RESEARCH ARTICLE

Tensile Bond Strength and Marginal Integrity of a Self-adhering and a Self-etch Adhesive Flowable Composite after Artificial Thermomechanical Aging

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Abstract

Aim: This study aims to compare the self-etch adhesive (SEA) and self-adhesive flowable composite (SAF) concerning tensile bond strength (TBS) and marginal integrity by microleakage (*μ*LK) test in deciduous molars after artificial thermomechanical aging.

Materials and methods: 120 extracted primary molars were collected. Sixty teeth were mounted for testing TBS. Teeth were restored using SAF ($n = 30$) and SEA-conventional flowable (CF) composite ($n = 30$) and subjected to artificial thermal aging. Half the teeth ($n = 15$) from each material were subjected to mechanical loading (SEA-TBS-L and SAF-TBS-L). The specimens with no-load (NL) served as control (SEA-TBS-NL and SAF-TBS-NL). Class V cavity prepared and restored with SAF ($n = 30$) and SEA-CF ($n = 30$) to test *μ*LK after thermal aging. The subgroups were as same as the TBS based on with or without mechanical loading (SEA-*μ*LK-L, SEA-*μ*LK-NL, SAF-*μ*LK-L, SAF-*μ*LK-L; $n = 15$ each). *μ*LK was determined by employing the dye immersion technique.

Results: Concerning TBS, there is a significant difference between SEA and SAF with load or no load. Concerning *μ*LK, there is a significant difference between the materials under loading and no difference was found when not mechanically loaded. Also, concerning both TBS and *μ*LK, a significant difference was observed between the load and no-load subgroups within each material.

Conclusion: SAF exhibited higher TBS than the SEA. Mechanical loading not only adversely affected the TBS but also increased the µLK of the compared materials.

Clinical significance: Restoring the primary teeth with SAF not only shortens the laborious operatory time but also yields good clinical serviceability with the good bond strength and minimal *μ*LK, thus preventing premature loss of teeth and consequential malocclusion.

Keywords: Adhesive, Artificial aging, Bond strength, Composite resin, Microleakage.

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Introduction

Adhesion is a complex set of physical, chemical, and mechanical processes that allow the attachment and binding of one substance to another dissimilar substrate. The goal and functions of a dental bonding adhesive system are to provide strong durable bond resisting separation of an adherend substrate from a restorative or cementing material so that there exists optimum retention, distribution of stress along with bonded interfaces, better color stability, and sealing the interface between dentin and/or enamel and the bonded material ensuring no or minimal microleakage (µLK). Hence, an adhesive system should assure decreased risk of postoperative sensitivity, marginal leakage, and secondary caries.[1]

Hitherto, without sacrificing the bonding efficacy of the adhesives, curtailing the bonding steps was being attempted.[2] Recently, all-in-one adhesives or one-step self-etch adhesives (SEA) have been evolved that combine etching, priming, and conditioning in one solution. Since solitary-bottle systems have a high affinity toward dentinal tubular water movement, they were the most favorable material for adhesion.[3] Prolonged mouth opening and maintaining the operatory field moisture-free during composite restoration procedures in young children are a challenge to every pediatric dentist. Moisture contamination during bonding and restorative procedures jeopardizes the bond strength and amplifies *μ*LK. This eventually and adversely affects the clinical longevity and service of a composite resin restoration. Conventional, total-etch, or SEA, systems are protracting and laborious in pediatric care. However, the introduction of self-adhesive flowable composites (SAF) bypassed both etching-bonding steps and dramatically decreased the chair-side time.

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Differences concerning the coefficient of thermal expansion between enamel/dentin and adhesives or restorative resins might induce degradation of the tooth-restoration interface. Therefore, artificial thermal aging of composite resin restorations ought to be employed for evaluating TBS and μLK to simulate changing intraoral temperature conditions. Nevertheless, teeth are subjected to occlusal stresses when they come in contact in both centric and eccentric positions.[4] A restoration in a class V Cavity would debond at the tooth-restoration interface leading to marginal disintegrity and μLK due to cyclic masticatory loads exerted.[5] Multifarious studies have been executed in the past concerning the shear bond strength of SAF composites on permanent teeth with hardly employing artificial aging protocols and barely adhering to testing guidelines.[6,7] Since there is a paucity of information about SAF composite on primary teeth under mechanical loading, the present study aims to compare SAF with SEA in terms of tensile bond strength (TBS) and μLK on primary molars that are subjected to artificial thermomechanical aging. There will be no difference in the tested parameters between the two composites with or without artificial mechanical aging is the null assumption.

Materials and Methods

The TBS testing was executed at Annamalai University, Chidambaram, and the μLK assessment was done at Vivekanandha Dental College for Women, Tiruchengode. The institutional ethical committee (Approval No.: VDCW/IEC/13/2015) approved the research. A self-etch adhesive (SEA; 3M ESPE Adper Easy one SEA, Deutschland GmbH, Germany), conventional flowable (CF) composite resin (Filtek Z350 XT, 3M ESPE India Ltd. Bangalore), and self-adhering flowable composite resin (SAF; Constic, DMG, Hamburg, Germany) were used.

A total of 120 intact human deciduous molars were extracted at the brink of exfoliation, decontaminated, and stored. The irregular radicular remnants were flattened 2 mm cervical to cementoenamel junction leaving the neat crown part. The teeth collection, utility, dimensions of the specimens/cavity, thermal aging, and testing protocols strictly adhered to ISO/TS 11405:2015 guidelines (International Organization of Standardization).[8] Exclusion criteria included carious teeth, teeth with extrinsic and intrinsic stains, enamel hypoplasia, and fractures.

The two main groups ($n = 60$ teeth per group) were determined by the materials to be tested. Teeth were randomly and equally allocated based on the testing methods employed [(TBS, $n = 30$)]; microleakage [(μLK, $n = 30$)]. The subgroups were subdivided based on the mechanical cyclic load application videlicet with load (L, $n = 15$) and without load (NL, $n = 15$). The materials (groups), testing parameters, mechanical aging, subgroups, and teeth distribution are tabulated in Table 1.

TBS Testing

Punch holes were drilled on the lingual surfaces with a No. 2 round bur for retaining the teeth within the autopolymerizing resin. The buccal surfaces of the teeth were ground flat using a diamond disk until the superficial dentin subjacent to enamel were exposed. The flat dentinal surfaces were finished with a 600 grit silica-carbide emery sheet under running water and air-dried to obtain a standardized smear layer. For SEA ($n = 30$), the flat dentinal surfaces were treated with the SEA-bonding agent and photopolymerized for the 20 s. A customized hollow metal split mold with 6-mm height and 4-mm diameter was positioned on the dentinal surface and then CF of thickness 2 mm was dispensed inside the mold. Surgical ligature wire was swiveled to form round tensile-loading provision. The swiveled end was secured into the 2 mm of unset material and photopolymerized for 20 s.[9,10] Remaining mold height was filled and cured incrementally (Fig. 1). The other thirty samples were restored with SAF composite using the same hollow metal split mold. SAF of 2-mm thickness was dispensed directly onto the dentinal surface and rubbed for 25 s with the brush supplied by the manufacturer. The tensile-loading provisions were placed as mentioned above and were photopolymerized for 20 s. The remaining mold height was filled and cured incrementally. Following polymerization, the metal mold was split and removed.

All the samples were stored in distilled water for 24 h. Thermocycling (SD Mechatronik thermocycler TC-4, GmbH; Feldkirchen-Westerham, Germany) was performed for 500 cycles between 5°C and 55°C for a dwell time of 30 s. For TBS-L, 5,000 sets of 90 N artificial vertical force at a rate of 1 Hz were administered on the plane resin surface adjacent to the tensile-loading provisions in a masticatory simulator (SD Mechatronik, Chewing simulator CS-4.8, GmbH; Feldkirchen-Westerham, Germany).[11,12] For TBS-NL, this step was eschewed. The tensile-loading provision was then engaged to the hook of the universal tester's upper jaw (UNITEK-94100; FIE Pvt. Ltd) and pulled (crosshead speed: 0.75 mm/min).

μLK Testing

Class V cavities were prepared with rounded outlines (3-mm length, 2-mm width, and 1.5-mm depth) with diamond bur (Mani Inc, standard; ISO 544/019;4.4/19.2, Tochigi, Japan) in airotor handpiece. This was followed by restoration with SEA-CF and SAF ($n = 30$ each) according to the manufacturer's instructions. After immersing in distilled water for 24 h, the samples were thermocycled. Subsequently, the pulp chambers were sealed with low-fusing compound and nail varnish was coated except for restorations and their margins.[9] The teeth specimens of μLK-L were temporarily mounted in autopolymerizing resin for the above-mentioned mechanical loading. For μLK-NL, mechanical loading was eschewed. Teeth were drenched in a 2% basic fuchsine for a day at room temperature. After discarding the basic fuchsine, the teeth were thoroughly rinsed, blotted, and cut through the midpoint of the restorations buccolingually. The cut sections were observed through a stereomicroscope (Fig. 2; Magnus, Olympus Opto Systems India Pvt. Ltd.,) to examine the dye infiltration. Table 2 describes the scoring criteria for the degree of μLK.[13]

The obtained data were processed for the statistical report (SPSS; Version 21.0; SPSS Inc., Chicago, IL, USA). The data

Table 1: Materials, testing parameters, mechanical aging, and subgroups with teeth distribution

Materials (Groups)	*SEA*				*SAF*			
Testing parameters	TBS		μLK		TBS		μLK	
Mechanical aging	L	NL	L	NL	L	NL	L	NL
Subgroups	SEA-TBS-L	SEA-TBS-NL	SEA- μLK-L	SEA- μLK-NL	SAF-TBS-L	SAF-TBS-NL	SAF- μLK-L	SAF- μLK-NL
Teeth (*n*)	15	15	15	15	15	15	15	15

(SAF: self-adhesive flowable; SEA: self-etch adhesive; TBS: tensile bone strength)

Fig. 1: Diameter of composite with loop for tensile loading

Fig. 2: Stereomicroscope

distribution concerning TBS was normal (Kolmogorov-Smirnov test; $p > 0.05$). For comparing the loaded and nonloaded TBS of material, paired *t*-test was employed. For the materials' comparison, an Independent *t*-test was exercised. For comparing the μLK, the scores of the specimens were presented as cross tables and subjected to Pearson Chi-square (χ^2) test. $p < 0.05$ was considered for statistical significance.

Results

The mean [± standard deviation (SD)] TBS of SEA-TBS-L, SEA-TBS-NL, SAF-TBS-L, and SAF-TBS-NL were 4.78 ± 1.02 MPa, 10.31 ± 1.46 MPa, 11.33 ± 1.09 MPa, and 13.82 ± 1.20 MPa, respectively (Table 3). SAF had greater TBS than the SEA composite resin ($p = 0.000$). Mechanical loading adversely affected the TBS for both SAF and SEA, wherein SAF was better than SEA ($p = 0.000$). Table 4 describes the χ^2 test for μLK. SAF had lesser μLK than SEA. Mechanical loading adversely affected the μLK for both the composites, wherein SAF was better than SEA ($p = 0.018$). However, there is no statistically significant difference ($p = 0.062$) between SAF and SEA when not mechanically loaded. Figures 3 and 4 show the μLK scores of SAF-μLK-NL and SAF-μLK-L, respectively, under a stereomicroscope.

Discussion

In this present *in vitro* study, the increased TBS and decreased μLK of Constic SAF when compared to SEA can be attributed to the compositional difference of the resin matrices. Constic SAF contains 10-methacryloyloxydecyl dihydrogen phosphate (MDP) as an acidic monomer. SEA contains 6-(methacryloyloxy)hexyl dihydrogen phosphate (MHP) as the acidic monomer. MDP possesses a lengthy and water-repelling spacer chain when compared to the MHP. MDP forms stable MDP-calcium complexes without reprehensible decalcification resulting in imperishable chemical bonding with hydroxyapatite.[14] Yoshihara et al.[15] concluded that the type and spacer group length of phosphate-based acidic monomers significantly affect the chemical interlinkage capacity with dental hard tissues. Nonetheless, the efficacious etching by MDP possessing a long decyl spacer group resulted in water-stable nanolayering and durable bonding. Not only the nanolayering of MHP was less intensive than MDP but also its calcium complexes were less stable than the MDP.[15]

Table 2: Scoring criteria for microleakage assessment

Score	*Dye penetration criteria*
0	None.
1	Dye penetration within ½ of occlusal or gingival wall.
2	Dye penetration extending to the end of occlusal or gingival walls.
3	Dye penetration through the gingival or occlusal wall to ⅓ of axial wall.
4	Dye penetration through the gingival or occlusal wall to ⅔ of axial wall.
5	Dye penetration throughout the axial wall.

SAF eschews the need for the application of adhesive bonding agents in a separate clinical step and hence, simplifying the procedure in pediatric dental care. Though Bektas et al.[13] have concluded that using SAF resin would increase the bond strength, other researchers obtained no significant difference in the μLK between CF and SAF.[16,17] Owing to SAF's novel technology, it increased the inquisitiveness to research further about its bonding durability. In this current research, irrespective of the materials compared, cyclic loading drastically decreased the TBS. Previous studies found no significant difference in the shear bond strength among the Vertise flow SAF and other conventional SEA systems.[18,19] In the current study, Constic SAF exhibited higher TBS than SEA either with or without cyclic mechanical loading.

Osorio et al.[20] studied the effect of load cycling and *in vitro* degeneration on resin-dentin bonds using self-etching primer adhesive and concluded that cyclic loading significantly compromised the bond strength. Belli et al.[21] studied the effects of load cycles on micro-TBS of dual-cure resin cement to dentin with all-in-one adhesive and concluded that load cycles significantly reduced the adhesion. Artificial mechanical aging was exercised to evaluate its deleterious effect on the bonding durability of the dental adhesives.[22,23] The TBS was affected by the mechanical aging in the present research is congruent with the erstwhile studies in the literature.

The decreased μLK of SAF shall be possibly attributed to hygroscopic expansion and comparatively less polymerization shrinkage. Acidic acrylic monomers in SAF exhibit increased water affinity when compared to conventional resin monomers

Table 3: TBS (MPa)—rows: Paired t-test; columns: Independent t-test

	Group: Mean ± SD	*Compared group: Mean ± SD*	*t-value*	*p-value*
Group: Mean ± SD	SEA-TBS-L: 4.78 ± 1.02	SEA-TBS-NL: 10.31 ± 1.46	10.162	0.000
Compared group: Mean ± SD	SAF-TBS-L: 11.33 ± 1.09	SAF-TBS-NL: 13.82 ± 1.20	6.570	0.000
t-value	14.437	5.481		
p-value	0.000	0.000		

(SAF: self-adhesive flowable; SEA: self-etch adhesive; TBS: tensile bone strength)

Table 4: μLK—rows: Paired t-test; columns: Independent t-test

	Group	*Compared group*	*χ^2-value*	*p-value*
Group	SEA-μLK-L	SEA-μLK-NL	24.000	0.000
Compared group	SAF-μLK-L	SAF-μLK-NL	15.000	0.000
χ^2-value	8.000	5.550		
p-value	0.018	0.062		

(SEA: self-etch adhesive; TBS: tensile bone strength)

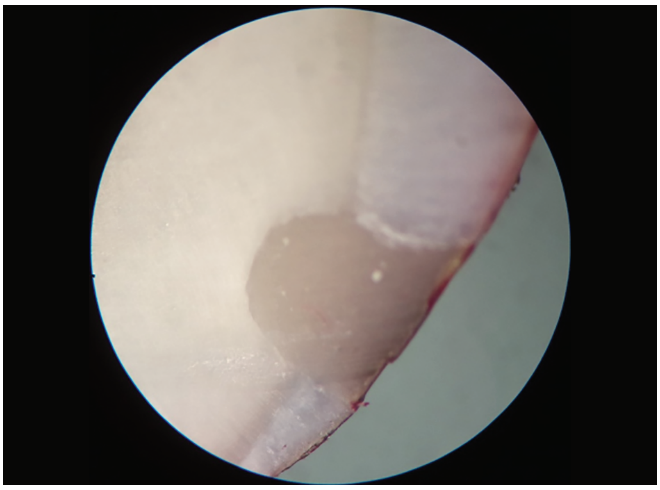

Fig. 3: Self-adhesive flowable (SAF)—μLK-NL with score 0 (40× magnification)

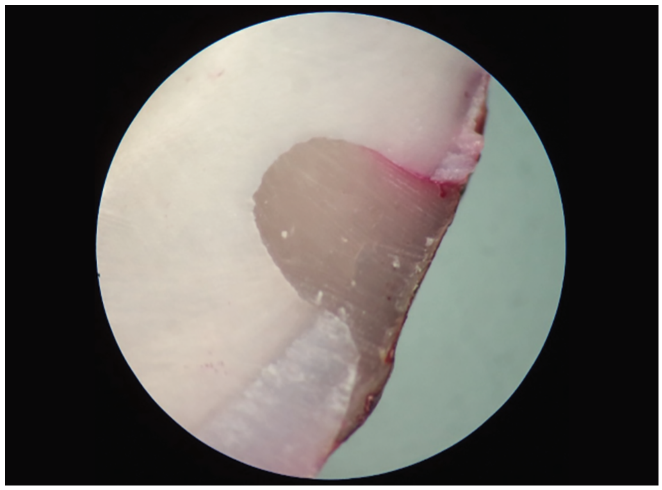

Fig. 4: Self-adhesive flowable (SAF)—μLK-L with score 2 (40× magnification)

resulting in hygroscopic expansion,[24,25] which in turn recompenses the polymerization shrinkage and yields a good marginal seal.[26] Nevertheless, the distinct polymerization-bonding process of SAF also explicates the μLK reduction. The synchronous bonding and restorative processes occur concurrently in SAF. Therefore, the association between the bonding and the photopolymerization stress is less.[27] On the contrary, CFs have a separate bonding process followed by a restorative phase that results in the development of residual stress during photopolymerization deteriorating the adhesive-tooth interfacial bonding and increasing the μLK.[28]

Some research demonstrated increased μLK due to cyclic loading[29-32] while some authors found that cyclic loading did not affect the marginal seal.[33-35] Molar restorations were drastically deteriorated by masticatory forces than cuspid restorations.[32] This may be attributed to the greater cavity's extent in the molars than the cuspids. The masticatory loads and mandibular excursions while chewing are complex and affected by a multitude of factors.[36] Nevertheless, the testing machine exerts axial forces, while the *in-vivo* chewing exerts a complex of both axial and oblique forces.[37] Hence, the ersatz analytical strategies do not simulate the in vivo scenario and must be contemplated while interpreting the results. Sachdeva et al.[38] evaluated the nanoleakage of conventional and SAF composites to dentin. It was concluded that the nanoleakage of both conventional and SAF composites was commensurable. SAF composites have combined properties of restorative composites and SEA monomers, thereby eschewing the adhesive application step and in turn, simplifying direct restorative procedures in children. In comparison, there were no differences in μLK between SAF and SEA composites without mechanical loading. However, in terms of loading, there was a significant difference between SAF and SEA composites. Hence, load cycling increased the μLK of the materials, which was by the previous scientific literature.

Therefore, from the above context, concerning TBS and μLK, the null assumption is rejected since there are significant differences in the TBS and μLK between SAF and SEA composites with and without artificial mechanical loading. *In vitro* simulations of the oral environment videlicet mechanical loading, thermocycling, the effect of pulpal pressure, and salivary effects are hardly achieved. This is because the *in vivo* bond breakdown factors are multifarious and incompletely identified. Therefore, further clinical trials are mandatory to obtain more validated, authentic, and proximal results.

Conclusion

Within the research constraints, it is concluded that the SAF composite resin had higher TBS than the SEA. Both the materials, when not mechanically loaded, exhibited μLK which was not comparable. Mechanical loading not only adversely affected the TBS but also increased μLK in the compared materials.

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