durability of the tested flowable composite was not determined only by the type of DBAs used, as characterized by their number of application steps. Therefore, it could not be conclusively said that simplified adhesives, such as all-in-one systems, were to be preferred for adhesion of flowable composites to dentin. It is thus recommended that the results of the present study be leveraged to conduct further *in vivo* or *in vitro* researches.

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