MICROTENSILE BOND STRENGTH OF RESTORATIVE COMPOSITE BONDED WITH SELF-ADHESIVE RESIN CEMENTS TO ENAMEL AND DENTIN

ABSTRACT

PURPOSE: To determine the microtensile bond strength (µTBS) of composite restorations when bonded with self-adhesive resin-cements. **METHODS:** Thirty caries-free extracted molars were sterilized, and divided into 5 equal groups according to adhesive used: SBMP (Scotch-Bond-Multipurpose, total-etch 3-step adhesive, 3M/ESPE), PAN (PanaviaF-2.0, resin-cement with self-etch primer, Kuraray), RXU (RelyX-Unicem, self-adhesive resin-cement, 3M/ESPE), BRZ (Breeze, self-adhesive resin-cement, Pentron) and MON (Monocem, self-adhesive resin-cement, Shofu). Each group was divided into 2 subgroups (dentin or enamel). Bonding agents, used according to manufacturers' directions, or a thin layer of resin cement was applied onto teeth flat surfaces. Six mm-thick Filtek-Z250 (3M/ESPE) composite build up was made in three increments. Teeth were sectioned to obtain rectangular specimens which were subjected to tensile force until failure. Specimens were subjected to 1,000 thermo-cycles between 5oC-55°C. Means and standard deviation (SD) were calculated and statistically-analyzed with ANOVA and Tukey's t-test. Specimens' failure modes were reported. **RESULTS:** SBMP showed the highest µTBS results with enamel (24.6(6.1) MPa), PAN showed high µTBS with enamel (12.1(3.9)MPa) and dentin (11.6(4.7)MPa) compared to the other self-adhesive cements. Failure modes were adhesive and mixed for self-adhesive resin-cements. MON subgroups and BRZ enamel subgroup underwent premature failure. **CONCLUSION:** self-adhesive resin-cements showed low µTBS compared to SBMP.

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INTRODUCTION

One major drawback of composite restorations is the tooth-restoration interfacial bond failure.[1] Microgaps at the interface permit the ingress of fluids and bacteria, and contribute to the development of recurrent caries and pulp damage.[2-4] Resin composite materials undergo volumetric polymerization shrinkage of about 2%.[5] Stresses generated by polymerization shrinkage of resin composites can reach values ranging from 13-17 MPa.[6,7] Such high resultant stresses can cause bond failure with the material pulling away from cavity margins during polymerization and subsequent gap formation.[8] In the posterior composite restoration, especially when margins are located subgingivally, the bond strength at the gingival margin is less than ideal and the gingival margin is particularly susceptible to marginal gap formation and bond failure.[9]

Different approaches have been developed to resist contraction stresses along the tooth-restoration interfaces during polymerization of large posterior composite restorations. These utilized different light polymerization techniques and strategic incremental placement techniques to reduce the C-factor.[7-11] Most importantly, applying an intermediate layer with low-modulus of elasticity at the cavity walls was reported to significantly relief the polymerization stress at the tooth-restoration interface.[12] Flowable composites were found to produce stresses similar to those of non-flowables, and they don't significantly reduce the stresses when used under non-flowable composites.[1]

Simplified self-adhesive resin cements have been designed for cementation of crowns, fixed partial dentures and inlays/onlays. Their effective bond strength and favorable marginal seal made them the materials of choice for indirect resin restorations.[13,14] Furthermore, by eliminating the etching and priming steps, usage of the self-adhesive resin cements is simpler and less prone to the technique errors associated with these steps. Their simplified bonding procedure is similar to self-etch bonding agents; however, self-adhesive cements are partially filled and would have improved mechanical properties over self-etch bonding agents. Moreover, self-adhesive cement provides a thicker tooth-composite bonding interface than an interface that was bonded using a self-etch primer. It is speculated that the self-adhesive cement interface could potentially better counteract polymerization shrinkage stress by viscous flow prior to its complete set and improve the stress distribution as an intermediary layer between the higher modulus dentin and composite substrates.

The use of a self-adhesive cement interface is comparable to the technique of placing resin-modified glass-ionomer or flowable resin composite materials at the

gingival margin of posterior restorations, which similarly offers the benefit of an intermediary elastic layer. The reported low-modulus of elasticity[15,16] and high mechanical properties[17-19] of the self-adhesive resin cements increase their potential for use as cavity liners for direct composite restorations.

The objective of this study was to determine the microtensile bond strength (µTBS) of composite restorations that are lined with self-etch or self-adhesive resin cements and to compare their bond strength relative to that of a conventionally bonded resin composite restoration (total-etch 3-step bonding agent). The null hypothesis was that there will be no significant difference in µTBS results among the different tested groups.

MATERIAL AND METHODS

Specimen preparation:

Thirty caries-free extracted human molars were collected from the maxillofacial surgery clinic at the Faculty of Dentistry; University of Toronto. Teeth were sterilized with gamma irradiation (Gamma cell 220, Atomic Energy Ltd, Mississauga, Canada). They were then cleaned with periodontal scalers and stored in water at 4°C. The teeth were pumiced and divided into five equal groups (n=6) according to the materials used for bonding at the dentin-composite interface: SBMP-control (Scotch-Bond-Multipurpose,

total-etch adhesive, 3M/ESPE), PAN (PanaviaF 2.0, resin cement with self-etch primer, Kuraray), RXU (RelyX-Unicem, self-adhesive resin cement, 3M/ESPE), BRZ (Breeze, self-adhesive resin cement, Pentron Clinical), and MON (Monocem, self-adhesive resin cement, Shofu). Then each group was divided into two subgroups (n=3) according to bonding substrate (dentin or enamel). Flat dentin and enamel surfaces were prepared to measure the µTBS of the assigned adhesive or cement material. In the dentin subgroup, a 3 mm-thick layer of occlusal enamel was removed under running water with a low speed micro-slicing machine (Isomet, Buehler, Lake Bluff, IL). The exposed flat dentin surfaces were wet-ground by means of a carbide bur (#245, SS White, Great White Series, Lakewood, NJ, USA) to prepare a surface similar to cavity preparation. In the enamel subgroup, buccal enamel surfaces were flattened by a carbide bur parallel to long-axis of the teeth to standardize the orientation of enamel prisms and similarly minimize the surface regional effects on the µTBS results.[20] After surface preparation, a thin layer of the predetermined adhesive or cement assigned to each group was carefully applied and cured with the Demi LED light polymerization unit (Kerr Corporation, Middleton, WI, USA, 1100-1200 mW/cm^2) following the manufacturer's instructions (Table 1). After completion of the bonding procedures, 2 mm horizontal increments of

composite were built up to a height of 6 mm on the bonded surface, and each increment was light cured for 20 seconds. The hybrid resin composite (Filtek Z250, 3M ESPE) was incrementally placed on the prepared surfaces of all specimens. The specimens were then stored in distilled water at 37°C for 7 days. The teeth were subjected to artificial thermocycling (NAAKE N2, Montgomery, TX USA) for 1,000 cycles between 5°C and 55°C.[21]

Microtensile bond strength testing & Evaluation of mode of failure:

After thermocycling teeth were sectioned perpendicular to the adhesive-tooth interface into 1 mm-thick slabs with the low speed micro-slicing machine. Then slabs were serially cut to rectangular specimens 1 mm^2 in cross section, according to the "non-trimming" method of the µTBS test (Figure 1). Twelve specimens were randomly chosen from each subgroup, and each specimen was measured with a digital caliper to confirm dimensions. Cyanoacrylate adhesive and accelerator (Zapit, DVA, Anaheim, CA, USA) were used to attach the specimens to opposing free-sliding halves, which were designed to fit the µTBS Instron universal testing machine (Bisco Inc. Schaumburg, IL). Specimens were then stressed until fracture occurred at crosshead speed of 1mm/min, and the force required to break each specimen was recorded. Microtensile bond strength was expressed in

MPa, as derived from dividing the tensile force (N) at the time of fracture by the bond area (mm^2). Then the two halves of each specimen were inspected by a single operator under a stereomicroscope at 40x magnification. The appearance of interfacial failure (adhesive layer on tooth, adhesive layer on composite surface and adhesive remnants on both sides) was categorized as an adhesive failure mode. When adhesive failure was accompanied by partial fracture of either one or both of the adherends, a category of mixed failure mode was assigned. Finally, the third failure mode category was cohesive, when the failure happened within dentin, enamel or composite and showed intact adhesive interface.

Figure 1: Illustration scheme showing specimen preparation for µTBS test. Preparation for dentin subgroups was conducted from step B through E, and for enamel subgroups from step F through I.
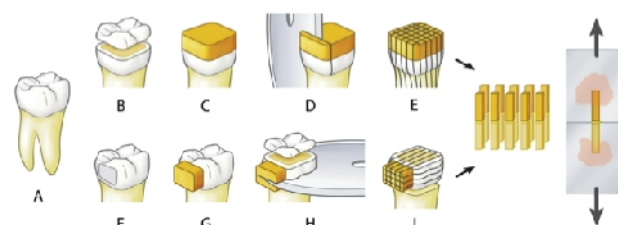
Scanning electron microscopy (SEM):

For SEM analysis, one fractured specimen from each of the dentin subgroups, already classified as adhesive failure, was chosen at random and allowed to dry overnight at 37º C with ascending ethanol solutions. The two halves of the specimen were mounted, fracture face up, on a 12 mm metal

SEM stub using cyanoacrylate adhesive. The surfaces were then sputter coated with gold (EMS-76M; Earnest F) and evaluated under a SEM at different magnifications. Photographs were taken and stored digitally (Figures 4-7).

Statistical analysis:

Means and SDs of the µTBS were computed using SPSS (PC+ version 15 software, Chicago, IL, USA). The µTBS results were analyzed using one-way analysis of variance (ANOVA) (p ≤ 0.05) at 95% confidence level. Each variable was investigated for significance in means using Tukey's t-tests (p ≤ 0.05) at 95% confidence level. Specimens that were exposed to premature failure were eliminated from the statistical analysis.

Table 1: Steps followed for materials application.

Materials (Manufacturers)	Etchant	Primer	Adhesive	Luting resin cement	Resin filling
Scotch Bond Multi-Purpose -control, *(3M ESPE, St Paul, USA)* Lot #20080516	37% phosphoric acid etching (15 s), water rinse (15s), air-dry (5s).	Apply primer and air-dry (5s).	Apply adhesive and light-cure (10s).	--------	Incremental application of resin composite restoration (Filtek Z250).
PanaviaF 2.0 *(Kuraray Medical Inc., Okayama, Japan)* Lot #61155*)*	-------	Mix equal amounts of A & B ED primer II, apply the mix, and wait (30s), gently air-dry.	---------	Mix equal amounts of A & B pastes (20s), apply thin layer of the mixture, Light-cured (20s).	Incremental application of resin composite restoration (Filtek Z250).
RelyX Unicem *(3M ESPE, St Paul, USA)* Lot # 330621	-------	------	------	Apply thin layer of the self-adhesive resin cement on all cavity surfaces with micro brush, Light-cured (20s).	Incremental application of resin composite restoration (Filtek Z250).
Breeze *(Pentron Clinical, Wallingford, USA)* Lot #165893	-------	------	------	Apply thin layer of the self-adhesive resin cement on all cavity surfaces with micro brush, Light-cured (40s).	Incremental application of resin composite restoration (Filtek Z250).
Monocem *(Shofu Dental Co., San Marcos,USA)* Lot #080118	-------	------	------	Apply thin layer of the self-adhesive resin cement on all cavity surfaces with micro brush, Light-cured (20s).	Incremental application of resin composite restoration (Filtek Z250).

RESULTS

After thermal cycling and during the preparation of the specimens, BRZ enamel subgroup specimens and both dentin and enamel MON subgroup specimens prematurely failed. A total of 74 specimens were collected for µTBS testing, data analysis and mode of failure evaluation. The number of specimens of each group, means expressed in MPa and SDs data for µTBS are shown in Table 2.

For dentin subgroups, the higher µTBS values were obtained by the control group SBMP, followed by the PAN, RXU and BRZ. For enamel bonding subgroups, the higher µTBS values were obtained by SBMP, followed by PAN, and RXU. The one-way ANOVA revealed a significant difference in µTBS among the dentin subgroups ($p < 0.001$). Table 3 shows the p-values of the Tukey's t-test for the dentin and enamel bonding subgroups.

Table 2: Means (MPa) and SDs of the µTBS of dentin and enamel subgroups. Specimens of MON subgroups and BRZ enamel subgroup underwent premature failure.

		Dentin			Enamel	
	n	Mean (MPa)	SD	n	Mean (MPa)	SD
SBMP	12	18.61	6.65	12	24.55	6.14
PAN	12	11.57	4.72	12	12.06	3.86
RXU	10	6.69	3.30	9	4.13	1.35
BRZ	9	4.02	1.88	0	--	--
MON	0	--	--	0	--	--

Table 3: p-values (Tukey's t-test) for the µTBS test subgroups. a. Underlined values are for the enamel subgroups while the non-underlined values are for the dentin subgroups.

Groups	p-values of all groups[a]			
	SBMP	PAN	RXU	BRZ
SBMP	--	0.036	0.000	0.000
PAN	0.000	--	0.299	0.037
RXU	0.000	0.001	--	0.855
BRZ	--	--	--	--

Mode of failure:

Most of the failures of the dentin bonding subgroups were adhesive then cohesive (failure of dentin or composite), and no specimens showed a mixed mode of failure. In the enamel bonding subgroups, most of the failures were adhesive then mixed; only a few cohesive mode of failure were observed. Percentages of failure modes in each subgroup are illustrated in bar charts (Figures 2 and 3).

DISCUSSION

The results of the study showed significant differences in the µTBS among the different tested groups, which refutes the null hypothesis. Gamma irradiation was used to sterilize the teeth of the current study as it has been shown to sterilize non-carious teeth effectively and without affecting the tooth structure prosperities.[22] It was also reported that gamma irradiation doesn't affect the bond strength to tooth structure.[23] Although 7 days storage in distilled water at room temperature of the samples of the current study is considered a brief period in comparison to the

life expectancy of composite restorations, it was deemed to be appropriate considering that cements are more susceptible to dissolution during, and immediately after their initial set than resin composites.[24] The rectangular shape of the specimens is considered popular in the µTBS studies as it showed higher bond strength compared to the hour-glass specimens. It has been reported that trimmed (hour-glass) specimens often exhibit fracture lines at the bur action area, which relatively lowers the bond strength at the specimen's interface.[25]

Figure 2: Bar chart showing the % distribution of different failure modes of dentin subgroups.
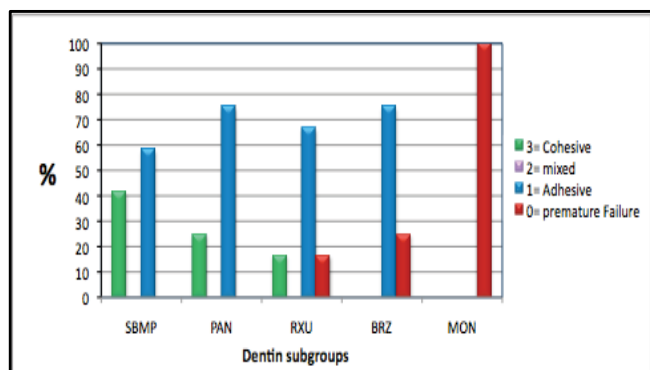

Figure 3: Bar chart showing the % distribution of different failure modes of enamel subgroups.
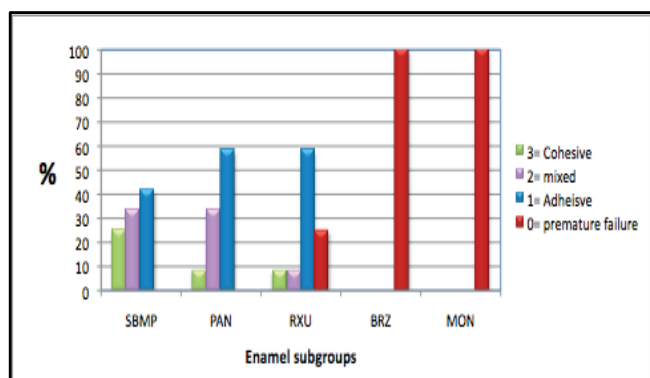

Figure 4: RelyX Unicem (3M ESPE) bonded to bur cut dentin, SEM photomicrograph of a fractured µTBS specimen viewed at angle of 90°. The specimen failed 100% adhesively, between the dentin and the cement. The photomicrograph shows cement remnants over the composite surface (R). Magnification: x5000.
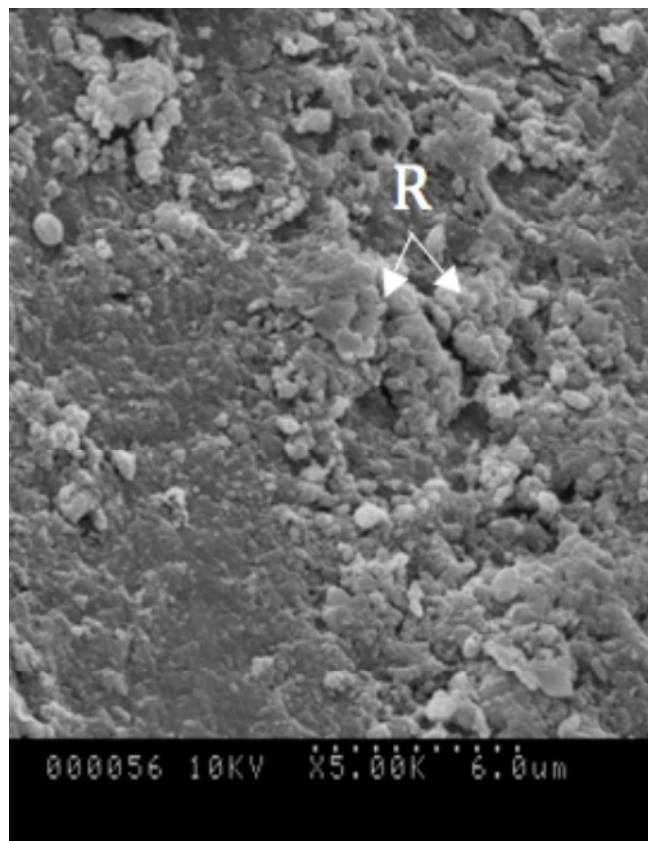
Figure 5: Breeze (Pentron) bonded to bur cut dentin, SEM photomicrograph of a fractured µTBS specimen viewed at angle of 90°. The specimen failed 100% adhesively, between the dentin and the cement. The photomicrograph shows the smear layer (SM). Magnification: x5000.
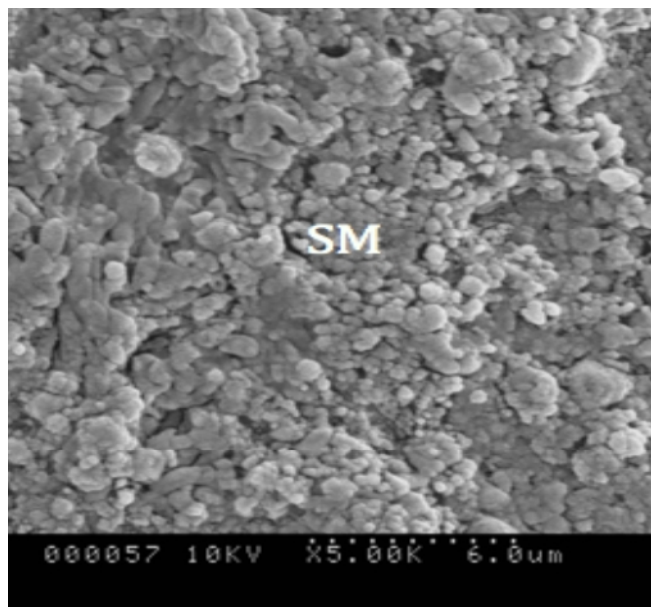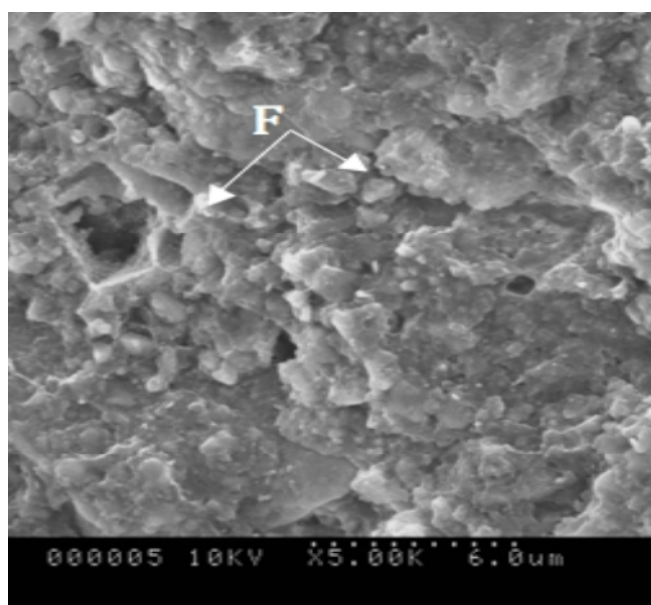

Figure 6: Panavia F 2.0 (Kuraray) bonded to bur cut dentin, SEM photomicrograph of a fractured µTBS specimen viewed at angle of 90°. The specimen failed 100% adhesively. The photomicrograph showing the cement is packed with large filler particles (F). Magnification: x5000.

Figures 7: Scotch Bond Multi Purpose (3M ESPE) bonded to bur cut dentin, SEM photomicrograph of a fractured µTBS specimen viewed at angle of 90°. The specimen failed 100% adhesively. The photomicrograph showing a typical example of completely removed smear layer after dentin surface conditioned with 35% phosphoric acid. Open dentin tubules and resin tags are visible. Therefore, failure occurred either on top of dentin or through the hybrid layer. (D) dentin surface; (DT) dentinal tubules. Magnification: x5000.
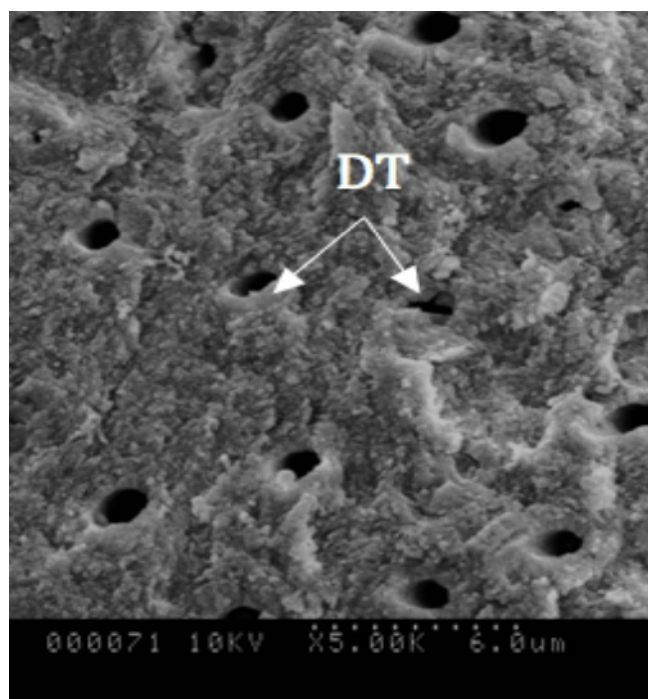
Studies in the literature reported the PAN and RXU high bond strength with the pre-polymerized resin composite blocks.[26-28] The relatively low µTBS of the self-adhesive resin cements used in the current study can be attributed to several factors such as their low acidity, different chemical composition, different functional monomers, and direct nature of the restoration that was used in the current study. SBMP showed the significantly highest µTBS (24.6(6.1) MPa) among the other groups (p < 0.05). BRZ and MON prematurely failed due to their weak bond strength to

enamel. The current study results are in line with many studies.[19, 26, 27, 29] It was established in the literature that there is significant correlation between the pH of the adhesive and enamel bond strength, indicating that the bond strength tends to increase as the acidity of the enamel conditioner is increased. On the other hand, Perdigao et al.[30] (2006) found that the enamel bond strength of the new self-etching, self-priming adhesive systems approaches the enamel bond strength of the total-etch (phosphoric acid) adhesive systems, which are gradually replacing the conventional total-etch systems. Du Munck et al.[27] (2004) reported that phosphoric acid treatment of the enamel surface prior to RXU application increased the µTBS to the same level as that of PAN. Additional layer application of the acidic primer and conditioning the surface with phosphoric acid was proven to improve the bond strength of the self-adhesive resin cements when bonded to enamel.[19, 26, 30] At the dentin interface significant difference in µTBS was found between SBMP and self-adhesive resin cements (p < 0.05), self-adhesive resin cements PAN, RXU and BRZ showed better bond strength with dentin than with enamel. Despite the initial low pH (2.1) of RXU,[15] an intimate adaptation and only a slight superficial demineralization of the dentin surface were observed during SEM morphological interface examination.[18, 29] Furthermore, the direct light-polymerization of

the material together with rapid decrease of the acidity (6 pH in 5 minutes)[15] may lead to limited penetration and interaction with tooth substrates. Other studies reported that complete dissolution of the smear layer and/or hybridization at the micrometer level was not possible.[29, 31, 32] In general, this limited micro-mechanical retention of the self-adhesive cements might be responsible for the relatively low µTBS to tooth structure measured in the current study. It has been found that the mild-acidic (pH=2.4) ED primer II/Panavia F2.0 produced minimal dentin demineralization, however resin penetration was found. The hydrophilic monomers (HEMA, 10-MDP, 5-NMSA), with low molecular weight, may have selectively diffused into dentin,[31] forming thin hybrid layer.[33] This might explain the higher bond strength of PAN compared to the other self-adhesive resin cements. Furthermore, phosphoric acid etching, prior to the application of RXU, has been shown to be detrimental to effective dentin bonding due to the thick, weak and exposed collagen layer that prevents the viscous cement to reach the deeper unaffected dentin. Using RXU with no phosphoric acid pre-treatment, however, gave substantial higher bond strength to dentin.[27]

The self-adhesive resin cements are composed of polyfunctional dimethacrylate-based monomers, such as BIS-GMA and/or urethane dimethacrylate, and inorganic filler of glass and silica. Due to their resinous content,

self-adhesive resin cements are expected to show lower solubility in the oral environment, despite the fact that they lose some of their contents when exposed to water.[34] Sano et al.[35] (1995) concluded that the impaired infiltration of the dental hard tissue by the high molecular weight functional monomer of the resin cement and the insufficient polymerization of the adhesive resin may lead to water diffusion at the interfacial aspect of the hybrid layer, and thus decrease the bond strength. MON showed premature failure of all specimens during μTBS testing. Although the basic adhesion mechanism appears similar for all self-adhesive cements, these materials are still relatively new, and detailed information on their composition and adhesive properties is very limited. These poor results of MON cannot be explained by the factors of polymerization shrinkage, pH, elasticity, and layer thickness. The composition of MON, most likely, plays a significant role in its poor performance in the current study. The fact that one operator who followed a standardized method conducted specimen preparations of the current study, strongly suggests that the reported premature failures should be ascribed to less effective bonding and not to manipulation errors.

Under the indirect restoration technique, although the C-factor is less favourable for the resin cement that is sandwiched between the dentin and overlying indirect restoration, the resin cement is subjected to minimal polymerization contraction stresses because there is no overlying resin composite material undergoing photopolymerization, It is possible that the slower-setting chemically-cured PAN was unable to resist the debonding forces caused by polymerization contraction of the subsequently placed resin composite material in the direct restoration situation but is able to maintain its bond to dentin in the indirect restoration situation. Moreover, all specimens in the current study were subjected to 1000 cycles between 5°C and 55°C, which is considered an adequate artificial thermal aging test according to the ISO TR 11450 standard (1994).[21] The coefficient of thermal expansion for Filtek-Z250 was reported as $41.5 \times 10^{-6}/°C$,[29] and $11 \times 10^{-6}/°C$ and $17 \times 10^{-6}/°C$ for enamel and dentin, respectively.[38] This mismatch of the coefficient of thermal expansion may apply stresses at the tooth-restoration bonding interface and allows a hot water percolation. Also, it has been reported that the warm water bath ($55°C$) may accelerate the solubility and hydrolysis of the component of the interfacial material (bonding agent) with subsequent water absorption and extraction of the broken down collagen or poorly polymerized resin oligomers.[39] Solubility is also related to the composition of the self-adhesive resin cement within the hybrid layer, as higher solubility and lower bond strength are attributed to a

lower concentration of hydrophobic monomers.[38]

The cement materials were more viscous than the adhesive materials and showed thicker intermediate layer than did the bonding agent groups, especially when applied under direct composite restoration, where the cement layer thickness most likely determined by the viscosity of the cement. The results of this study did not confirm the benefits of a thick intermediate layer at the tooth-composite interface. Indeed, a layer of resin cement would undergo a greater degree of polymerization contraction than an equivalent thickness of restorative resin composite. It is possible that other factors such as chemical composition or degree of polymerization contraction played a larger role in µTBS results than did the cement thickness or elasticity.

The main purpose of applying a low-stiffness intermediate layer is to partially absorb the composite polymerization shrinkage stresses. It has been reported that increasing the thickness with adding a second adhesive layer, will increase the bond stability, and thus, improve the bond strength at the dentin interface.[39] Coelho et al.[40] (2008) showed that the µTBS of the filled self-etch adhesives increases when the adhesive thickness increases. Van Meerbeek et al.[41] (1993) confirmed the positive effect of the elastic and low viscosity intermediate layer on the marginal adaptation and retention of the composite restoration. Braga et al.[7] (2004) also suggested that an elastic intermediate layer will increase cavity wall compliance and therefore decrease the destructive contraction stresses of the direct composite restorations. However, a thick intermediate layer with low solubility at the tooth-restoration interface can negatively affect the restoration durability.[42]

The significantly higher µTBS results of SBMP to dentin, might be attributed to the hydrophobic overlaying adhesive layer. It appears that the hydrophobic layer is important for the development of a strong and resistant bond to dentin. It is also likely just as important for enamel bonding. Self-adhesive resin cements have a low bond strength result to enamel because of the inadequate enamel etching.

In vitro investigations provide important information when evaluating biomaterials; however, they have limitations and do not replace clinical studies. The current study has some limitations that should be considered.

1. Dentin is heterogeneous, and differences in dentin depth, permeability, degree of mineralization and tubule orientation would affect microtensile bond results, Dentin variability would be further increased by experimental conditions such as storage, which could affect its physical and mechanical properties.

2. Standardization of composite build up over the flat tooth surface was difficult to standardize. However, all possible effort was made to produce a standardized specimen.

According to the current study findings, the following areas might need further investigation:

- The nature of bond between the tooth structure and the different adhesives.
- The morphology and the composition of the hybrid layer, which is formed by the self-adhesive resin cements.

The mechanical and physical properties of the self-adhesive resin-cements, including durability and solubility.

CONCLUSION

Within the limits of this in vitro study, it can be concluded that:

1. Self-adhesive resin cements showed low microtensile bond strength with both enamel and dentin, and may not be the ideal materials for bonding direct resin restoration.

2. Future investigation should be conducted to study the physical, chemical and mechanical properties of the self-adhesive resin cements.

ACKNOWLEDGEMENTS

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