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Effects of the light tip position on the degree of conversion and dentin bond strength of a universal adhesive

Утицај растојања и положаја светлосног извора на степен конверзије и јачину везе универзалног адхезива

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SUMMARY

Introduction/Objectives To measure degree of conversion (DC), immediate and long-term microshear bond strength (μSBS) to dentin of a universal adhesive relative to the light tip position and adhesive application protocol.

Methods Mid-coronal flat dentin of 48 human third molars was exposed and split in halves. Single Bond Universal (SBU; 3M) adhesive was applied to each half following 'total-etch' (TE) or 'self-etch' (SE) approach. Depending on the light tip (bluephase G2, Ivoclar Vivadent) angle and distance from adhesive surface, three groups were compared: "1 mm_90°" (control); "8 mm_90°" and "8 mm_60°". Cylindrical composite build-ups (ø1.7mm, Filtek Z250, 3M) were prepared in each half. DC was measured using Raman spectroscopy. μSBS was measured after 24 hours and six months storage in distilled water at 37°C. Fracture types were analyzed.

Results No significant difference in DC was detected between groups "1 mm_90°" (89.1 ± 6.2%) and "8 mm_90°" (94.6 ± 1.2%) ($p > 0.05$), both showing significantly higher DC ($p < 0.05$) than "8 mm_60°" group (74.9 ± 9.5%) ($p < 0.05$). Initially, there were no significant differences in μSBS between groups ($p > 0.05$). Group "1 mm_90°" TE (12.8 ± 4.3MPa) and group "8 mm_60°" TE (14.7 ± 5.7MPa) showed significantly lower μSBS after aging (8.4 ± 4.3MPa and 9.2 ± 2.6MPa, respectively) ($p < 0.05$). Adhesive fractures were predominantly detected.

Conclusion Initially, both application protocols resulted in similar bond strength to dentin of a universal adhesive in suboptimal curing conditions. In the long-term, SE showed greater adhesive resistance to degradation resulting in smaller decrease in bond strength compared to TE. Light tip angulation affected DC and μSBS more than tip-to-surface distance.

Keywords: adhesive; scotchbond; degree of conversion; bond strength; light

САЖЕТАК

Увод/Циљ Измерити степен конверзије (СК) универзалног адхезива и јачину везе за дентин смицањем (ЈВС) након 24 сата и шест месеци зависно од положаја светлосног извора и протокола апликације.

Метод Дентин у пределу екватора крунице 48 интактних хуманих умњака експониран је пресецањем дијамантском тестером, након чега је сваки зуб пресечен на пола. *Single Bond Universal* (*SBU; 3M*) адхезива је апликован на једну половину по '*total-etch*' (ТЕ) протоколу, док је на другој коришћен '*self-etch*' (СЕ) протокол. Према ангулацији и растојању светлосног извора (*bluephase G2, Ivoclar Vivadent*) од адхезива формиране су три групе: "1 *mm*_90°" (контрола); "8 *mm*_90°" и "8 *mm*_60°". Композитна надоградња (ø1,7 *mm*; *Filtek* Z250, *3M*) израђена је на сваком узорку. СК је мерен Раман спектрофотометром, ЈВС мерена је након 24 сата и шест месеци старења у дестилованој води на 37°*C*. Анализиране су врсте фрактура.

Резлтати Групе "1 *mm*_90°" (89,1 ± 6,2%) и "8 *mm*_90°" (94,6 ± 1,2%) су показале статистички значајно виши СК ($p < 0,05$) у односу на групу "8 *mm*_60°" (74,9 ± 9,5%) док између њих није уочена статистички значајна разлика ($p > 0,05$). Након 24 сата није било статистички значајне разлике у ЈВС између група ($p > 0,05$). Групе "1 *mm*_90°" ТЕ (12,8 ± 4,3*MPa*) и "8 *mm*_60°" ТЕ (14,7 ± 5,7*MPa*) показале су значајно нижу ЈВС након старења него иницијално (8,4 ± 4,3*MPa* односно 9,2 ± 2,6*MPa*) ($p < 0,05$). Адхезивне фрактуре су биле најзаступљеније.

Закључак: Иницијално оба протокола нагризања су показала сличну ЈВС. Након старења СЕ протокол се показао ефикаснијим у одупирању деградацији хибридног слоја што је резултовало већом ЈВС него ТЕ протокол. Ангулација врха лампе је имала више утицаја на СК и ЈВС него раздаљина од површине адхезива.

Кључне речи: адхезив, степен конверзије, јачина везе, полимеризација, удаљеност

INTRODUCTION

Over the past few years, "universal adhesives" also known as "multi – mode adhesives" have been implemented in the dental practice. According to manufacturers, one of the most important benefits of universal adhesives is their versatility, as indications for use include bonding to tooth tissues but also to materials for indirect restorations, zirconia, glass-ceramics, and alloys. Furthermore, universal adhesives are recommended with any of the

three currently accepted protocols for application to dental tissues: total-etch (TE), self- etch (SE) and selective - etch.

Two recent reviews concluded that mild universal adhesives yield the best results following a selective-etch protocol *i.e.* when adhesive is applied to previously acid etched enamel and un-etched dentin [1,2]. Dentin etching with phosphoric acid as used in the TE protocol may improve bonding of ultra-mild (2) and intermediately strong universal adhesives [1], *i.e.* those with pH of >3.0 and pH ≤1.5, respectively [1] compared to the SE protocol.

Enamel etching improves immediate and long-term bond strength of universal adhesives [1]. The same beneficial effect of enamel etching with phosphoric acid is evident for universal adhesives, as was previously seen with other adhesive groups [3]. As would be expected, long-term bonding to dentin is not so consistent and depends on adhesive pH [1]. Mild adhesives with pH between 2 and 3 showed greater degradation resistance irrespective of the application protocol than ultra-mild and intermediately strong which were associated with significantly reduced bond strength after aging [1]. Dentin etching has shown a detrimental effect on adhesive bond strength of several universal adhesives [4,5]. Collagen degradation and resin hydrolysis were associated with biodegradation of universal adhesives and the resulting decrease in bond strength to dentin [4].

It is widely accepted that curing regimes, characterized by such factors as light irradiance, curing time and distance, affect curing efficiency in all light-cured materials. Light energy is directly related to the degree of conversion (DC) of light-cured resin-based materials, as shown on a model BisGMA/TEGDMA composite [6]. In clinical conditions, curing characteristics, especially the distance and tilt angle of the light tip from the material, may vary considerably, thus influencing light energy delivered to the material. Apart from various clinical conditions hampering an ideal position of the light source, Price showed

differences between irradiance delivered to restorations in two groups of clinicians, and also the importance of light curing instructions to achieve optimal polymerization [7]. A study of light energy transfer using a MARC patient simulator revealed that as much as 31% of light energy was attenuated at a tilt angle of 20°. A wide difference in the amount of energy transferred to the material was seen with increasing distances of the light tip, this difference being highly dependent on the light-curing unit [8]. Other factors influencing the amount of delivered light energy in a clinical setting include inter-incisal opening, cavity location and operator experience with up to 17% difference between operator groups and 32% difference between anterior and posterior cavities [9].

Previous studies reported conflicting results on bond strength to dentin of adhesives following different application protocols [10,11]. The differences could be related to multiple factors, such as the type of dentin, adhesive composition, application protocol, light-curing unit, and/or bond strength testing methods. Current literature lacks data on the effect of variable curing conditions, namely tip-to-surface distance and angle, on the DC, immediate and long-term bond strength to dentin of universal adhesives.

Therefore, the aim of this study was to measure the DC, immediate and long-term microshear bond strength (μSBS) of a universal adhesive to dentin depending on curing distance and angle of the light tip. The null hypotheses were: (1) there are no significant differences in DC of a universal adhesives cured at different tip-to-surface distances and angles of the light-curing unit; (2) there are no significant differences in μSBS to dentin of a universal adhesive cured at different tip-to-surface distances and angles of the light-curing unit and (3) there are no significant differences in the μSBS to dentin of a universal adhesive following artificial aging.

MATERIALS AND METHODS

Specimen preparation and bond strength testing

Forty-eight intact human third molars, extracted for orthodontic reasons were used in this study. Ethical approval was granted by the School Ethics Committee to use such teeth for research purposes. Following extraction, the teeth were cleaned from debris and refrigerated at +4°C in 0.2% thymol until the beginning of the experiment.

Teeth were embedded in gypsum up to the enamel-cementum junction. Enamel and superficial dentin were removed to expose mid-coronal flat dentin of each tooth. A 2 mm deep notch was made parallel to the long axis using a slow-speed diamond saw (Isomet 4000, Buehler, Lake Bluff, IL, USA), thus splitting the exposed dentin surface into two halves. All surfaces were inspected using a magnifying glass to ensure the absence of residual enamel.

The materials used in this study are shown in **Table 1**. SBU adhesive was applied to one dentin half following the TE and the other dentin half following the SE approach **(Figure 1).** In the TE protocol, dentin was first etched with 37% phosphoric acid for 15 s, rinsed and blot-dried. In the SE protocol, no acid etching was performed on dentin. SBU was applied to dentin for 20 s, according to manufacturer's instructions. Depending on the position and distance between the light tip and adhesive surface, prepared specimens were allocated to three groups (N=8/group) **(Figure 1)**.

After adhesive polymerization, a silicone mold (ø1.7 mm x 2 mm) was placed on dentin and filled with the micro-hybrid composite (Z250) to produce cylinder composite build-ups with 2.27 mm^2 of adhesive surface area. In each group composite was light cured for 20 s at 1 mm distance using a high-intensity LED light-curing unit (bluephase G2, Ivoclar Vivadent, Schaan, Liechtenstein). Before μSBS testing, half of the teeth in each group were stored in distilled water for 24 h and the other half was stored in distilled water at 37°C for six months.

Irradiance of the LED light-curing unit was measured using a radiometer (Bluemeter II, Ivoclar Vivadent, Schaan, Liechtenstein). A metal foil with an 8x8mm window was placed on the radiometer sensor. The light tip was placed and oriented against sensor according to corresponding group.

A universal testing machine (PCE-200, PCE Group, Southampton, UK) was used for μSBS testing at 1 mm/min cross-head speed until specimen fracture. μSBS (*MPa)* to dentin was calculated by dividing maximum force at fracture (*N)* with bonded surface area (mm^2). Fracture types were analyzed under a stereomicroscope at x30 magnification and classified as: (1) adhesive - fracture occurring within the adhesive layer with no composite or dentin involved; (2) cohesive - fracture occurring within either composite of dentin and (3) mixed - fracture involving areas of adhesive layer extending into composite and/or dentin.

Raman spectroscopy

The Raman spectra were recorded at room temperature with a DXR Raman Microscope (Thermo Scientific, Waltham, MA, USA). The samples were excited by the 532 nm emission line of a diode laser with 10 mW of power focused on a 2.1-μm spot on the surface of the sample using an objective magnification of 10×. The scattered light was analyzed by the spectrograph with a 900 lines mm^{-1} grating. The spectrum was obtained as an average of three measurements on different places on the sample surface (10 exposures, 30 s each, per spot). All the Raman spectra were corrected for fluorescence by the OMNIC software (Thermo Scientific).

The DC was calculated using the following formula: DC = (1−R*cured*/R*uncured*) x 100 where *R* is the ratio of peak heights at 1639 cm^{-1} and 1609 cm^{-1} in cured and uncured material which was used as reference. The 1639 cm^{-1} peak in the Raman spectrum is

associated with the aliphatic C=C double bonds whilst the 1609 cm^{-1} peak is associated with the aromatic C=C double bonds in cured/uncured material.

Statistical analysis

Data were statistically analyzed in the software package Minitab 16 (Minitab Inc., State College, PA, USA). Two-way analysis of variance (ANOVA) was used to test the effects and interaction of the factors 'application protocol' and 'curing regime'. Intragroup comparison was done using paired t-tests. The level of significance was set at α=0.05.

RESULTS

Light irradiance depending on the light-curing tip position is presented in Table 2. About 86% and 74% of the maximum irradiance observed in the control group was recorded in the 8mm_90° and 8mm_60° groups, respectively.

Regarding DC, no significant difference ($p>0.05$) was detected between control (89.1%±6.2%) and "8mm_90°" group (94.6%±1.2%). Those two groups showed significantly higher DC ($p<0.05$) than "8mm_60°" group (74.9%±9.5%) ($p<0.05$) **(Figure 2)**.

Regarding μSBS, no significant differences in μSBS of the tested groups were detected initially ($p>0.05$) **(Figure 3)**. After aging, control TE group (8.4±4.3 MPa) and "8mm_60°" TE group (9.2±2.6 MPa) showed significantly lower μSBS than "8mm_60°" SE group (12.6±4.2 MPa) ($p<0.05$). Generaly all initial μSBS values were higher than those after aging irrespective of the application protocol. Nevertheless, greater differences between initial and long-term bond strength in all tested groups were associated with the TE protocol. These differences reached statistical significance in the control group and "8mm_60°" group ($p<0.05$).

In all groups, predominantly adhesive fractures were detected **(Figure 4).** It is worth noting that the percentage of adhesive fractures increased and mixed fractures decreased in

the aged specimens compared to those tested initially. This increase in adhesive fractures was more extensive in groups following the TE than SE protocol.

DISCUSSION

All three tested hypotheses were rejected. At least one tested group showed significantly different DC (H_1). Although no differences were found between groups initially, long-term bond strength values of at least one group differed significantly compared to other curing conditions (H_2). Aging resulted in significantly lower bond strength values in two of three tested groups associated with the TE protocol compared to initial values (H_3).

SBU, a universal adhesive containing 10-MDP in its monomer mixture, was used in the present study due to its wide use in clinical practice. 10-MDP bonds chemically to the residual hydroxyapatite, thus creating secondary chemical bonding (nanolayering) in addition to micromechanical interlocking [12]. The influence of nanolayering between 10-MDP monomer and hydroxyapatite on adhesive-dentin bond strength and its longevity is questionable, especially in commercial adhesives, due to the scarcity of this phenomenon at the adhesive-dentin interface [13].

In everyday clinical practice, dentists may not achieve a minimal distance or perpendicular light tip position due to cavity depth, light-curing unit design or curing malpractice resulting in large variations of the energy of the light source delivered to the material and its photo-initiators [9]. With this in mind, two different distances (1 mm and 8 mm) and two different positions between the light tip and tooth surface (90° and 60°) were selected in order to examine if similar results can be achieved with less than ideal curing conditions. A high-intensity polywave light-curing unit was chosen as these units are recommended for photocurable materials containing both camphorquinone and alternative

photoinitiators due to emission spectra compatible to absorption spectra of various photoinitiators [14].

The lowest light irradiance was detected in the 8mm_60° group, about 25% reduction compared to the control maximum irradiance. Irradiance was also reduced, by about 15% in the 8mm_90° group, but still remained above 1000 mW/cm^2. Irradiance indicates radiant power (flux) incident on a known surface area and is expressed as an average value over the surface area. The difference in irradiance between 8mm_60° and 8mm_90° groups clearly shows the adverse effect of light tip tilting which disrupts the beam profile against the irradiated surface. Light tip tilt causes inhomogeneous incident light beam reaching the irradiated surface resulting in spots with higher and lower radiant power.

Shear load was employed instead of tensile because of the complexity of experimental design i.e. dentin halves were used for different application protocols. The current test may be considered μSBS as it was stated by Van Meerbeek et al. [15] that "macro" bond tests are those with bonding areas exceeding 3 mm^2. Bonding area in the present study was 2.27 mm^2, larger than usual 1 mm^2, to mitigate a potential adverse effect of a thicker adhesive layer on very small composite build-up diameter resulting "in considerable bending and variable and non-uniform loading conditions" [15]. Although microtensile bond strength (μTBS) seems to be the preferred testing method [16], shear bond strength (SBS) or μSBS were also used in previous studies involving universal adhesives, mostly on enamel [17,18] and less frequently on dentin [19]. Furthermore, Bracher&Özcan [20] reported no significant differences in adhesion to dentin between testing methods.

In line with irradiance recordings, the results of DC measurements indicated that 60° tilt of the light tip had a more detrimental effect on conversion than 8 mm tip-to-surface distance. The latter actually showed slightly higher mean DC values compared to the control group, albeit with no statistical significance. These findings may be related to lower light

energy delivered to the material with the tilted light tip, whilst the effect of distance was dependent on the light-curing unit, as suggested in a previous study [8]. Somewhat lower DC in the control ("1mm_90°") than "8mm_90°" group could be associated with greater solvent evaporation due to higher temperatures generated in the adhesive when the light tip was held at 1 mm than 8 mm distance. It has been recently shown that a high-intensity LED light-curing unit exhibits about 10°C lower temperature at a distance of 4-5 mm compared to the temperature at the light tip [21]. Greater heat generated with the light tip held close to the adhesive surface may have facilitated solvent evaporation to the point where it adversely affected the final DC. A study on model adhesives showed that the absence of solvent actually reduced the final DC compared to a system with 10-20% solvent [22]. Furthermore, greater SD values in the control group compared to "8mm_90°" group indicated greater heterogeneity in monomer conversion, which could be associated with uneven solvent evaporation.

The present results showed that the protocol of application of a universal adhesive, with or without phosphoric acid etching, had no significant influence on the initial bond strength to dentin, no matter which polymerization regime was used. That is similar to other *in vitro* studies, whether a composite resin [23,24] or ceramic materials [25] were bonded to dentin. Universal adhesives showed similar shear [25], microshear [26] or microtensile bond strength to dentin for both TE and SE protocols [27]. The present results indicate sufficient capacity of acidic monomers in SBU for partial dentin demineralization in the SE protocol as is achieved with phosphoric acid in the TE protocol.

Aging in water for 6 months significantly reduced bond strength to dentin of SBU following the TE protocol in the control group and "8mm_60°" group, whilst the values in the "8mm_90°" group were also lower but did not reach statistical significance. Similar reduction in bond strength to dentin of universal adhesives after 6 or 12 months of storage was reported

in recent studies [25,26,28]. This may be explained by adhesive bond degradation within the hybrid layer. Acid etching of dentin and subsequent adhesive application create a zone in which collagen fibers are not incapsulated with resin because of shallower penetration of adhesive than previous acid etching [29]. This aggressive procedure exposes collagen fibers to degradation by matrix metalloproteinases activated in acidic environment such as the one caused by phosphoric acid etching and self-etching primers [30]. Furthermore, the present study indicated lower DC in the adhesive following curing at a distance (8 mm) and tilted angle of the light tip (60°) which could have also contributed to a hybrid layer more prone to degradation due to resin hydrolysis. The present results suggest that clinicians should accept with caution manufacturer's recommendation that SBU adhesive may be applied equally efficiently with or without phosphoric acid on dentin. Although initial bond strength may be comparable for both protocols, aging is associated with more pronounced bond deterioration when dentin is etched with phosphoric acid prior to adhesive application (TE protocol) compared to adhesive application to non-etched dentin (SE protocol). In line with other studies on the subject in which ideal curing conditions are applied, the present results indicate that the SE application protocol may be clinician's protocol of choice especially having in mind likely clinical deviation from the ideal curing conditions.

In line with changes in bond strength associated with TE protocol after storage is the notion that a greater increase in adhesive fractures and decrease in mixed fractures occurred in all TE groups compared to SE groups. It is generally known that adhesive fractures are associated with lower bond strengths than mixed fractures which occur at higher loads. Adhesive fracture was the predominant type for other universal adhesives [1] as well as for SBS and μSBS tests to dentin [20].

Relatively large standard deviations could be viewed as a limitation of the study. However, this commonly occurs in bond strength studies, especially testing adhesive-dentin

bond strength [20,25,26]. Likely reasons for rather inhomogeneous results of bond strength testing could be variations in the sensitive dentin substrate as well as operator variability during specimen preparation.

CONCLUSION

In general, angle tilt of 60° showed a greater adverse effect on the DC of a universal adhesive and its bond strength to dentin than tip-to-surface distance of 8 mm when cured with a high-intensity light-curing unit. TE and SE adhesive application protocols gave comparable results regarding initial bond strength to dentin of a universal adhesive. After 6 months of water storage, the SE application protocol was associated with greater adhesive resistance to degradation resulting in smaller decrease in bond strength compared to the TE protocol. A high-intensity LED unit allows some departure from an ideal curing position without jeopardizing adhesive bond strength to dentin as long as the incident light is perpendicular to the surface and the adhesive is applied without acid etching.

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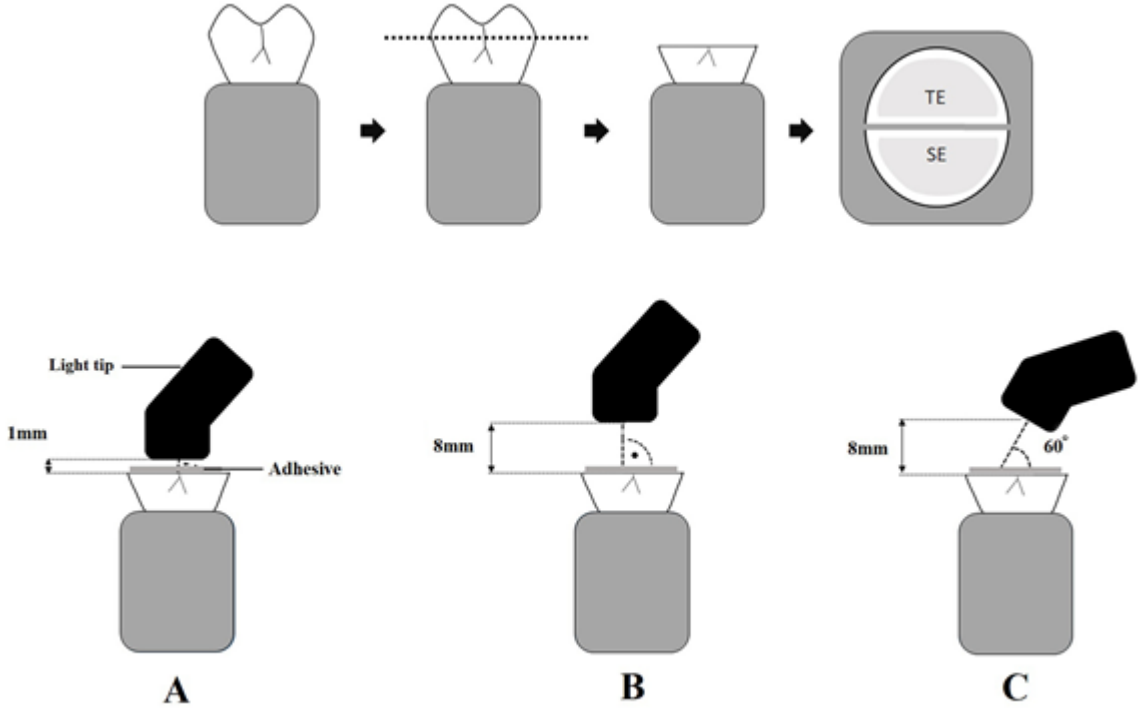

Figure 1. Specimen preparation A) Group "1mm_90°" (control); B) Group "8mm_90°; C) Group "8mm_60°";
TE – total-etch; SE – self-etch. NB; a thick adhesive layer is presented for easy identification in this schematic and does not accurately reflect the actual thickness of the adhesive layer

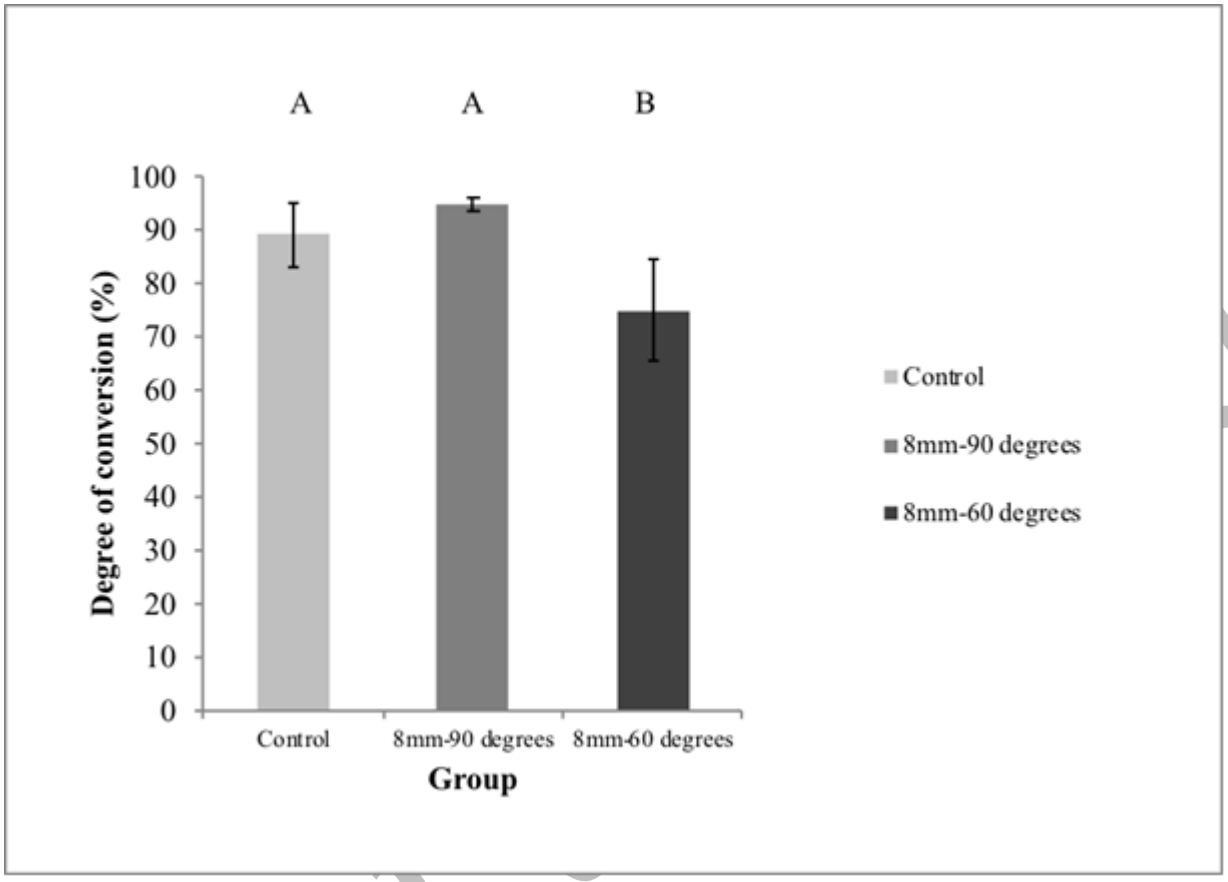

Figure 2. Mean and standard deviation values of the DC of SBU adhesive.

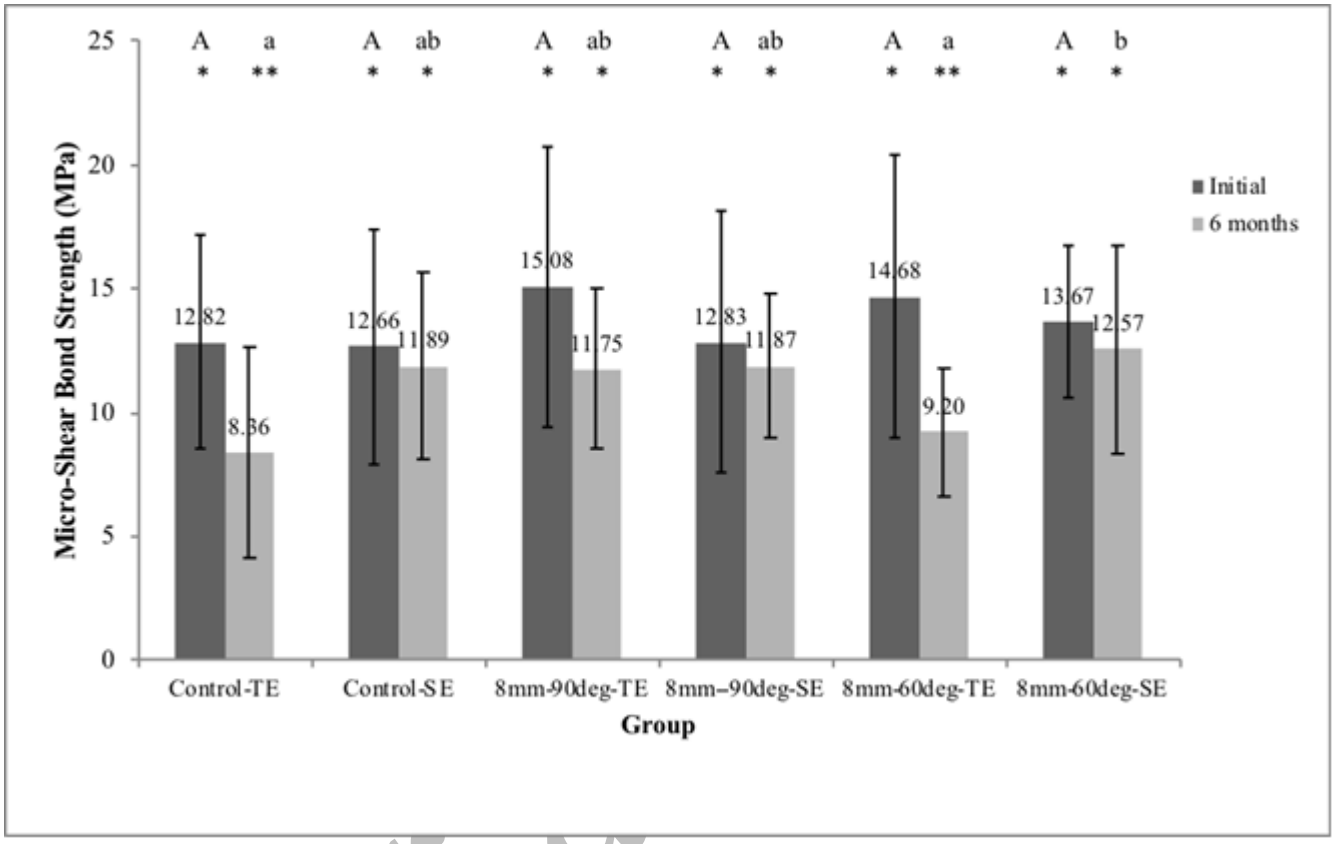

Figure 3. Mean and standard deviation values of the μSBS to dentin of SBU adhesive; upper case letters-initial-intergroup comparison; lower case letters-6months-intergroup comparison; Asterisk-initial *vs.* 6 months-intragroup comparison ($p < 0.05$).

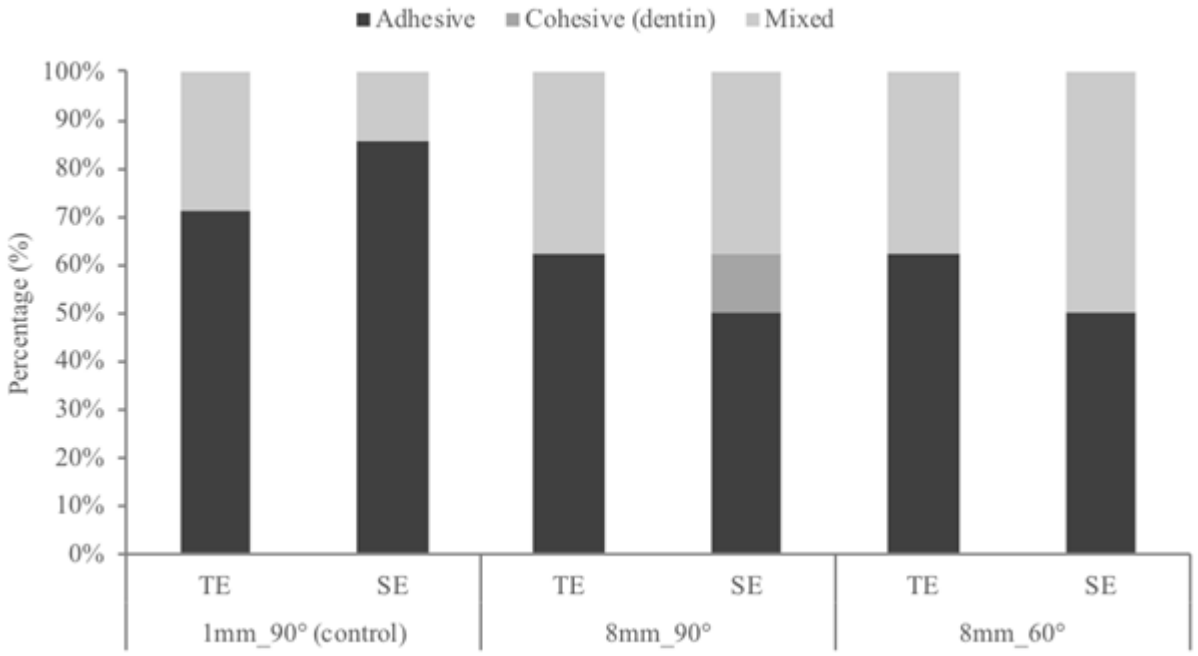

Figure 4a. Distribution of fracture types after 24 h (baseline) measurements

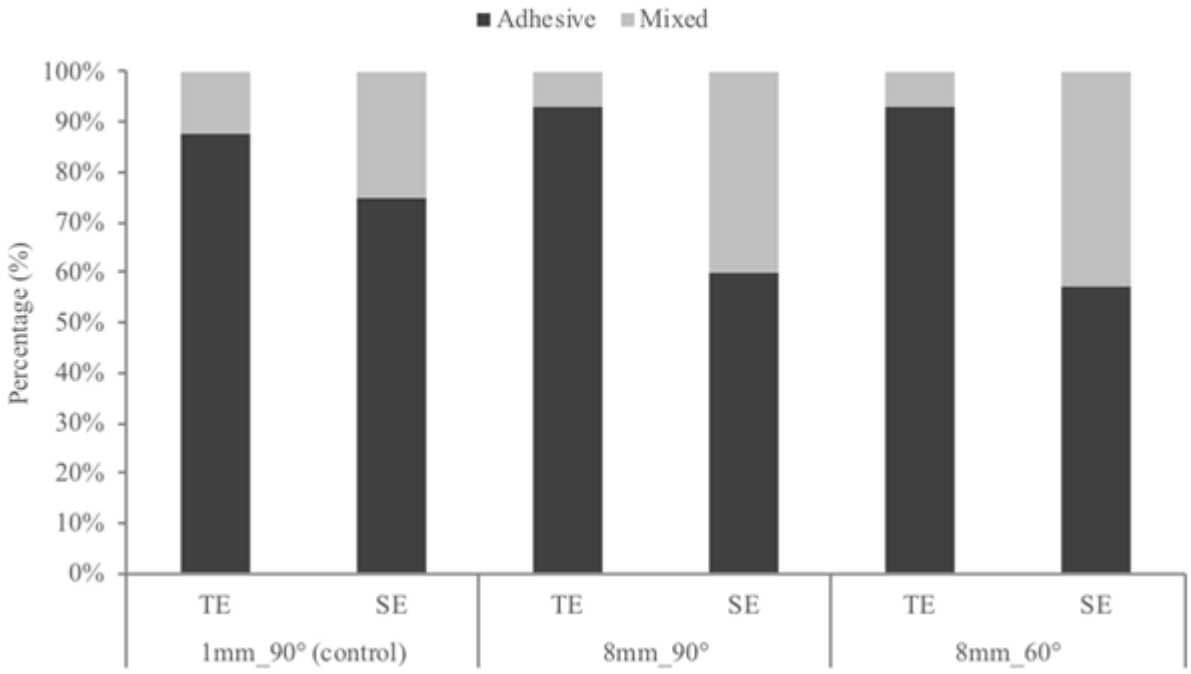

Figure 4b. Distribution of fracture types after six months measurements

Table 1. Materials used in study

Material (Code)	Manufacturer	Type	Composition
Single Bond Universal (SBU)	3M ESPE, St. Paul, MN, USA	Universal adhesive	BisGMA, HEMA, DMDMA, ethanol, water, reaction products with 1,10-decanediol and P_2O_5, silane treated silica, copolymer of acrylic and itaconic acid, camphorquinone, dimethylaminoethyl methacrylate, ethyl-dimethyalaminobenzoat
Filtek Z250 (Z250)		Microhybrid composite	BisGMA, UDMA, TEGDMA, BisEMA6, silane treated ceramic, benzotriazol, ethyl-dimethyalaminobenzoat

BisGMA – Bisphenol A diglycidyl ether dimethacrylate; HEMA – 2 hydroxyethyl methacrylate; DMDMA – decamethylene dimethacrylate; UDMA – diurethane dimethacrylate; TEGDMA – triethylene glycoldimethacrylate; BisEMA6 – Bisphenol A polyethylene glycol diether dimethacrylate

Table 2. Mean and standard deviation values of light irradiance in each group

Group	1 mm_90°	8 mm_90°	8 mm_60°
Light irradiance (mW/cm^2)	1195 ± 7	1017 ± 19	884 ± 11

