

Novel technique to increase adhesive bond strength

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ABSTRACT

Objective: The main aim was to evaluate the effect of postponing the curing of the adhesive layer until the first layer of composite resin is applied – hereby OIL formation and its detrimental effect on the DC of self-etch adhesives should be prevented. For this purpose, it was evaluated the degree of conversion and shear bond strength of four current market self-etch adhesives, assessing the effect of curing the adhesives anaerobically and then under two different thicknesses of composite resin, and compare this to the samples cured alone and in-air.

Materials and methods: The degrees of conversion were obtained by Fourier Transform Infrared spectroscopy, after the samples were prepared on a glass slide. The samples were either light cured in air or anaerobically under a clear matrix strip alone, under 2 mm of cured composite resin or under 4mm of cured composite resin. To determine the shear bond strength, extracted molars were halved, and set in acrylic. Prefabricated cured cylinders of composite resin (TPH 3, 2.4 mm in diameter) of two different lengths are placed over the adhesives under the following conditions: light cured conventionally (2 mm long cylinder), light cured anaerobically under the uncured end of the piece of composite resin (using both 2mm and 4mm long cylinders as separate treatments). After another incubation for 24 h at 37°C, the samples were subjected to shearing using the Bisco Shear Bond Strength Tester.

Results: The degree of conversion of the one-step self-etch adhesives was not statistically different when cured anaerobically under a clear matrix strip or cured anaerobically under 2mm of composite resin. These results were greater than those cured under 4mm. Shear bond strength between samples cured in air and anaerobically were similar under 2 mm of composite resin tubes, while those cured anaerobically under 4 mm of resin showed lower shear bonds strength.

Conclusion: When cured anaerobically, one-step self-etch adhesives show a greater degree of conversion and no significant difference in degree of conversion and shear bond strength when compared to those cured in air under the same thickness of composite resin.

Clinical relevance: The results obtained from DC and SBS analysis shows promise in placing the uncured adhesive under the composite resin and curing both the adhesive and restoration material simultaneously.

Key words

Self-etch adhesives, Degree of Conversion, Shear Bond Strength, Oxygen- inhibited layer.

INTRODUCTION

One-step self-etch dental adhesives do not require an initial separate etching step because they are composed of acidic monomers that eliminate the need for separate etching, conditioning and priming of the tooth surface [1-3]. This simplifies the bonding process. However, clinical and experimental knowledge on the long-term performance of one-step self-etch adhesives is currently limited [2, 4, 5]. One study showed that a one-step self-etch adhesive had a comparable clinical performance as an etch-and-rinse adhesive; however, the one-step adhesive showed increased marginal defects and discoloration [4].

When the adhesive is cured in air, the superficial layer of the resin is unhardened and tacky because oxygen inhibits its polymerization. It was previously believed that the oxygen-inhibited layer (OIL) was necessary for adding the restorative material, to improve the bonding and molecular interaction between the adhesive and the composite resin. However, studies have shown varying results on whether the effects of the OIL are desirable, benign or detrimental [6-8]. Self-etch adhesive monomers are also more acidic than their etch-and-rinse counterparts, and the effect of its OIL, when placed under composite resin, is not fully understood. One of the most recent studies on the effect of the oxygen-inhibited layer on the properties of dental adhesives has shown that 50% of the photoinitiator camphorquinone is decomposed after irradiation at 500 mW/cm² for 23 seconds [6]. It is currently thought that the adhesive's oxygen-inhibited layer is not necessary for adequate bonding to the composite resin.[6, 7, 9-12]

The degree of conversion (DC) of the bonding agents is used to predict clinical behavior of the adhesives. Low mechanical properties and an increase in the permeability of adhesives may be predicted by a low DC [5, 13, 14]. Additionally, the effect of the presence of an OIL is known to decrease DC values. Therefore, the current protocol for measuring the DC of adhesives

recommends using a Mylar polystyrene strip on top of the adhesive to prevent OIL formation [15-18]. Mylar strip is used because it simulates the clinical situation, where the first layer of the resin-based restorative material interacts with the outermost layer of the adhesive-displacing oxygen. However, the bonding agent inside the cavity is usually light-cured without any barrier for OIL formation, which results in an inconsistency between the published DC and the DC obtained from clinical procedures. The possibility of postponing curing of the adhesive until the first composite application would prevent OIL formation and consequently lower its interference on the DC obtained inside the cavity.

This study is aimed to investigate the possibility of a novel technique for avoiding OIL formation by applying of a composite layer before the adhesive's light curing process. This would simulate a technique for the placement of a restoration, where the self-etch adhesive would be left uncured before application of the first composite resin increment. Using this approach, the effect of the composite layer thickness on DC and shear bonding strength (SBS) of the four one-step self-etch adhesives on the current market was tested. The null hypotheses are: 1) DC and SBS are not affected if adhesive curing occurs after application of the first resin composite; and 2) the thickness of the composite increment does not influence the first hypothesis.

METHODOLOGY

Selection of Dentin Adhesives

Four ultra-mild adhesives: Scotchbond Universal Adhesive (SB) pH= 2.7, OptiBond All-In-One (OB) pH= 2.5, Prime and Bond Elect (PBE) pH=2.5 and iBond Self Etch (iB) pH= 2.2

were mixed according to manufacturer's recommendations. Their compositions are presented in Table 1.

Preparation of Degree of Conversion Samples

Two Dentoform teeth (46) were cut axially through the crown after a 9×5 mm rectangle (4 mm in depth) was drilled through the tooth, providing a matrix. In one crown, 2 mm of A3 TPH Spectra (Dentsply, Caulk, Milford, DE, USA) composite resin was applied and cured according to manufacturer's instructions, and 4 mm of the same composite resin was applied in the same way in the other dentoform tooth. The bonding agent samples (n=12) were prepared strictly according to manufacture's recommendations and light-cured in four different ways (Figure 1) on a glass slide as follows:

1) NS group: light-cured in air (without placing a mylar strip over the adhesive layer), to simulate clinical conditions; 2) S group: light-cured with a Mylar polystyrene strip (GC Epitex, CG America, IL, USA) over the adhesive layer; 3) S + 2 mm group: light-cured with the strip and less than 2 mm of pre-polymerized resin in the dentoform crown; and 4) S + 4 mm group: light-cured with the strip and less than 4 mm of pre-polymerized resin in the dentoform crown. The samples were light-cured using a light-emitting diode Valo Cordless tool (Ultradent Products Inc. South Jordan, UT, USA), the device intensity was 900 mw/cm^2 , which was measured using a radiometer (Model 100, Dementron Research Corporation, NY). The distance from the light-curing unit to adhesive layer was 4 mm. A curing time of 10 seconds was used according to manufacturer recommendations.

Evaluation of Degree of Conversion (FTIR Spectrometer)

Using FTIR analysis (Nicolet 6700 by ThermoScientific Waltham, MA, USA), samples underwent 32 scans between a frequency range of 4000 and 400 cm^{-1} , with 2 cm^{-1} resolution.

The DC was calculated using the following formula:

$$DC = 1 - \frac{\text{Area of band C = C (polymer) / area of band C = O (polymer)}}{\text{Area of band C = C (monomer) / area of band C = O (monomer)}} \times 100$$

The C=O and C=C carbon peaks were measured at 1720 and 1637 cm^{-1} , respectively, for all brands. An unpolymerized sample spectrum was recorded for each tested adhesive. Before identifying peaks, FTIR results underwent an auto-baseline correction in a pre-determined range.

Preparation of the Shear Bond Strength Samples

Molar teeth (extracted for orthodontic treatment) were sagittally halved and embedded into acrylic (Bosworth Fastray, Bosworth, IL, USA). In each hemi-tooth a central line was drawn for dividing it into two regions: mesial and distal. So, one tooth produced 4 distinct regions for bonding a tube of resin composite. In each region, a tube was bonded at the mesio third of dentin (~2 mm far from dentinoenamel junction). The surface was polished with sand paper 600 to reproduce a natural smear layer. The teeth were then incubated in distilled water at 37°C for 24 hours. Tubes of cured A2 composite resin (TPH 3 Spectra) were fabricated, with a diameter of 2.4 mm and a height of 2 mm or 4 mm. For each sample (n=12), a tube was bonded onto dentin using the respective adhesive. The control samples had the self-etch adhesive applied, then they were cured according to the manufacturer's instructions. A thin layer of composite was placed onto one end of the tube (2 mm) and then over the cured adhesive, the excess was carefully removed and the composite was light-cured according to manufacturer's instruction, under finger pressure. For the 2-mm and 4-mm samples, self-etch adhesive was not

cured before applying and curing the tube. After the tube was applied, all curing occurred under a light-blocking cardboard matrix, allowing light to penetrate only the top of the composite resin, mimicking light-curing during a clinical restoration. The samples were incubated again in distilled water at 37°C for 24 hours.

Evaluation of Shear Bond Strength

Each sample was subjected to shearing using the Bisco Shear Bond Tester (Bisco, Schaumburg, IL, USA), with a crosshead speed of 1 mm/min. The SBS was determined by converting the kilogram-force obtained, divided by the surface area of the tube bonded to the dentin surface. The kilogram-force/mm² unit was then converted to MPa. Tested samples were examined using a stereo microscope (Leica, Switzerland) and the fracture patterns were classified as: adhesive (along the dentin surface), cohesive (within the adhesive layer), or mix (in the combination of these two).

Statistical Analysis

Both the data from the SBS test and the calculated DC were analysed using a two-way ANOVA (factors: adhesives, treatments) and a Tukey's *post-hoc* test. For all analysis, statistical significance was set at $\alpha = 0.05$. Levene's test was used to detect the homogeneity of the variance.

RESULTS

Degree of Conversion

Table 2 shows the mean DC (%) of the four brands of cured self-etch dental adhesives, under four different treatments. ANOVA results show that the factor adhesive was not statistically different, while the factor treatment and the interaction between the two factors are significant different ($p < 0.0001$). A significant difference between the DC for self-etch dental adhesives undergoing different treatments can be seen in Figure 2. The greatest DC values are found in samples cured anaerobically under a clear matrix strip (81.82%); this value was not statically different from that of samples cured under 2 mm of composite (71.04%) but it was statically different when the composite thickness was 4 mm (42.47%). Samples cured without a clear matrix strip, and therefore in presence of oxygen, showed a lower DC (28.46%).

Figure 3 shows the interaction between adhesives and treatments. Treatments NS and S reflect the effect of OIL in the tested bonding agents. When a plastic strip (S) is used, according to the accepted protocol, all adhesives display a high level of DC.

Additionally, in Figure 3, three adhesives (SB, OB, PBE) had a reduction in the DC for treatment S + 2 mm, but DC values remained excellent (69.5%, 66.5% and 66.1%, respectively). However, IB showed a better performance under S + 2 mm (82.1%) than with the S treatment.

Shear Bond Strength

Table 3 shows the mean SBS (MPa) of the four brands of cured self-etch dental adhesives, under three different treatments. The average SBS between the brands showed no significant difference, except for Optibond, which showed significantly lower average SBS results. When comparing the mean SBS between the different treatments, no significant difference is seen between the aerobically cured control group and samples cured anaerobically under 2 mm of composite resin (Fig. 4). A significant difference was observed, however,

between these two groups and the samples cured anaerobically under 4 mm of composite resin. Figure 5 shows a trend towards greater SBS for all brands except for PBE in the 2 mm group. Both null hypotheses were rejected. Figure 7 shows SBS fracture patterns in percentage.

DISCUSSION

The aim of this study was to analyze the DC and SBS of a novel dental adhesive bonding technique that would improve the composite resin restoration process used by dentists today, and thereby reduce the chance of clinical error. Additionally, by providing an anaerobic environment for the adhesive to cure, a higher DC for the adhesive can be achieved, which lowers the fraction of unpolymerized resin monomers that would otherwise compromise the strength of the restoration. Using the conventional aerobic technique, Sakano et al. [19] demonstrated that OIL effects can reduce DC by 10 to 31% near the adhesive-resin joint. Low conversion rates can result in lower mechanical strength. Preventing OIL formation, this anaerobic technique would improve bonding strength.

Degree of Conversion

A lower DC is associated not only with lower mechanical strength, but also decreased biocompatibility and increased permeability, among other disadvantages [5, 19-22]. Figure 3 shows that less than 40% of the monomers become polymers when no strip is used. Additionally, Figure 3 reveals that oxygen differently affects the DC of each tested adhesive. For example, OB had the largest reduction in DC (79.5%) from treatment S to NS, and IB the lowest reduction (51.4%). This important finding can be used to explain DC results from *in vitro* tests and clinical performance of dental adhesives. When the accepted protocol (S) is followed, all test adhesive

had an excellent DC. However, the actual clinical DC values may be nearer to those found in the NS treatment, because the current adhesive technique does not prevent oxygen contact with the bonding agents.

FTIR results showed that anaerobic curing may increase the overall DC of self-etch dental adhesives by hindering oxygen inhibition. ANOVA followed by Tukey's *post hoc* test showed that the DC of the dental adhesives does not decrease significantly when cured under a 2-mm layer of composite resin ($p = 2.07 \times 10^{-6}$), replicating the first layer of restorative material applied by the dentist when restoring a tooth in the clinic. Even when cured anaerobically under 4 mm of composite resin, the DC of these samples shows greater polymerization than those samples cured aerobically (NS). The differences found between samples cured under 2 mm and 4 mm of composite resin can be attributed to the ability of the curing light to penetrate material and provide sufficient curing. The extent of polymerization can also be influenced by several factors [21], such as the resin's composition, photoinitiator concentration, exposure duration, and variability in the curing unit (light intensity), as well as its use, presence, and volume of the filler that can lead to the reflection of light. A resin layer up to 2 mm appears to be a safe thickness for near-maximum DC, while a 4-mm layer would be discouraged because it has an average DC of less than 50%.

Shear Bond Strength

SBS test results indicated that samples where the adhesive and composite resin were cured simultaneously had a greater SBS at a 2-mm thickness than the control, which used the conventional and manufacturer-recommended method of curing the adhesive before applying the composite resin. Simultaneous curing of the adhesive and composite resin may thus create an

anaerobic interface between the adhesive and the composite resin, thereby reducing oxygen inhibition of the polymerization process. The average SBS of all samples was weakest when the adhesive was cured anaerobically under 4 mm, which is expected because of the reduced DC that results from the small amount of light that penetrates through the greater thickness of the composite resin.

When each brand is compared per treatment (Fig. 5), there was a trend where the SBS was greatest in dental adhesive samples cured anaerobically under 2 mm of composite resin, followed by the control cured using the conventional method, and then anaerobic curing under 4 mm of composite resin. This observation was observed for all brands except for PBE, which showed a significantly lower SBS in the S + 2 mm treatment. PBE also had the lowest DC at S + 2 mm. OB showed the overall lowest SBS results, while the DC results were comparable to two of the three other adhesives. This may be because the OB is the only adhesive that contains fillers (Table 1). Although fillers are thought to fortify the resin, adhesive-containing fillers tend to have the worst SBS results [5]. Fillers used in adhesives may not be efficiently coated, or not coated, with silane, which would provide greater mechanical and physical interaction between the resin monomers and fillers. These fillers may act as porosities and compromise the SBS, and they may have greater use under compressive stress. The different solvent composition of the adhesives (Table 1) does not appear to affect the SBS results.

The SBS results of this study are overall significantly lower than those obtained in other studies. Studies show greater shear bond strengths obtained for OB and iB [23, 24]. Because the procedures used in this study are similar to those used in other shear bond strength tests, it is difficult to determine the reason that our results are different. It is possible that the cardboard matrix used to block excess light decreases the degree of cure of the adhesive, as other studies. In

one such study, Suh [6] used a gelatin matrix to place the composite resin on top of the adhesive. Further testing may be needed to determine the cause of the lower SBS, and micro-tensile bond strength testing could also be implemented to further characterize the bond between the one-step self-etch adhesives that are cured anaerobically under a layer of composite resin.

The manufacturer's recommendation for the self-etch adhesives in this study is to cure the adhesive first and subsequently apply and cure the composite resin. If the resin were to be applied before adhesive curing, an anaerobic environment may be created. This allows for a greater degree of polymerization and, in theory, allows for a stronger bond and better restoration characteristics. The results obtained from DC and SBS analysis shows promise in placing the uncured adhesive under the composite resin and curing both the adhesive and restoration material simultaneously.

Further investigation is needed to evaluate the performance of this technique. Some suggestions would be to use a thin layer of flowable light shade resin (rather than TPH A3), and to use conventional etch& rinse and multi-step primer/adhesive systems. Also, determining a method to evaluate the degree of cure of the adhesive and the composite resin when cured simultaneously will bring more light in this discussion.

CONCLUSION

This study showed that current one-step self-etch adhesives acquire a larger DC when cured anaerobically. Additionally, the DC is also clinically acceptable when the adhesive is cured anaerobically under 2 mm of composite resin. The SBS of the dental adhesives shows no significant difference when cured in air compared with those cured anaerobically by placing 2

mm of composite resin over the uncured adhesive, where the composite resin in contact with the adhesive is left uncured.

COMPLIANCE WITH ETHICAL STANDARDS

Conflict of Interest: MP declares that she has no conflict of interest. AH declares that she has no conflict of interest. RF declares that he has no conflict of interest.

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Ethical approval: This article does not contain any studies with human participants or animals performed by any of the authors.

Informed consent: For this type of study, formal consent is not required.

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Figures

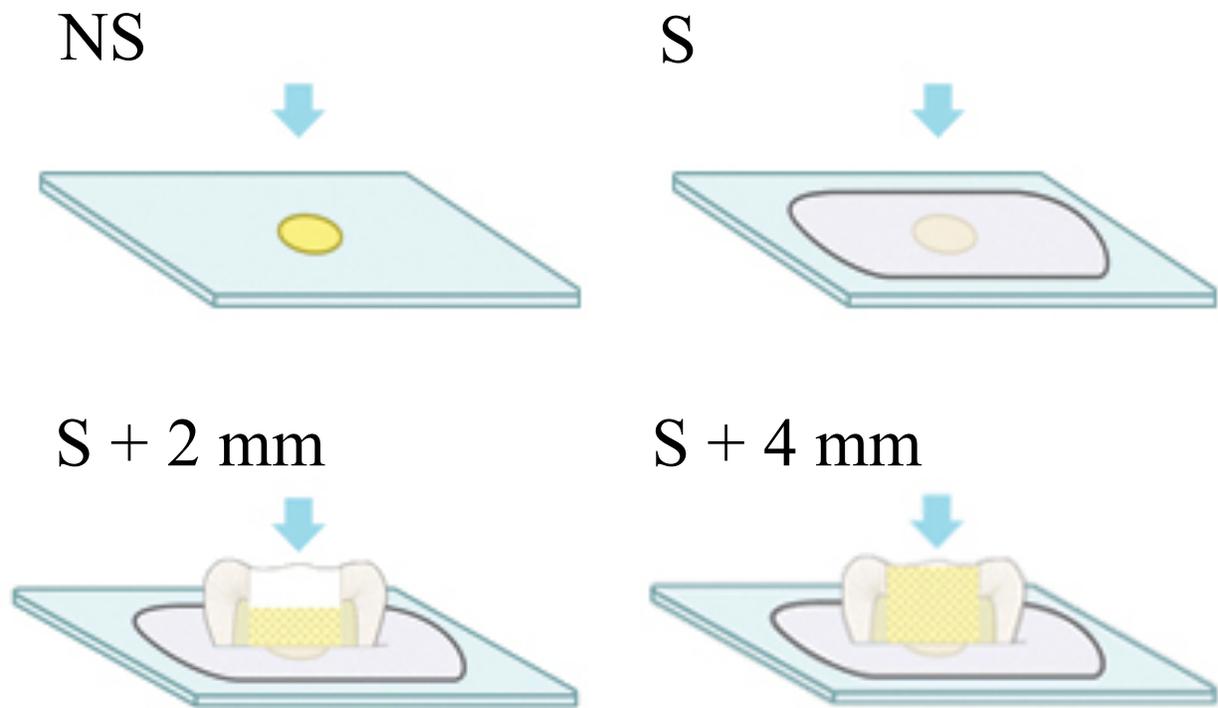


Figure 1. Schematic representation of the sample preparation, according to each group: NS) cured under clear matrix strip, S) cured under a clear matrix strip, S + 2mm) cured with clear matrix strip and 2 mm of cured composite resin, S + 4 mm) cured under clear matrix strip with 4 mm of cured composite resin.

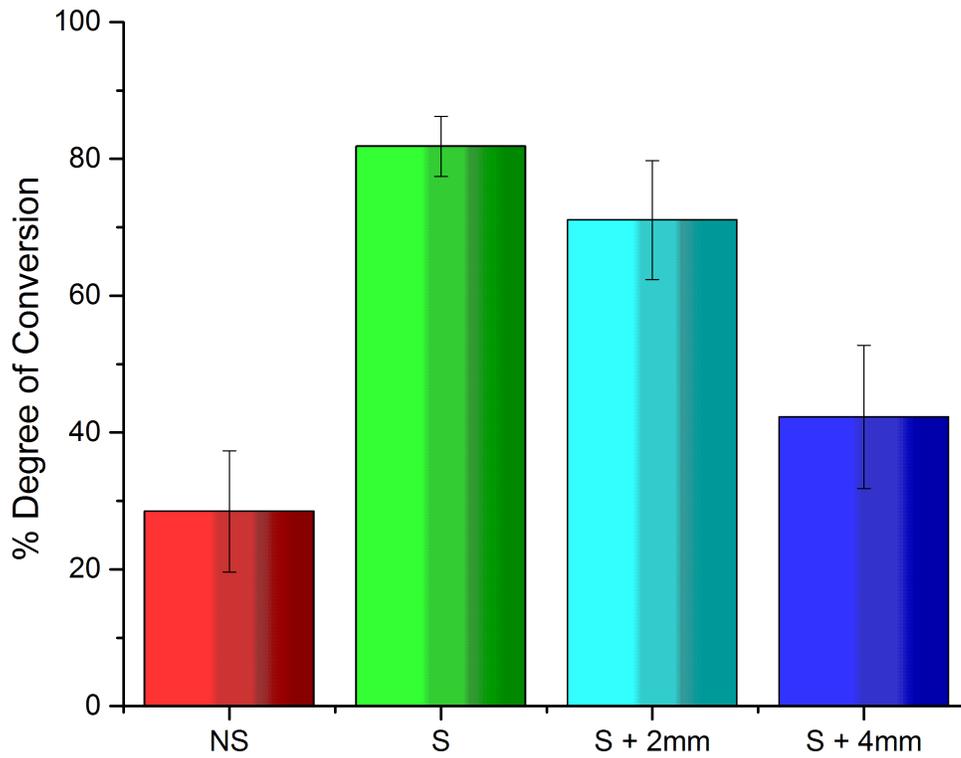


Figure 2. Mean DC (\pm SD) of all samples, categorized by treatment (n=12). Treatments: : NS) cured under clear matrix strip, S) cured under a clear matrix strip, S + 2mm) cured with clear matrix strip and 2 mm of cured composite resin, S + 4 mm) cured under clear matrix strip with 4 mm of cured composite resin.

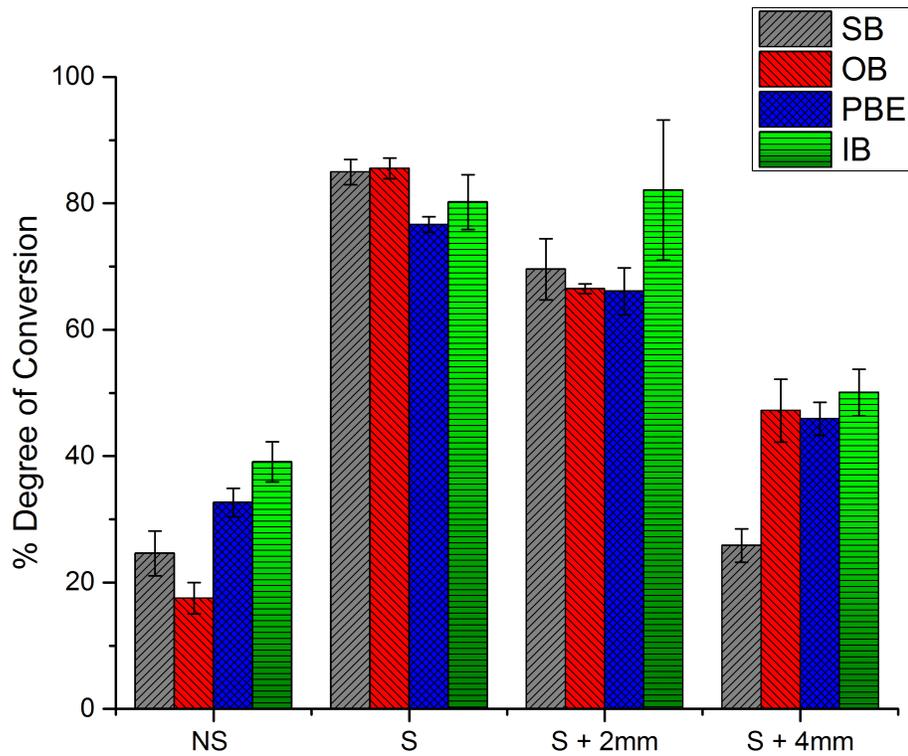


Figure 3. Mean DC (\pm SD) of all samples, categorized by brand and treatment. Treatments (X-axis): cured under clear matrix strip, cured under a clear matrix strip, cured with clear matrix strip and 2 mm of cured composite resin shade A2, cured under clear matrix strip with 4 of cured composite resin shade A2. Brands (legend): Scotchbond, Optibond, Prime & Bond, and iBond

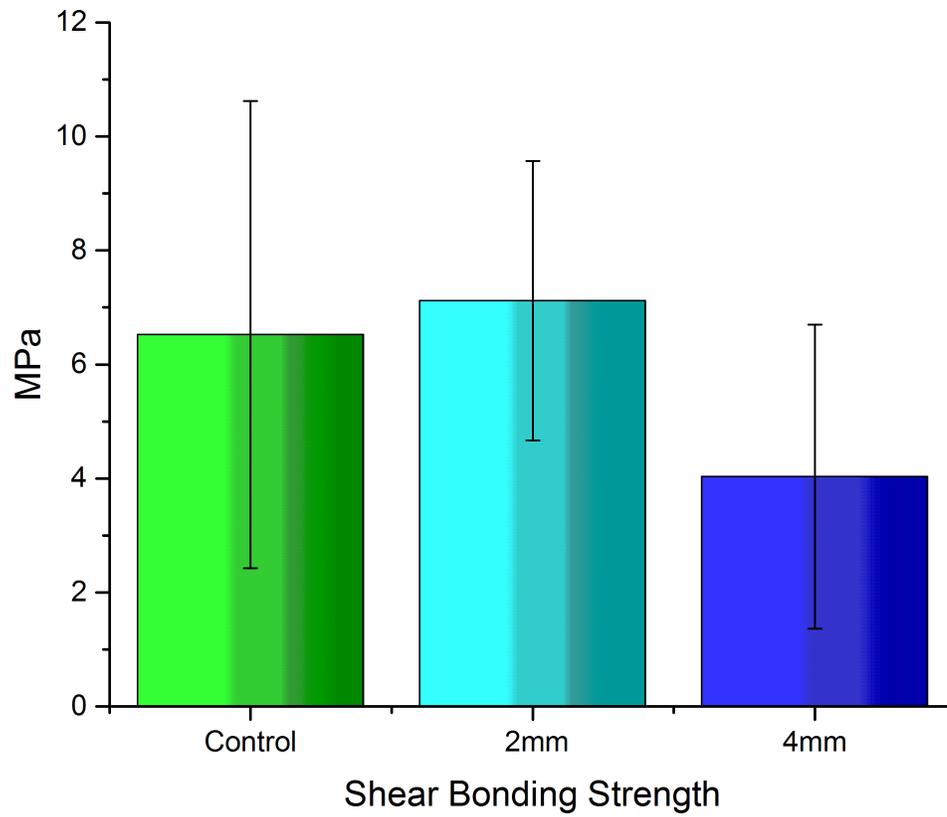


Figure 4. Mean SBS (\pm SD) of all samples, categorized by treatment (n=12). Treatments: cured conventionally under a 2 mm composite resin cylinder, cured simultaneously and anaerobically with a 2 mm composite resin cylinder, cured simultaneously and anaerobically with a 4 mm composite resin cylinder.

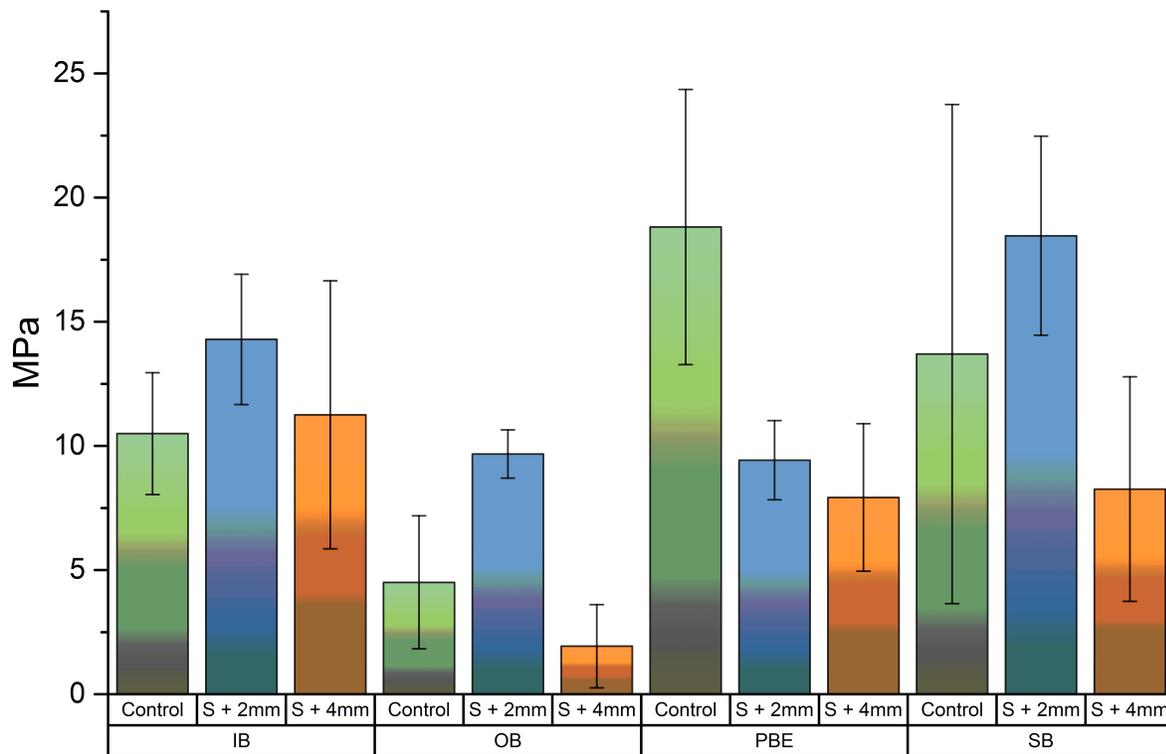


Figure 5. Mean SBS (\pm SD) of all samples, categorized by brand and treatment.

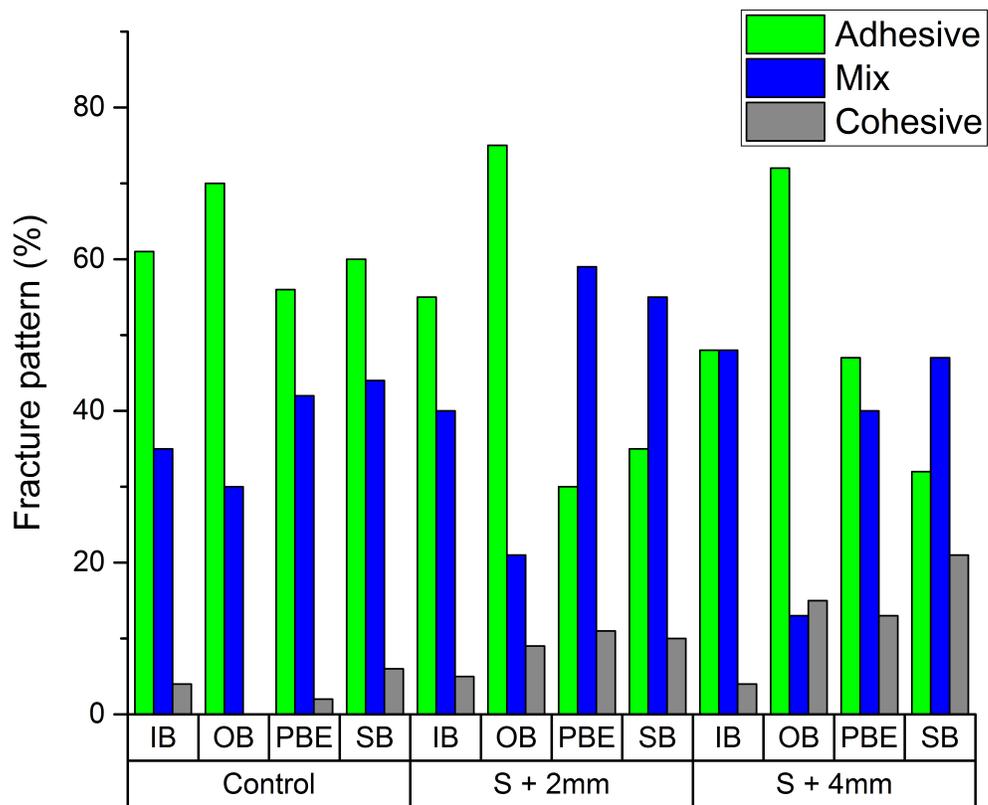


Figure 6. SBS fracture patterns in percentage, categorized by treatment and adhesive brand.

Table 1. Composition of one-step self-etch dental adhesives used in the study

Adhesive	Manufacturer	Composition	
		Solvent	Monomers and others
Scotchbond Universal	3M ESPE, St Paul, USA	Ethanol/water	MDP Phosphate monomer, DMA resins, HEMA, Vitrebond copolymer, filler, initiators, silane
Optibond All-In-One	Kerr, Orange, CA, USA	Acetone/ethanol/water	GPDM, co-monomers (mono- and di-functional methacrylate), CQ, fillers, sodium hexafluorosilicate, ytterbium fluoride
Prime and Bond Elect	DENTSPLY, Caulk, Milford, DE, USA	Acetone/water	MMA, DMA, TMA, PENTA, diketone, organic phosphine oxide, stabilizers
iBond Self-Etch	Heraeus Kulzer, Hanau, Germany	Acetone/water	UDMA, 4-META, glutaraldehyde, CQ, stabilizers

Data provided by the respective manufacturers.