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Mikrozatezna čvrstoća veze dvaju adhezivnih sustava "sve u jednome" te jednoga s jetkanjem i ispiranjem

Micro-Tensile Dentin Bond Strength of Two All-in-One Adhesives and an Etch-and-Rinse Adhesive

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Sažetak
Svrha: Istraživanjem se željela ocijeniti mikrozatezna čvrstoća dentinske veze dvaju jednokomponentnih adhezivnih sustava te jednoga koji se jetka i ispire. **Ispitanici i postupak:** Zubi su bili nasumce podijeljeni u tri skupine kako bi se ispitali u postupku sa sljedećim adhezivnim sustavima: Adhese One®, Futurabond M® i Adper Singlebond® (kontrola). Zbog toga su korijeni i apikalni dio dna pulpne komore bili uklonjeni mikrotomom. Nakon toga su nadogradnje rezane okomito u pravokutne (ff1mm x 1mm) štapiće, te su oni testirani - uz konstantnu brzinu glave (1mm/min) - na univerzalnom stroju za ispitivanje čvrstoće. Na taj je način bio ispitan svaki adhezivni sustav. Dobilo se petnaest uzoraka i testiran je bio svaki materijal povezan s dentinom. Frakturirane površine pregledane su kako bi se odredila vrsta oštećenja. **Rezultati:** Čvrstoća veze Adhese One (5,83±3,13 MPa) bila je znatno niža nego Futurabonda M (15,76±4,2 MPa) (p:0,0001) i Adper Singlebonda (21,14±5,04 MPa) (p:0,0001). Također je čvrstoća veze Futurabonda M bila statistički mnogo niža nego Adper Singlebonda (p:0,003). **Zaključak:** Ispitani jednokomponentni adhezivni sustavi pokazali su manju zateznu čvrstoću dentinske veze nego onaj s jetkanjem i ispiranjem.

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Ključne riječi
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Uvod

Sustavi dentinskih adheziva različito se razvijaju od početaka vezanja za caklinu, dakle od šezdesetih godina od kada se rabi tehnika jetkanja. Današnji samojetkajući sustavi često se upotrebljavaju u dječjoj dentalnoj medicini te u restaurativnoj i estetskoj stomatologiji jer je postupak s njima vrlo jednostavan, a i manje su osjetljivi. Hibridizacija dentina sa smolastim monomerom osnova je postupka kojim se osigurava učinkovita dentinska veza (1). Danas se na tržištu mogu nabaviti dentinski adhezivni sustavi za jetkanje i ispiranje (tro- i dvokomponentni), ili suvremeni hidrofilni kiseli monomeri pod zajedničkim nazivom "samojetkajući adhezivi" (sve u jednom, dvo- ili jednokomponentni) (2, 3). Moramo istaknuti da ti posljednji skraćuju postupak vezivanja i sprječavaju gubitak cakline, no ne ugrožavaju klinički učinak. Unatoč jednostavnom postupku, adhezivni sustavi "sve u jednom" imaju nižu čvrstoću veze *in vitro* (3-5). Kao organski materijal često se upotrebljava Bis-GMA (2,2-bis[4-(2-hidroksi-3-metilkriloiloksipropoksi)-fenil] propan). Bis-GMA je jako viskozan zbog vodikovih veza između hidrofilnih skupina i monomernih molekula. Zato ga treba razrijediti tekućom smolom, kao što je, primjerice,

Introduction

Dental adhesive systems have taken several divergent paths over the years beginning with enamel bonding using the acid etch technique in the 1960's. Today self-etching adhesive systems are beneficial for pediatric dentistry practice as well as restorative and aesthetic dentistry because the bonding procedures are simplified and technique sensitivity is reduced. Hybridization of dentine with resin by monomer interdiffusion has been demonstrated as the fundamental mechanism in achieving effective dentine bonding (1). Today dental adhesives constitute both traditional 'etch-and-rinse adhesives' (3-step or 2-step) and contemporary systems containing hydrophilic functional monomers called as 'self-etch adhesives' (2-step or 1-step (all-in-one), (2,3); the letter adhesives are currently offered to shorten bonding procedures and eliminate enamel loss without jeopardizing clinical performance. In spite of their user-friendliness and possible low technique sensitivity, all-in one adhesives have resulted in low bonding effectiveness in vitro (3-5). As an organic material, Bis-GMA (2,2-bis[4-(2-hydroxy- 3-methycryloyloxy-propoxy)-phenyl] propane), is often in use. However, Bis-GMA reveals a very high viscosity because of the hydrogen trietilenglikol-dimetakrilat (TEGMA). Danas se kao alternativni monomer često odabire uretan-dimetilakrilat (UDMA), jer ima niži viskozitet, učinkovitije se svjetlosno veže, slabije upija vodu i tvrđi je u usporedbi s Bis-GMA-om (6). Punila se samojetkajućim adhezivnim sustavima dodaju zato što osiguravaju pojačanje, povećavaju krutost i smanjuju dimenzionalne promjene te utječu na polimerizacijsku kontrakciju (6-9). Općenito, punila od silikatnih čestica temelje se na oksidima barija, stroncija, cinka, aluminija i cirkonija (10). Kako bi se dobila čvrsta veza između organskih monomera i anorganskih čestica punila, potrebni su silani i funkcijske metrakrilatne skupine. Najčešće se upotrebljava 3-metakriloksipropil trimetoksisilan. Novost su na tržištu punila nanočestice koje djeluju kao križna poveznica da bi se pojačale veze hibridnoga sloja. Kako bi se odredila zatezna čvrstoća veze, Sano i njegovi suradnici (11) predložili su mikrotenzijski test (µTBS) i to je izvrsna metoda za mjerenje adhezivne snage između strukture zuba i kompozitne smole.

Svrha istraživanja bila je ocijeniti mikrozateznu čvrstoću nedavno razvijenih modifikacija jednokomponentnog adhezivnog sustava s punilom od silicijeva dioksida (1), novoga jednokomponentnog sustava s nanopunilom (2) te jednoga klasičnog adhezivnog sustava koji zahtijeva jetkanje i ispiranje.

Materijali i metode

Istraživanje se obavljalo na ekstrahiranim ljudskim impaktiranim trećim molarima koji su mjesec dana bili pohranjeni u slini. Zbog etičkih načela nisu bili korišteni zdravi mliječni molari. Svi pacijenti uključeni u istraživanje složili su se s postupkom. Caklina je bila potpuno uklonjena 600-mikronskim zrnatim karbidnim papirom u uređaju za poliranje (Pheonix Beta®, Buehler, Lake Bluff, Il, SAD) sve do ravne koronarne površine dentina debljine ≈3 mm (mjereno mikrometrom Mitutoyo®, Hampshire, Velika Britanija). Korijenski dijelovi zuba bili su uklonjeni mikrotomom (Isomet®, Buehler, Lake Bluff, Il, SAD) u području dna pulpne komorice. Zubi su zatim prema slučajnom izboru podijeljeni u tri skupine te podvrgnuti preporučenim postupcima za Adhese One® (Ivoclar Vivadent AG, Schaan, Liechtenstein) i Futurabond M® (Voco AG, Cuxhaven, Njemačka) ili kontroli s Adper Singlebondom® (3M Dental Products, St. Paul, MN, SAD) (Tablica 1). Kod uporabe Adhese One i Futurabonda M, materijal je vezan neposredno na dentin prema uputama proizvođača (Tablica 2.). Klasični postupak jetkanja bio je primijenjen za Adper Singlebond, također prema uputama proizvođača (Tablica 2.). Adhezivni sloj bio je polimeriziran svjetiljkom za polimerizaciju (Optilux 501®, Kerr Corporation, West Collins Orange, CA, SAD) sa snopom ne manjim od 550 mW/cm^2 i to prema uputama proizvođača 10 sekundi za sve testirane adhezivne sustave. Površina je nadograđena dvama slojevima kompozitnog materijala resina (Filtek Z250®, 3M Dental Products, St. Paul, MN, SAD) do visine od četiri milimetra. Svaki dio bio je polimeriziran

bonding interactions that occur between the hydroxyl groups on the monomer molecules. Therefore, Bis-GMA has to be diluted with a more fluid resin, for instance, triethyleneglycol-dimethacrylate. Nowadays, alternative monomer systems such as urethane-dimethacrylate (UDMA) are offered. UDMA presents lower viscosity, more effective light curing, lower water sorption, and greater toughness in comparison with Bis-GMA (6). Fillers were added to the self-etch adhesives to provide strengthening, increase stiffness, reduce dimensional changes and to influence polymerization shrinkage (6-9). Generally fillers with silicate particles are based on oxides of barium, strontium, zinc, aluminum, or zirconium (10). To provide a strong bond between the organic monomer and the inorganic filler particles, silanes with functional methacrylate groups are necessary. The most commonly used silane is 3- methacryloxypropyl trimethoxysilane. Most recently, nanoparticles were introduced to market as fillers thus acting as cross links which reinforce the bond and hybrid layer.

To evaluate strengths of adhesive bondings, Sano et al (11) had introduced the microtensile bond test (µTBS) which is an accurate method to measure the adhesive strength between tooth structure and composite resin. Thus, the purpose of the present study was to evaluate the micro-tensile dentin bond strength of (1) a recently developed modification of the all-in-one adhesive system filled with silicon dioxide; (2) a newly developed nano-filled all-in-one adhesive system to dentine with regards to conventional etching technique.

Materials and methods

Impacted human third molar teeth were extracted, stored in saline for one month and used in the present study. Sound primary molars were not used for the experiments regarding ethical issues. The present study was under the permission from informed consents from patients. With 600-grit silicon carbide paper, enamel was fully removed in a polisher (Pheonix Beta®, Buehler, Lake Bluff, Il, USA) where a flat coronal dentin surface was exposed until the remaining dentin thickness was ≈3 mm (measured with a micrometer (Mitutoyo®, Hampshire, England)). Roots and the apical floor of pulp chambers were removed with a microtome (Isomet®, Buehler, Lake Bluff, Il, USA). Then, the teeth were randomly divided into three groups for treatment with either Adhese One® (Ivoclar Vivadent AG, Schaan, Liechtenstein), Futurabond M® (Voco AG, Cuxhaven, Germany) or control as Adper Singlebond® (3M Dental Products, St. Paul, MN, USA) (Table 1). For Adhese One and Futurabond M, the materials were bonded directly to dentin according to manufacturer's instructions. (Table 2) Conventional etching procedure was applied for Adper Singlebond according to manufacturer's instructions.(Table 2) The adhesive layer was polymerized using a light-curing unit (Optilux 501®, Kerr Corporation, West Collins Orange, CA, USA) with a light output not less than 550 mW/cm^2 for 10 s for all tested adhesives according to the instructions of the manufacturers. The surface was built up with two layers of composite resin (Filtek Z250®, 3M Dental Products, St. Paul, MN, USA) to a height of 4 mm. Each increment was light cured for 20

Tablica 1. Korišteni materijal
Table 1 Materials used

Materijal • Materials	Komponente • Components	Batch # • Batch no	Proizvođač • Manufacturer
Adhese One®	derivati bis-akrilamida, voda, bis-metakrilamid dihidrogen fosfat, aminokiselina akrilamida, hidroksi-alkil metakrilamid, visoko raspršen silicijev dioksid, katalizatori i stabilizatori • derivatives of bis-acrylamide, water, bis-methacrylamide dihydrogen phosphate, amino acid acrylamide, hydroxy alkyl methacrylamide, highly dispersed silicon dioxide, catalysts and stabilizers	L17898	Ivoclar Vivadent AG, Schaan, Liechtenstein
Futurabond M®	organska kiselina, UDMA, HEMA, kamforkinon, BHT • Organic acid, UDMA, HEMA, camperchinon, BHT	01350E1	Voco AG, Cuxhaven, Njemačka • Germany
Adper Singlebond®	35% ortofosforna kiselina • Etch: 35% phosphoric acid smole: Bis-GMA, HEMA, kopolimer, polialkelične kiseline, voda, etanol, dimetakrilati • Resin: Bis-GMA, HEMA, polyalkeonic acid copolymer, water, ethanol, dimethacrylates	1122	3M Dental Products, St. Paul, MN, SAD • USA

UDMA: urethane dimethacrylate; HEMA: 2-hydroxy ethyl methacrylate; BHT: butylhydroxytoluene

Tablica 2. Postupak primjene pojedinih materijala
Table 2 Bonding procedure used

Materijal • Material	Postupak • Procedure
Adhese One®	sušenje, jetkanje 30 sekundi, sušenje, polimerizacija 10 sekundi (500 mW/cm²) • Dry, Condition with vivapen for 30 s, dry, polymerize for 10s (500 mW/cm²)
Futurabond M®	sušenje, jetkanje 20 sekundi, sušenje, polimerizacija 10 sekundi (500 mW/cm²) • Dry, Condition for 20 s, dry, polymerize for 10s (500 mW/cm²)
Adper Singlebond®	Jetkanje 15 sekundi, ispiranje i sušenje – površina mora ostati vlažna - kondicioniranje 5 sekundi, polimerizacija 10 sekundi (500 mW/cm²) • Etch 15 s, rinse and blot leaving surface moist, condition 5 s, polymerize for 10s (500 mW/cm²)

20 sekundi. Nakon toga su svi uzorci bili 24 sata pohranjeni u toploj vodi (37°C). Poslije su nadogradnje bile mikrotomom (Isomet®, Buehler, Lake Bluff, Il, SAD) okomito rezane u kvadratne šipke (≈ 1mm x 1mm). Šipke, sastavljene od dentina i kompozitnog materijala, zalijepljene su na nosač cianoakrilatnim gelom (Pattex®, Henkel, İstambul, Turska) te je ispitana njihova mikročvrstoća sve do frakturiranja na univerzalnom uređaju (Instron 3345®, Norwood, Mass, SAD) pri konstantnoj brzini glave od 1mm/min. Za svaki adhezivni sustav testirano je 15 uzoraka. Nakon što je bila ispitana čvrstoća, frakturne su površine analizirane pod mikroskopom s povećanjem od 25 puta (Opmi® Pico Carl Zeiss Meditec, Inc, Dublin, CA, SAD) kako bi se odredila vrsta oštećenja. Frakture su klasificirane u adhezivne i miješane te kohezivne u adhezivu ili kohezivne u dentinu. Srednja vrijednost dentinske veze bila je statistički ocijenjena pomoću jednosmjerne varijance, a korijen Hi-kvadratom korištenjem kompjutorskog sustava NCSS 2007® (Kaysville, UT, SAD). Vrijednost $p<0.05$ bila je postavljena kao granica statističke značajnosti.

Rezultati

Mikrozatezna čvrstoća dvaju jednokomponentnih i jednoga adhezivnog sustava s jetkanjem i ispiranjem prikazana je u Tablici 3. Rezultati jednosmjernog testa varijance za mikrozateznu čvrstoću dvaju jednokomponentnih i jednoga adhezivnog sustava s jetkanjem pokazuju statistički značajne rezultate (p:0,0001). Mikrozatezna čvrstoća za Adhese

sec. Each specimen was stored in water at 37°C for 24 h. The build-ups were vertically sectioned into quadrangular (≈ 1mm x 1mm) compound bars with a microtome (Isomet®, Buehler, Lake Bluff, Il, USA). The bars, consisting of dentin and composite resin, were glued with a cyanoacrylate adhesive gel (Pattex®, Henkel, İstanbul, Turkey) to the probe and submitted to a micro-tensile bond strength tensile tests at constant crosshead speed (1mm/min) using a universal testing machine (Instron 3345®, Norwood, Mass, USA) until fracture. For each adhesive 15 specimens were tested. After the µTBS tests, fractured surfaces were inspected at magnification (25x) (Opmi® Pico Carl Zeiss Meditec, Inc, Dublin, CA, USA) to determine the mode of fracture. Fractures were classified as adhesive, mixed, cohesive in resin or cohesive in dentin. Regarding statistical analysis, the mean dentin bond strengths were evaluated by One-way variance, Tukey and Chi-square tests using NCSS 2007® (Kaysville, UT, USA) package programme. A *p*-value <0.05 was considered as statistically significant.

Results

The mean micro-tensile bond strengths for two filled all-in-one adhesive systems and for the total-etch adhesive are shown in Table 3. The results for the One-way variants test, the micro-tensile bond strengths of two adhesive systems and the total-etch adhesive were significantly different (p:0,0001). The micro-tensile bond strengths of Adhese One

Tablica 3. Mikrozatezna čvrstoća (µTBS) dentina kod testiranih materijala
Table 3 The micro-tensile bond strength (µTBS) to dentin of the tested adhesives

	µTBS (Mpa)
Adhese One	5,83±3,13 *,**
Futurabond	15,76±4,2 *,***
Adper Singlebond	21,14±5,04 **,***
F^{Ψ}	51,35
p^{Ψ}	0,0001

$^{\Psi}$ Jednosmjerni test varijance; Tukeyev test * p:0,0001; ** p:0,0001; ***p:0,003 • $^{\Psi}$ One way varians test; Tukey test * p:0,0001; ** p:0,0001; ***p:0.003

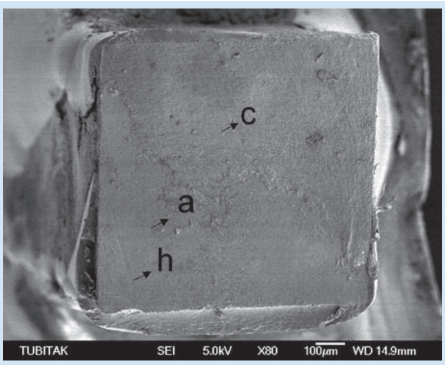
One (5,83±3,13 MPa) bila je statistički mnogo niža Futurabonda M (15,76±4,2 MPa) (p:0,0001) i Adper Singlebonda (21,14±5,04 MPa) (p:0,0001), a mikrozatezna čvrstoća Futurabonda M bila je statistički značajno niža od Adper Singlebonda (p:0,003). Vrste fraktura adhezivne veze za svaku skupinu nalaze se u Tablici 4. Pomoću mikrofrakturnog testa čvrstoće ustanovljeno je da su frakture bile pretežno adhezivne, a tri miješane i to kod Adper Singlebonda (80% adheziv, 20% miješane) (Slika 1.). Statistička analiza upućuje na veliku razliku u vrsti frakture (p:0,04). Kod skupina Adhese One i Futurabond M bilo je pronađeno 100% adhezivnih fraktura.

(5,83±3,13 MPa) was significantly lower than that of Futurabond M (15,76±4,2 MPa) (p:0,0001) and Adper Singlebond (21,14±5,04 MPa) (p:0,0001) while micro-tensile bond strength of Futurabond M was significantly lower than Adper Singlebond (p:0,003). The modes of failure for each group are shown in Table 4. With the µTBS tests, failures were predominantly adhesive in nature, where 3 mixed failures were recorded in Adper Singlebond treated group (80% adhesive, 20% mixed).(Figure 1) Statistical analysis showed that Adper Singlebond showed statistically significant difference in fracture pattern (p:0,04). 100% adhesive failures were observed for Adhese One and Futurabond M).

Tablica 4. Broj i postotak uzoraka svrstan ovisno o vrsti frakture
Table 4 The number and percentage of specimens categorized into fracture modes

	Adheziv u smoli • Adhesive in resin n (%)	Miješan u smoli • Mixed in resin n (%)	Kohezivan u smoli • Cohesive in resin n (%)
Adhese One	15 (100%)	0 (0%)	0 (0%)
Futurabond	15 (100%)	0 (0%)	0 (0%)
Adper Singlebond	12 (80%)	3 (20%)*	0 (0%)

* Hi-kvadrat test; p:0,04 • Chi-square test; p:0,04



Slika 1. SEM slika miješane frakture za Adper Singlebond; c: kompozitna smola, h: hibridni sloj, a: fraktura adheziva
Figure 1 SEM illustrating the mixed failure for Adper Singlebond; c: Composite Resin, h: Hybrid layer, a: adhesive failure

Rasprava

Dentalni adhezivi zanimljiva su mješavina sastojaka. Njihove dobre karakteristike ključne su za istraživanja i kliničku praksu. Klinička svojstva određuje im kemijski sastav. Bez obzira na broj bočica, adhezivni sustav sadržava smolaste monomere, inicijator polimerizacije, inhibitore ili stabilizatore, otapala i ponekad anorganska punila (12). Primarni mehanizam vezanja za dentin kod adhezivnih sustava s jetkanjem i ispiranjem temelji se na difuziji te ovisi o hibridizaciji i mikromehaničkom povezivanju i prožimanju smole s mrežom

Discussion

Dental adhesives are intricate mixtures of ingredients. Profound knowledge of these ingredients is one key to better understanding the behavior of adhesives in studies and in clinic. Their chemical formulation determines to a large extent their adhesive performance in clinic. Irrespective of the number of bottles, an adhesive system typically contains resin monomers, curing initiators, inhibitors or stabilizers, solvents and sometimes inorganic filler (12). At dentin, the primary bonding mechanism of etch-and-rinse adhesives is ogoljelih kolagenih dentinskih vlakana (13). Samojetkajući adhezivni sustavi koriste se kiselim monomerima koji se ne ispiru, a istodobno jetkaju i konvertiraju dentin. Takvi blagi i jaki sustavi obično ne prodiru duboko u dentin i adhezija se postiže mikromehanički kroz plitku hibridizaciju te dodatnom kemijskom interakcijom posebnih karboksil/fosfatnih funkcijskih monomera sa zaostalim hidroksilapatitom (14). Adhezivi koji sadržavaju punila smatraju se "napunjenima" za razliku od sustava "bez punila" (15). Tradicionalno su adhezivni sustavi bili bez punila, a danas se dodaju iz nekoliko razloga. Smatra se da je adhezivni sloj između kompozitnih materijala i zuba najslabija karika zbog slabe zatezne čvrstoće veze i niskoga modula elastičnosti (12, 16). U skladu s trenutačnim preporukama savjetuje se dodati punila kako bi se pojačao adhezivni sloj (17-19). Većina današnjih adhezivnih smola s punilom sadržava samo čisti silicijev dioksid. U ovom istraživanju Adhese One® sadržava silicijev dioksid, a Futurabond M® nanočestice silicijeva dioksida. Nedavno su Basaran i njegovi suradnici (20) istaknuli da manje čestice punila potiču veće polimerizacijsko skupljanje, a posljedica je manja snaga vezivanja, što je opaženo kod adhezivnih sustava s nanopunilom.

U ovom istraživanju ustanovljeno je da se adhezivni sustavi s nanopunilom snažnije vežu negoli sustavi s punilom od silicijeva dioksida. Prema tvrdnjama proizvođača, adhezivni sustavi s nanopunilom, zbog male veličine punila, omogućuju potpuno prožimanje dentina, što rezultira visokom silom adhezije (20). U određenim okolnostima ovo istraživanje može potvrditi tu tvrdnju. U dostupnoj literaturi istaknuto je - unatoč sve većoj popularnosti jednokomponentnih samojetkajućih adhezivnih sustava – da se "zlatnim standardom" još smatra jetkanje ortofosfornom kiselinom (3, 21). Naše istraživanje podupire tu tvrdnju. Jednokomponentni adhezivni sustavi imaju manju silu adhezije nego oni konvencionalni s jetkanjem i ispiranjem, a među njima sustavi s nanopunilom imaju nešto veću silu vezivanja od sustava s punilom od silicijeva dioksida. Mora se istaknuti da se u nekim istraživanjima tvrdi da samojetkajući adhezivni sustavi postižu istu snagu adhezivne veze kao i oni s jetkanjem i ispiranjem (22-24).

S obzirom na vrstu frakture kod svake skupine, može se ustvrditi da su bile uglavnom adhezivne, a tri miješane ustanovljene su jedino kod zuba tretiranih klasičnim adhezivnim sustavom s jetkanjem i ispiranjem. Uočena vrsta frakture ista je kao i kod nedavnih istraživanja u kojima je također bila adhezivna (23).

Zaključak

Jednokomponentni adhezivni sustavi postižu manju silu vezivanja nego konvencionalni s jetkanjem i ispiranjem, a sustavi s nanopunilom stvaraju jaču vezu od onih s punilom od silicijeva dioksida. Dječji stomatolozi trebali bi biti svjesni prednosti i nedostataka novih, pojednostavljenih tehnika i materijala.

primarily diffusion-based and depends upon hybridization or micro-mechanical interlocking of resin within the exposed collagen fibril scaffold (13). 'Self-etch' adhesives use non-rinse acidic monomers that simultaneously condition and prime dentin. Mild and strong self-etch adhesives are generally diffuse to dentin and adhesion is consequently obtained micro-mechanically through shallow hybridization and by additional chemical interaction of specific carboxyl/phosphate groups of functional monomers with residual hydroxyapatite (14).

Adhesive containing fillers are said to be 'filled', in contrast to 'unfilled' adhesives (15). Traditionally adhesives systems were unfilled while today fillers can be added to adhesives for several reasons. It was stated that the adhesive resin layer established between the composite resin and the tooth is considered to be a weak link due to its low tensile strength and low elastic modulus (12,16). Regarding the present situation, it has been suggested that the addition of fillers may fortify the adhesive layer (17-19). Today the most filled adhesive resins for bonding composites contain only pure silicon dioxide. In the present study, Adhese One® contains silicon dioxide while Futurabond M® contains nano particles with silicium dioxide. Currently Basaran et al[20] investigated and suggested that smaller filler particles lead to greater shrinkage which results in low bond strengths, which was observed with a nanofilled adhesive. However in the present study, it was found that nanofilled adhesive system showed greater bond strength than a silicon dioxide filled adhesive system. According to manufacturer of a nanofilled adhesive system, it was claimed that the minute size of the nanofillers allows complete penetration which contributes to high adhesion (20). The present study may support the regarding statement in certain circumstances. In recent literature, it was undermined that despite the increased popularity of self-etch adhesives, etching with phosphoric acid is still considered the golden standard against which new materials are tested (3,21). The present study supports the regarding hypothesis. All-in-one adhesives showed lower bond strengths than conventional etching adhesive, while nanofilled adhesives showed greater bond strength than a silicon dioxide filled adhesives. However there are also some reports that self-etching primer systems are producing the same bond strengths as the etch-and-rinse adhesive (22-24).

Regarding modes of of failure for each group, failures were predominantly adhesive in nature; where 3 mixed failures were recorded only in conventional etching adhesive treated group. The present mode of failure mimics a recent study's results where fractures were mostly adhesive in nature (23).

Conclusion

In conclusion, all-in-one adhesives showed lower bond strengths than conventional etching adhesive while nanofilled adhesives showed greater bond strength than a silicon dioxide filled adhesives. Paediatric dentists should be aware of advantages and disadvantages of new bonding procedures that are simplified.

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Abstract

Aim: The purpose of the present study is to evaluate the micro-tensile bond strength to dentin of two all-in-one adhesives and an etch-and-rinse adhesive. **Material and Methods**: The teeth were randomly divided into three groups for treatment with one of the following adhesives: Adhese One®, Futurabond M® and Adper Singlebond® (control). Roots and the apical floor of pulp chambers were removed with a microtome. The build-ups were vertically sectioned into rectangular (ﬀ1mmx 1mm) compound bars with microtome. The bars were submitted to tensile tests at constant crosshead speed (1mm/min) using a universal testing machine and tested for each adhesive. Fifteen specimens were manufactured and tested for each material for permanent dentin. Fractured surfaces were inspected to determine the mode of fracture. **Results:** The bond strengths of Adhese One (5,83±3,13 MPa) was significantly lower than Futurabond M (15,76±4,2 MPa) (p:0,0001) and Adper Singlebond (21,14±5,04 MPa) (p:0,0001) while dentin bond strenght of Futurabond M was significantly lower than that of Adper Singlebond (p:0,003). **Conclusions:** The tested all-in-one adhesives showed lower dentin bond strengths than an etch-and-rinse adhesive.

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Key words
Adhesives; Dentin-Bonding Agents; Tensile Strength

References

1. Nakabayshi N, Pashley DH. Hybridization of dental hard tissues. Tokyo: Quintessence; 1998.
2. Hashimoto M, Ohno H, Sano H, Tay FR, Kaga M, Kudou Y et al. Micromorphological changes in resin-dentin bonds after 1 year of water storage. J Biomed Mater Res. 2002;63(3):306-11.
3. Perdigão J. New developments in dental adhesion. Dent Clin North Am. 2007 Apr;51(2):333-57, viii.
4. Van Meerbeek B, De Munck J, Yoshida Y, Inoue S, Vargas M, Vijay P et al. Buonocore memorial lecture. Adhesion to enamel and dentin: current status and future challenges. Oper Dent. 2003 May-Jun;28(3):215-35.
5. Goracci C, Sadek FT, Monticelli F, Cardoso PE, Ferrari M. Microtensile bond strength of self-etching adhesives to enamel and dentin. J Adhes Dent. 2004 Winter;6(4):313-8.
6. Faltermeier A, Rosentritt M, Faltermeier R, Reicheneder C, Müssig D. Influence of filler level on the bond strength of orthodontic adhesives. Angle Orthod. 2007 May;77(3):494-8.
7. Ferracane JL, Antonio RC, Matsumoto H. Variables affecting the fracture toughness of dental composites. J Dent Res. 1987 Jun;66(6):1140-5.
8. Kim KH, Park JH, Imai Y, Kishi T. Microfracture mechanisms of dental resin composites containing spherically-shaped filler particles. J Dent Res. 1994 Feb;73(2):499-504.
9. Miyazaki M, Hinoura K, Onose H, Moore BK. Effect of filler content of light-cured composites on bond strength to bovine dentine. J Dent. 1991 Oct;19(5):301-3.
10. Khan AM, Suzuki H, Nomura Y, Taira M, Wakasa K, Shintani H, Yamaki M. Characterization of inorganic fillers in visible-light-cured dental composite resins. J Oral Rehabil. 1992 Jul;19(4):361-70.
11. Sano H, Shono T, Sonoda H, Takatsu T, Ciucchi B, Carvalho R et al. Relationship between surface area for adhesion and tensile bond strength--evaluation of a micro-tensile bond test. Dent Mater. 1994 Jul;10(4):236-40.
12. Van Landuyt KL, Snauwaert J, De Munck J, Peumans M, Yoshida Y, Poitevin A et al. Systematic review of the chemical composition of contemporary dental adhesives. Biomaterials. 2007 Sep;28(26):3757-85.
13. Yoshida Y, Nagakane K, Fukuda R, Nakayama Y, Okazaki M, Shintani H et al. Comparative study on adhesive performance of functional monomers. J Dent Res. 2004 Jun;83(6):454-8.
14. Peumans M, Kanumilli P, De Munck J, Van Landuyt K, Lambrechts P, Van Meerbeek B. Clinical effectiveness of contemporary adhesives: a systematic review of current clinical trials. Dent Mater. 2005 Sep;21(9):864-81.
15. Frankenberger R, Lopes M, Perdigão J, Ambrose WW, Rosa BT. The use of flowable composites as filled adhesives. Dent Mater. 2002 May;18(3):227-38.
16. Bae JH, Cho BH, Kim JS, Kim MS, Lee IB, Son HH et al. Adhesive layer properties as a determinant of dentin bond strength. J Biomed Mater Res B Appl Biomater. 2005 Aug;74(2):822-8.
17. Van Meerbeek B, Dhem A, Goret-Nicaise M, Braem M, Lambrechts P, VanHerle G. Comparative SEM and TEM examination of the ultrastructure of the resin-dentin interdiffusion zone. J Dent Res. 1993 Feb;72(2):495-501.
18. Sano H, Yoshiyama M, Ebisu S, Burrow MF, Takatsu T, Ciucchi B et al. Comparative SEM and TEM observations of nanoleakage within the hybrid layer. Oper Dent. 1995 Jul-Aug;20(4):160-7.
19. Miyazaki M, Ando S, Hinoura K, Onose H, Moore BK. Influence of filler addition to bonding agents on shear bond strength to bovine dentin. Dent Mater. 1995 Jul;11(4):234-8.
20. Başaran G, Ozer T, Devecioğlu Kama J. Comparison of a recently developed nanofiller self-etching primer adhesive with other self-etching primers and conventional acid etching. Eur J Orthod. 2009 Jun;31(3):271-5.
21. Perdigão J, Gomes G, Duarte S Jr, Lopes MM. Enamel bond strengths of pairs of adhesives from the same manufacturer. Oper Dent. 2005 Jul-Aug;30(4):492-9.
22. Brackett WW, Tay FR, Looney SW, Ito S, Haisch LD, Pashley DH. Microtensile dentin and enamel bond strengths of recent self-etching resins. Oper Dent. 2008 Jan-Feb;33(1):89-95.
23. Burrow MF, Nopnakeepong U, Phrukkanon S. A comparison of microtensile bond strengths of several dentin bonding systems to primary and permanent dentin. Dent Mater. 2002 May;18(3):239-45.
24. Hashimoto M, De Munck J, Ito S, Sano H, Kaga M, Oguchi H et al. In vitro effect of nanoleakage expression on resin-dentin bond strengths analyzed by microtensile bond test, SEM/EDX and TEM. Biomaterials. 2004 Nov;25(25):5565-74.