

Pregled strategije adhezije na caklinu i dentin

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Sažetak

U uvodu se ističe važnost kemijskoga predtretmana tvrdih zubnih tkiva tijekom restorativnoga postupka. Adhezijski restorativni postupak modificira temeljna načela preparacije kaviteta i načela prevencije zubnoga karijesa.

Adhezijski se postupak početkom pedesetih godina ograničio samo na kemijski tretman caklinskih rubova. Mnoga tadašnja istraživanja bavila su se koncentracijom kiselina i vremenom jetkanja, te ultramikroskopskim izgledom jetkane caklinske površine.

Kemijska obrada dentina i vezivanje kompozitnog ispuna na dentin pokazali su se težom i složenijom zadaćom. No, stalan napredak i poboljšanja dentinskih adheziva te pet generacija daju danas optimalne rezultate glede same procedure kliničkog adhezijskog postupka kao i čvrstoće veze kompozitnog ispuna naspram demineraliziranog dentina i cakline.

Novije histopatološke studije pulpnoga tkiva, nakon provedenog adhezijskog restorativnog postupka, pokazale su da ne slabi funkcija i vitalitet zubne pulpe. Biokompatibilnost zubne pulpe naspram kemijskog predtretmana i uporabe adhezijskih sustava i dalje se provjerava različitim parametrima bioloških testiranja kako bi klinički uspjesi imali potporu i potvrdu.

Ključne riječi: adhezija, caklina, dentin, adhezijski sustavi

Acta Stomatol Croat
1998; 323—330

PREGLEDNI RAD
Primljeno: 28. studenog 1997.

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Uvod

Uporaba kiseline tijekom kliničkoga postupka obrade i pripreme cakline, kako bi se kompozitni materijali vezivali za tvrdo zubno tkivo uzrokom je znatnih promjena kliničkoga postupka nadoknade izgubljenoga tvrdoga zubnog tkiva. Već sama mogućnost da se ispun čvršće i bolje vezanje za caklinu modificira temeljna načela preparacije kaviteta, načela prevencije zubnoga karijesa, te mijenja estetska načela nadoknade izgubljenoga tvrdog zubnog tkiva. Najveća prednost obrade cakline kiselinom jest što se smanjuje smanjenje rubna pukotina i nema mikropropuštanja sline i bakterija.

Adhezija restorativnih materijala na caklinu postaje danas rutinski klinički postupak, a adhezija ispunjena na dentin pokazala se je kao teži zadatak i s manje predvidljivih rezultata. Mnoge poteškoće kod vezivanja za dentin rezultat su složene histološke strukture, mineralnog sastava i specifične fiziologije pulpo-dentinskog kompleksa.

Kemijska obrada cakline

Temeljna načela adhezijske preventivne i restorativne stomatologije postavio je sredinom ovoga stoljeća Buonocore (1) predlažući uporabu kiselina sa svrhom da se promijeni caklinska površina. Svrha mijenjanja površine bila je stvoriti uvjete za bolje vezivanje ispunjena za caklinske zidove kaviteta. Buonocoreva se hipoteza temelji na uporabi kiselina u industriji kako bi se poboljšala adhezija boja i akrilnih prevlaka za metalne površine. Proročanski je predložio više potencijalnih mogućnosti uporabe tehnike jetkanja cakline i čvršćeg vezivanja ispunjena za caklinsko tkivo. Njegove su zamisli našle kliničku potvrdu u gotovo svakoj stomatološkoj disciplini.

Daljnji radovi Gwinnetta i Matsua, Retiefa i Silvestrona (2, 3, 4, 5) unose više jasnoće i logike u mehanizam vezivanja niskoviskozne smole za jetkanu caklinu. Obradom caklinske površine fosforom kiselinom odstranjuje se oko 10 mikrometara caklinskoga tkiva, ostavljajući mikroporoznu površinu. Niskoviskozna smola popunjava te mikropore tzv. smolastim produljcima, polimerizira se i stvara mikromehaničku vezu s caklinom. Jetkanjem se osim toga znatno umnaža retencijska površina i po-

većava razina površinske energije cakline, što rezultira optimalnim vlaženjem površine i dobrom adhezijom.

Sljedeća su istraživanja bila usmjerena na različitost koncentracija fosforne kiseline i učinak na čvrstoću veze. Brojni radovi Gwinnetta (6) i drugih istraživača potvrdili su da fosforna kiselina u koncentraciji između 30 i 40 % daje najbolje rezultate jetkanja s obzirom na adhezijske zahtjeve, te opisali tri ultramikroskopske slike jetkane caklinske površine. S podacima dostupnim iz strane literature slažu se i rezultati istraživanja domaćih autora (7).

I vrijeme jetkanja bila je česta tema istraživanja. Buonocore je preporučio 60 sekundi, a Mardaga (8) je 1982. godine izvjestio da je vrijeme jetkanja od 15 sekundi dovoljno. Laboratorijski testovi čvrstoće veze cakline i kompozita, te rubnoga mikropropuštanja, potvrdili su dostatnost kraćeg vremena jetkanja (9,10). Kliničke studije više autora potvrdile su da se stupanj mikropropuštanja ne smanjuje proporcionalno smanjenjem vremena jetkanja (11,12).

Suvremeni izbor kiselina slabijih koncentracija temelji se na istodobnom tretmanu cakline i dentina (10 %-tna fosforna kiselina, 10 %-na maleična kiselina, 2.5 %-tna dušična kiselina). Mnogi autori zaključuju da od uporabe kiseline blage koncentracije nastaju znatno manje sile vezivanja, ali još uvijek dovoljne za dobro rubno zatvaranje i smanjeno mikropropuštanje (13).

Kemijska obrada dentina

Kemijska obrada dentinske površine kaviteta tijekom adhezijskoga restorativnog postupka mora odgovoriti mnogo složenijem histološkom, strukturnom i mineralnom sastavu dentinskoga tkiva. Dok je caklina 92 % volumena anorganski hidroksiapatit, dentin je tek 45 % volumena anorganskog sastava. Raspored kristala hidroksiapatita unutar organskog (kolagen) matriksa dentina je nepravilniji. Dentin je osim toga histološki i fiziološki znatnije vezan za zubnu pulpu, a dentinski tubulusi svojim sadržajem i funkcijom zahtijevaju restorativni tretman temeljen na strogim biološkim načelima. Permeabilnost dentina u izravnoj je korelaciji sa stupnjem hipersenzibiliteta vitalnoga zuba, pa smanjivanjem propusnosti dentinskih tubulusa, bilo prirodnim procesom zatvaranja tubulusa koji traži dulje vrijeme,

bilo različitim terapijskim postupcima, uspješno se zbrinjava preosjetljivost vitalnoga zubnog tkiva.

Broj tubulusa varira ovisno o mjestu istraživanja (45.000 na mm² blizu pulpe, a 20.000 na mm² na CDS-u) (14). Pashley (15) je izračunao da tubulusi zauzimaju 22 % površine blizu pulpe, te samo 1 % blizu cakline. Heymann i Bayne (16) su utvrdili 28 % i 4 % volumena dentinskih tubulusa za ista područja.

Poznato je da se varijacija volumena dentinskih tubulusa po područjima reflektira u permeabilnosti dentinskoga tkiva. Tako je permeabilnost okluzalnog dentina veća na pulpalnim vrhovima nego u sredini okluzalne površine. Slično tomu, dentin bliže pulpi propustljiviji je od okluzalnog dentina, a koronarni je dentin propustljiviji od dentina korijena. Tekućina u dentinskim tubulusima je pod malim ali konstantnim tlakom iz pulpe. Procjenjuje se da intrapulpalni tlak iznosi 25-30 mm Hg (17).

Kemijska priprema dentinskih zidova kaviteta i vezivanje ispuna za dentin se osim spomenutih složenih uvjeta, komplicira i postojećim strugotinskim slojem (engl. smear layer). Taj sloj debljine 0,5 - 5 mikrometara zatvara otvore dentinskih tubulusa, djeluje kao difuzijska barijera smanjujući permeabilnost dentina. Debljina, sastav i izgled variraju ovisno o specifičnosti supstrata, tehnici obrade i upotrebljenim instrumentima tijekom preparacije kaviteta. Bakterije u tome sloju mogu preživjeti i množiti se te su stalan uzrok upale zubne pulpe (18,19).

Kad se je rješavao problem vezivanja ispuna na dentinsko tkivo, sve naprijed spomenute specifičnosti i zapreke trebalo je riješiti timskim istraživačkim radom. Razvoj dentinskih adheziva na bazi smole možemo pratiti unatrag 4 desetljeća, svrstavajući ih u generacije.

U prvoj generaciji, prije 40 godina, preporučena je smola koja sadržava dimetilakrilat glicerofosforne kiseline. Ona se može vezati na dentinsku površinu obrađenu hidroklornom kiselinom. Nedostatak te smole bila je velika osjetljivost na vodu, pa je Bowen sintetizirao N-feniglicin glicidil metakrilat (NPG-GMA), površinski aktivni komonomer, koji teoretski može stvoriti voodotporno kemijsko vezivanje smola na dentinski kalcij (12).

Druga generacija dentinskih adheziva razvijena je ranih osamdesetih godina i oni su većinom bili halofosforni esteri nepunjenih smola, kao bisfenol A-

glicidil metakrilat (bis-GMA) ili hidroksietil metakrilat (HEMA). Glavni razlog slabe izvedbe tih vezivnih sredstava je činjenica da se oni vežu na strugotinski sloj (smear layer), a ne na sam dentin. Zato je njihova sila vezivanja ograničena kohezivnom silom strugotinskog sloja i adhezijom na dentin koji se nalazi ispod (20).

Sljedeća, treća generacija dentinskih adheziva, razvijena posljednjih godina, zahtijeva modificiranje ili uklanjanje smear layera kako bi se omogućio prodao smole u dentin koji je ispod toga sloja. Ta je generacija dentinskih adheziva djelotvornije smanjivala mikropropuštanje na rubovima dentina ili cementa. Daljnje su joj prednosti što čuva i podupire strukture tvrdoga zubnog tkiva i omogućuje bolju prevenciju rubnoga karijesa. Poboljšana čvrstoća veze dentinskim adhezivima potvrđena je mnogim kliničkim i laboratorijskim istraživanjima. Prethodna obrada kiselinom i ovdje postaje pravilo. Fusayama i suradnici prvi su godine 1979. počeli kemijskom obradom dentina, i ona od tada postaje prilično uobičajena praksa u Japanu (21). No koncepcija potpune obrade kiselinom (total etch) prihvaćena je nešto kasnije u SAD-u (20,22). Pomak prema potpunoj obradi kiselinom jest radikalni zahvat u američkoj restorativnoj stomatologiji jer se je obrada dentina kiselinom tradicionalno obeshrabivala. Podaci iz studija rađenih sedamdesetih godina upućuju da obrada dentina fosfornom kiselinom izaziva pulpalnu inflamaciju (23,24). No vrlo malo kiselina zapravo prodire u dentin, te se čini nemogućim da je upravo kiselina izravan uzrok bilo kakvu oštećenju pulpe.

Sadašnji podaci uglavnom upućuju na to da je nedovoljno rubno brtvljenje glavni uzrok pulpalne inflamacije kod trajnih restoracija. Vrlo slaba ili pak nikakva upala ne može se razviti ako restoracija brtvi dovoljno dobro da spriječi prodor sline i bakterija prema pulpi (25,26,27).

Adhezijski sustavi četvrte generacije imaju kondicionere kojima se uklanja strugotinski sloj (smear layer) i demineralizira dentinska površina do određene dubine.

Iako se rabe razni kondicioneri, temeljni premaži (primer) i adhezijske smole, mehanizmi vezivanja raznih adhezijskih sustava na dentin obrađen kiselinom u velikoj su mjeri slični (28). Jetkanje kiselinom, prvi korak adhezijskog postupka, otklanja strugotinski sloj (smear layer), otvara dentinske tu-

buluse, demineralizira intertubulusni i peritubulusni dentin povećavajući dentinsku permeabilnost. Kristali hidroksiapatita otapaju se i uklanjaju ispiranjem vodom - drugi korak u postupku - ostavljajući kolabiranu kolagenu mrežu zbog gubitka anorganske potpore (29,30).

Osnovni premaz (primer - treći korak postupka) ovlažuje i prodire u kolagenu mrežu, te ju tako "diše" gotovo do njezine originalne razine i volumena. Osnovni premaz vlaženjem također povezuje površinsku energiju. Voda olakšava reakciju izmjenjane iona (31). Sljedeći, četvrti korak postupka jest nanošenje nepunjene smole (bonding) koja prodire kroz dentin obrađen osnovnim premazom, kopolimerizir s osnovnim premazom, te tvori izmiješani sloj kolagena i smole, koji se naziva zona učvršćene smole, sloj infiltrirane smole ili hibridni sloj (32,33,34). Stvaranje hibridnoga sloja dentina i smole prvi su opisali Nakabayashi i suradnici, te se smatra primarnim mehanizmom vezivanja većine suvremenih adhezivnih sustava (35).

Složenost kliničkoga postupka uporabe svih dosadašnjih dentinskih adheziva i stalna težnja da se poboljšaju rezultati adhezivnog učinka dala je petu generaciju dentinskih adheziva.

Ono što je bitno za adhezivnu vezu pete generacije i od čega je postignut klinički uspjeh današnjih adhezivnih sustava jest predtretman dentina. Strukturno složeni i hidrofilni dentinski supstrat tim predtretmanom postaje receptivniji za adhezivno vezivanje kompozitnog ispuna (36).

Kemijski predtretman smear layera tijekom kliničkog adhezivnog postupka nudi tri strateška smjera.

Jedna strategija kemijskoga predtretmana ima cilj modificirati smear layer i inkorporirati ga u vezu. Drugi strateški smjer zahtijeva da se potpuno ukloni smear layera i istodobno demineralizira dentinska površina.

Posljednji, suvremeni adhezivni sustavi pete generacije jednom intermedijarnom strategijom osiguravaju blagim kiselinama i skraćenim vremenom jetkanja djelomičnu demineralizaciju cijelom debljinom smear layera i dentinske površine neposredno ispod smear layera. Najnovija poboljšana tehnologija dentinskih adheziva uključuje smear layer kao supstrat kroz koji se provodi vezivanje, ali s novom i složenijom ulogom, budući da su tehnološki po-

boljšani proizvodi sposobni djelovati kroz smear layer na dentinski matriks (37).

Na izbor adhezivnih sustava, dostupnih na današnjem dentalnom tržištu, odlučuje i klinička procedura. Naime, razlikujemo adhezivne sustave gdje je sjedinjena kiselina i primer, pa se klinički postupak provodi samo u dva koraka (bonding steps): 1. kiselina/primer, 2. adhezivna smola. Postoji i druga inačica, kad su sjedinjeni primer i adhezivna smola, pa se klinički postupak provodi također u dva koraka: 1. kiselina i 2. primer/adhezivna smola (38).

Adhezivni sustavi prve skupine sadržavaju kiseli phenyl-P monomer i HEMA u svrhu istodobnog kondicioniranja (etching) i premazivanja (priming) cakline i dentina.

Osim što se skraćuje postupak i pojednostavljuje procedura, prednost je tih sustava što površinski demineraliziraju dentin i ujedno ga penetriraju s monomerima koji mogu biti polimerizirani *in situ*. Tako stvoreni kontinuitet, od nepromijenjenoga dentina do adhezivne smole, nema sloja nepolimeriziranih hidrofilnih monomera koji bi bio visoko osjetljiv na hidrolizu te na nastanak mikropukotina i mikropropuštanja (33,39,40).

Daljnja je prednost što ne treba ispirati vodom, nego samo umjereno sušiti zrakom kako bi se izbjeglo da se ispiru primer HEMA.

Nadalje, osim kiselih monomera kao što su 20%-tni phenyl-P i nazočnost 30%-tne HEMA-e, ta otopina sadržava vodu i alkohol. Ta hlapljiva otapala ispare kad se premazana površina osuši zrakom. Budući da je kontrakcija demineraliziranoga dentinskog matriksa (kolagenog) proporcionalna količini otapala koje može ishlapiti, trebalo bi biti manje kontrakcije dentinske površine tretirane tim sustavima nego prijašnjim generacijama, a time reducirati nastanak pukotine između kompozita i caklinsko - dentinskih zidova (41).

Postoji nekoliko kompetitivnih procesa koji se istodobno događaju kod toga procesa. Mineralna faza dentina mora biti rastopljena kako bi se napravilo mjesto za infiltraciju adhezivnom smolom. Ti smolasti monomeri moraju difundirati prema unutra istodobno s difundiranjem iona kalcija i fosfata prema van. Svaki proces je vjerojatno usporen onim drugim, vodeći do manje demineralizacije dentina i manje infiltracije smolom. No ipak je najvažnije da

je mikropukotina općenito manja nego kod uporabe adheziva prijašnjih generacija. Dio razloga tomu je što smolom infiltrirani smear layer postaje inkorporiran u hibridni sloj i služi kao unutarnji nisko-modulusni liner koji slabi stres.

Oslobađanjem stresa između kompozita i adhezijskog sloja smanjuje se mogućnost nastanka mikropukotine i povećava se trajnost kompozitne restoracije (41,42,43, 44).

U drugoj skupini naprijed spomenutih adhezivskih sustava najprije se nanosi slaba kiselina (10-15% fosforna kiselina) 15 sekundi na caklinu i dentin (total etching) (45). Pošto se kiseline ispere vodom, drugim korakom adhezijskoga postupka nanosi se na caklinsku i dentinsku površinu sjedinjeni primer/adhezijska smola (priming i bonding). Tako sjedinjeni primer/adhezijska smola sadrži dobro balansirane hidrofilne i hidrofobne monomere. Hidrofilnost BPDM monomera u mješavini pomaže infiltraciju poroznoga dentinskog supstrata, a hidrofobni monomer Bis-GMA osigurava dovoljno debeli sloj stvrdnute smole. Na površini toga sloja ostaje tanak, nestvrdnuti sloj (kisikom inhibiran) i pospješuje adaptaciju kompozitnoga materijala čvrstom vezom (46,47). Neutralizacije nepoželjnoga polimerizacijskog stresa postiže se tehnikom nanošenja u više slojeva, kad se niskoviskozni kompozit, niskog modula elastičnosti, postavlja na površinu adhezijske smole. Niskoviskozni kompozit zbog nižeg modula elastičnosti fleksijom kompezira stres i smanjuje mogućnost nastanka mikropukotine (48). Povećavajući čvrstoću i trajnost adhezijske veze osigurava se dugotrajnost cjelokupnoga kompozitnog ispuna.

Mikromehaničkim prožimanjem demineraliziranih područja adhezijskom smolom ostvaruje se čvrstoća veze koja premašuje 20 MPa (kontrakcijska sila tijekom polimerizacije kompozita, a koja može odvojiti kompozit od dentina, je oko 18 MPa) (29).

Podatak iz nedavne studije *in vitro* pokazuje da od infiltracije smole (hibridizacija) dentinskih tubulusa i intertubulusnog dentina nastaje ukupno vezivanje smole na dentin. Čvrstoća veze je uglavnom manja kad sredstvo vezivanja ne stvara hibridni sloj, ili je dentinska površina prethodno bila u doticaju s privremenim cementima na bazi eugenola (48,49).

SEM također otkriva da mnogi adhezivi stvaraju dugačke produljke smole (resin tags) unutar den-

tinskih tubulusa. Ti su završetci impresivni po izgledu, ali ako nisu čvrsto vezani za stijenku tubulusa, produžeci smole se malo ili nikako ne zadržavaju. Čak i konvencionalne (hidrofobne) smole za vezivanje na caklinu stvarat će duge produljke u dentinu obrađenom kiselinom, no neće dati znatnu silu vezivanja jer smola ne ovlažuje, odnosno ne veže se na adekvatan način na stijenke tubulusa. Daljnji podatak o relativnoj važnosti produljaka smole jest činjenica da su sile vezivanja na duboki, tubulusima bogati dentin uglavnom male (50).

Naposlijetku, završetci smole koji se stvaraju *in vivo* vjerojatno su kraći od onih na izvađenim zubima, jer su dentinski tubulusi ispunjeni tekućinom pod tlakom koja smanjuje prodor smole (51,52,53).

Vrlo važan problem zapažen na SEM i TEM studijama jest mogućnost nerazmjera između dubine demineralizacije i prodora smole adhezivnih sustava na dentin obrađen kiselinom (54). Čvrstoća veze adheziva na dentin obrađen kiselinom može se osnivati na njegovoj sposobnosti da potpuno zamijeni otopljeni hidroksiapatit polimeriziranom smolom. Ako je dentin demineraliziran toliko duboko da porozna zona ne može biti dobro impregirana smolom, krhki kolageni sloj može kolabirati i s vremenom se degradirati. Iako su suvremeni osnovni premazi (primeri) vrlo hidrofilni, adekvatna impregnacija smolom može zahtijevati da se upotrijebe tehnike obrade kiselinom koje ne rezultiraju velikim dubinama demineralizacije. Kraće vrijeme obrade ili manje agresivne kiseline mogli bi se rabiti u budućnosti (47). Upravo se na toj razini cjelokupnog adhezijskog postupka mogu u budućnosti očekivati promjene i tehnološka poboljšanja. Debljina hibridnoga sloja i čvrstoća veze interdifuzijske zone jesu parametri o kojima ovisi uspješnost adhezijskoga postupka. Suvremeni dentinski adhezivni sustavi upravo u tom segmentu nastoje tehnološkim inovacijama poboljšati koncepciju adhezijskoga postupka.

Novije histopatološke studije pulpnoga tkiva i procjena biokompatibilnosti kemijskoga predtretmana dentina, uporabe adhezijskih sustava, te izravnoga prekrivanja eksponirane pulpe pokazale su da ne slabi funkcija, vitalitet i biološki potencijal zubne pulpe. Također se pokazalo da ne slabi zacjeljivanje pulpnoga tkiva i premoščivanje dentinskog otvora u razdobljima određenima različitim kriterijima bioloških testiranja (55,56).

Inicijalni su odgovori pulpnoga tkiva na adhezijske sustave minimalni, stvaranje dentinskoga mosta je tanko i polaganije, no sastavljeno je pretežito od tubulusnog dentina. Adhezijska smola veže se čvrsto za eksponirano pulpno tkiva i dentinski most se stvara izravno ispod smole bez vidljive pukotine i zacjeljivanje ne inhibira (57,58).

Adhezijska tehnika i suvremeni materijali stalnim tehnološkim napretkom imaju veliku indikacijsku širinu u cjelokupnoj stomatologiji. Nadoknada izgubljenoga tvrdog zubnoga tkiva (karijesom i traumom), uz estetsku korekciju oblika, pozicije, dimenzije i obojenja kliničke krune zuba, ostaju najšire indikacijsko područje kompozitnih materijala i adhezijskoga restorativnog postupka.

Koncepcija restorativne stomatologije unatrag se nekoliko desetljeća stalno mijenja i dopunjava. Pri tome adhezijska tehnika i uporaba adhezijskih sustava postaje sve važnije u kliničkoj proceduri. Adheziju na caklinu slijedi adhezija na dentin. Danas su univerzalni ili višenamjenski adhezijski sustavi koji se vežu na caklinu, dentin, amalgam, porculan, metal i kompozite preplavili dentalno tržište, tako da je koji put za većinu kliničara teško pravilno izabrati proizvod za uporabu u svakodnevnoj kliničkoj praksi.

Ovim pregledom razvoja strategije adhezije na caklinu i dentin želja nam je bila objektivno procijeniti i kritički raspraviti temeljna načela adhezijske restorativne i preventivne stomatologije, glede prednosti i nedostataka u suvremenoj kliničkoj praksi.

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Review of Adhesion to Enamel and Dentin

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Summary

To begin with emphasis is made of the importance of chemical pre-treatment of hard dental tissues during the process of restoration. The adhesive restorative process modifies the basic principles of cavity preparation and the basic principles of prevention of dental caries.

In the early fifties the procedure was restricted to treatment of enamel edges. The concentration of acid, the time required for etching as well as the ultramicroscopic appearance of etched enamel were at that stage the main objective of research.

Chemical treatment of dentin and adhesion of the composite restoration to dentin proved to be a challenging and complex task. Continuous progress and improvement of dental adhesives through five generations lead to optimal results regarding the clinical adhesive treatment procedure and the bond strength of the composite restoration opposed to that of demineralised dentin and enamel.

More recent histopathological studies of pulp tissue have shown that there was no decline in the function or vitality of pulp tissue, following the clinical adhesive procedure. Biocompatibility of the dental pulp against the chemical pre-treatment and the use of the adhesive systems is constantly being examined by certain parameters of biological testing in order to provide substantial support and confirmation of clinical achievements.

Key words: *adhesion, enamel, dentin, adhesive system.*

Acta Stomatol Croat
1998; 331—336

REVIEW
Received: November 28, 1997

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Introduction

Usage of acid during the clinical procedure of treating and preparing the enamel, because of bonding composite materials to the hard dental tissue, lead to significant changes in the clinical procedure of restoring lost hard dental tissue. Because of the possibility of bonding the restoration to enamel in a better and stronger way, it modified the basic principles of cavity preparation, principles of dental caries prevention, and aesthetic principles of restoration of lost hard dental tissue. The greatest advantage of treating the enamel with acid is reduction of microleakage and reduction of ingress of oral fluids and bacteria. Today, adhesion of restorative materials to enamel has become a routine clinical procedure, while adhesion of the restoration to dentin showed more difficulty and less predictability. Difficulty in bonding to dentin is the result of a complex histological structure, mineral composition and specific pulpo-dental physiology.

Chemical Treatment of Enamel

Basic principles of adhesive, preventive and restorative dentistry were introduced by Buonocore (1) in the middle of this century. He proposed usage of acid agents to change the enamel surface. The purpose of changing the surface was to produce conditions for better bonding of the restoration to the enamel cavity walls. His hypothesis was based on usage of acids in industry, to improve adhesion of paints and resin coatings to metal surfaces. Buonocore's pioneering work suggested more possibilities for using the technique of enamel acid-etching and stronger bonding of restoration to enamel. His ideas have been clinically confirmed in almost every field of dentistry.

Further work of Gwinnett and Matsui, Retief and Silvestron (2,3,4,5) brought more clarity and logic to the bonding mechanism of low-viscosity resin to etched enamel. Phosphoric acid etching of enamel removes about 10 nm of the enamel surface. Low-viscosity resin fills these microporosities by so called resin tags. It polymerises and forms a micromechanical bond to enamel. Etching increases the retentive surface and increases the level of surface-free energy of the enamel, resulting in optimal surface

wetting and good adhesion. Gwinnett's (6) studies deal with differences in phosphoric acid concentration and their effect on bond strength. It was reported that use of phosphoric acid concentrations between 30% and 40% achieves optimal etching results in view of adhesive demands. Results of Croatian researchers agree with the facts available from international literature (7). Besides the acid concentration, etching time was very often studied. Buonocore recommended 60 seconds, while Mardaga (8) reported in 1982 that etching time of 15 seconds is long enough. Laboratory investigations of bond strength of composite to enamel, and marginal microleakage, confirmed that shorter etching time is sufficient (9, 10). Clinical studies of a group of authors (11, 12) confirmed that the level of microleaking does not decrease proportionally with the shorter etching time. Contemporary choice of lower-concentration acids is based on simultaneous treatment of enamel and dentin (10% phosphoric acid, 10% maleic acid, 2.5% nitric acid). Many authors conclude that usage of low-concentrated acid results in significantly less bonding strength, but still adequate for good marginal seal and reduced microleakage (13).

Chemical Treatment of Dentin

Chemical treatment of the dentin cavity surface during the adhesive restorative procedure must correspond to a much more complexed structure of dentin tissue, by its histological, structural and mineral composition. While the enamel is 92% by volume inorganic content with primary component hydroxyapatite, the dentin is only 45% by volume inorganic content. Distribution of hydroxyapatite crystals in dentin is uneven within the organic collagen matrix. In addition, dentin is histologically and physiologically significantly connected to the dental pulp. Dentinal tubuli, by their content and function, require restorative treatment based on strict biological principles. Permeability of dentin is in correlation with the level of hypersensitivity of vital the tooth. Thus, by decreasing the permeability of dentinal tubuli, either by the natural process of sealing the tubuli, which takes longer time, or by different therapeutic treatments, the hypersensitivity of vital dental tissue can be successfully solved. The number of tubuli varies, depending on the place of investi-

gation (about 45 000 per mm² near the pulp, and about 20 000 per mm² near the dentinoenamel junction) (14). Pashley (15) calculated that tubuli take up 22% of surface close to the pulp, and just 1% of surface close to the enamel. Heymann and Bayne (16) established amounts of 28% and 4% of dentinal tubuli for the same areas. It is known that the variation of volume of dentinal tubuli by area reflects the permeability of dentinal tissue. In this way, the permeability of occlusal dentin is greater at the pulp horns than in the centre of the occlusal surface. In the same way, the proximal dentin is more permeable than occlusal dentin, and coronal dentin is more permeable than dentin of the tooth root. The liquid in the dentinal tubuli is under slight but constant pressure from the pulp. The evaluated intrapulpal pressure is about 25-30 mm Hg (17).

The chemical preparation of dentinal cavity walls and the bonding of the restoration to the dentin is complicated by the presence of the smear layer. This layer, 0.5-5 μm thick, seals dentinal tubuli orifices, and acts as a diffusion barrier decreasing the permeability of dentin. Thickness, composition and appearance of the smear layer vary depending on the specificity of the substrate, treatment technique used, and on the instruments used during the cavity preparation. In that layer bacteria can survive and multiply, thus representing the permanent cause of dental pulp inflammation (18, 19). The problem and specificity of bonding the filling to the dentinal tissue was solved by group investigation work.

We can follow the development of resin-based dental adhesives for the last four decades, grouping them into generations. In the first generation, 40 years ago, resin that contains dimethacrylate of glycerophosphoric acid was recommended. It can be bonded to the dentinal surface treated with hydrochloric acid. The disadvantage of this resin was its great sensitivity to water, so Bowen synthesised N-Phenylglycine glycidyl methacrylate (NPG-GMA), surface active comonomer, which theoretically can produce water-resistant chemical bonding of resin to the dentinal calcium (12).

The second generation of dental adhesives developed in the early eighties, and were mostly halophosphoric esters of unfilled resins, such as bisphenol glycidyl methacrylate (bis-GMA) or Hydroxyethyl methacrylate (HEMA). The bonding mechanism includes surface wetting phenomenon, as well as ionic interaction between phosphate groups and den-

tinal calcium. The main reason for poor performance of those bonding agents is the fact that they bond to the smear layer, not to the dentin itself. Thus, their bond strength is limited by the cohesive force of the smear layer and by adhesion to the underlying dentin (20).

The third generation of dental adhesives, developed recently, requires modification or complete removal of the smear layer, thus making it possible for resin to penetrate the dentin underneath the smear layer. Bond strength to the dentin is close to the typical bond strength of resin to the treated enamel. This generation of dental adhesives proved to be more efficient in decreasing the marginal microleakage at the edges of the dentin or cement. Further advantages are conservation and support of the hard dental tissue structure. There is also greater prevention of tooth caries. The improved bond strength of the contemporary dental adhesives has been confirmed by numerous clinical and laboratory investigations. The previously mentioned acid-etching technique also becomes the rule in this generation.

In 1979, Fusayama et al., first started with chemical treatment of dentin, which later became very common procedure in Japan (21). However, the concept of total acid treatment (called total etch) was accepted in the USA some time later (20, 22).

The move towards total etch was a radical step in American restorative dentistry, because treating the dentin with acid was traditionally discouraged. Results of studies carried out in the seventies showed that dentin treated with phosphoric acid lead to pulp inflammation (23, 24). However, only a very small amount of acid actually penetrates the dentin, thus it seems impossible that acid is directly responsible for any pulp damage.

The facts we now have at our disposal mostly conclude that insufficient marginal sealing at the permanent restoration is the main cause of pulp inflammation. If the restoration seals well, preventing ingress of oral fluids and bacteria towards the pulp, then mild or no inflammation can develop (25, 26, 27).

The fourth generation of dental adhesives use conditioners which remove the smear layer and demineralize the dentin surface to a certain depth.

Although various acid-etchants, primers and resins can be used, the way that different adhesives bond to etched dentin is in many ways similar (28).

Acid-etching, the first step of the adhesive procedure, removes the smear layer, exposes the dentinal tubuli, demineralises the intertubular and peritubular dentin, and so increases the permeability of dentin. Hydroxiapatite crystals dissolve and can be rinsed away by water - which is the second step of the adhesive procedure. Due to loss of inorganic support this leaves a collapsed collagen matrix (29, 30). The primer - in the third step of the adhesive procedure - moistens and penetrates the collagen matrix, "elevating" it almost to its original level and volume. By moisturising, the primer also increases surface energy. The exchange of ions which takes place is simplified by the presence of water (31). The fourth step of the procedure is a application of an unfilled resin (bond), which penetrates the dentin previously treated with a primer, copolymerises with the primer and forms a mixed layer of collagen and resin, the so-called reinforced resin layer, infiltrated resin layer or just simply the hybrid layer (32, 33, 34). Nakabayashi et al. were first to describe the formation of this hybrid layer of dentin and resin, which in the majority of adhesive systems is considered the primary bonding process (35).

The complexity of the clinical procedure required by earlier dental adhesives and the constant aspiration for improvement of the results of the adhesive process resulted in a fifth generation of dental adhesives.

Pre-treatment of dentin is vital for fifth generation adhesives and is responsible for the clinical success of contemporary adhesive systems. Due to the pre-treatment, the complexly structured and hydrophilic dentinal substrate becomes more receptive to the adhesive bonding of the composite restoration (36).

The chemical pre-treatment of the smear layer during the chemical adhesive procedure offers three strategic possibilities.

The aim of the first strategy is to modify the smear layer and incorporate it into the bond.

The second strategic possibility simultaneously requires the removal of the whole smear layer and demineralization of the dentinal surface.

The latest, contemporary fifth generation of adhesive systems, through intermediate strategy, enables milder acids and shorter etching time to cause

partial demineralization through out the whole area of the smear layer and of the dentine surface directly below the smear layer. The most recent improvements in the technology of dental adhesives, include the smear layer as a substrate through which the bonding takes place. This gives them a new and more complex role, because the technologically improved products are able to act on the dentinal matrix through the smear layer (37).

The clinical procedure itself determines the choice of the adhesive system used, presently available on today's market.

In other words, we can distinguish adhesive systems in which the acid and the primer are combined, so that the clinical procedure can be performed in only two bonding steps:

- 1st step = acid / primer
- 2nd step = adhesive resin

There is another possibility, where the primer and adhesive resin are combined, and the clinical procedure is also carried out in two steps:

- ∇ 1st step = acid
- ∇ 2nd step = primer / adhesive resin (38).

Adhesive systems in the first group (•) contain the acidic phenyl-P monomer and HEMA, with the purpose of simultaneously etching and priming enamel and dentin. The advantage of these systems, apart from simplifying and reducing the time of the procedure, is that they superficially demineralise dentin and simultaneously penetrate dentin with monomers which polymerize in-situ (in its original place). Such continuity, from unchanged dentin to the adhesive resin, does not contain a layer of unpolymerized hydrophilic monomers, which would be highly sensitive to hydrolysis and formation of micropores and microrleakage (33, 39, 40). Further advantage is that it does not require rinsing with water, but only needs to be slightly air dried so as to avoid rinsing away of the primer, HEMA.

Furthermore, apart from containing acidic monomers such as 20% phenyl-P and the presence of 30% HEMA, this solution contains water and alcohol. This volatile dissolution vaporises when applied to the surface and is air dried. Because the contraction of the demineralized dentin the matrix (collagen) is proportional to the amount of dissolvent that can vaporise, the dentin surface treated by this system should experience less of contraction than

when treated by the previous generations. This, in turn, should reduce the formation of marginal leakage between the composite restoration and the dentino-enamel walls (41).

There are a few competitive processes occurring at the same time within this process. The mineral component of dentin needs to be dissolved to make room for infiltration of the adhesive resin. These resin monomers must diffuse inwards at the same time as the calcium and phosphate ions diffuse outwards. Each process is probably retarded by the other, leading to lesser demineralization of dentin and a lighter infiltration by the resin. Nevertheless, it is most important that marginal microleakage is generally less than during the use of earlier adhesive systems. Part of the reason for this is that the resin infiltrated smear layer is incorporated within the hybrid layer and acts as an inner low modular liner which decreases the chance of microleakage and enhances the durability of the composite restoration (41, 42, 43, 44).

In the second group (∇) of previously mentioned adhesive systems, a mild action (10-15% phosphoric acid) is firstly applied to enamel and dentin for a period of 15 seconds (total etching) (45). After rinsing with water, in the second step of the adhesive procedure, the application of the system follows which incorporates both primer and adhesive resin, to enamel and dentinal surface. Combined in this way, primer/adhesive resin contains well balanced hydrophilic and hydrophobic monomers. Because the BPDm monomer is so hydrophilic, the mixture assists the infiltration of porous dentinal substrate, while the hydrophobic monomer Bis-GMA assures an adequate layer of solid resin. A thin unset layer (restrained by oxygen) remains on the surface of this layer and by strong bonding contributes to the adaptation of the composite material (46, 47).

Neutralisation of the unwanted stress of polymerization is achieved by layering technique, application of a composite of low viscosity with a low module of elasticity to the surface of the adhesive resin. The low viscosity composite, because of its low module of elasticity, compensates for the strength by its flexibility and lowers the possibility of microleakage (48). By increasing the strength and the constancy of the adhesive bond, it assures durability of the complete composite reconstruction.

The strength of the bond which exceeds 20 MPa (the contraction force during the polymerization of the composite, which can separate the composite from dentin is approximately 18 MPa) is achieved by micromechanical saturation of demineralised areas by the adhesive resin (29).

The results of recent studies *in vitro* show that infiltration of dentinal tubuli and intertubular dentin by infiltrative resin (hybridization) is responsible for the total bonding of resin to dentin. The bond strength is weaker when there is no hybrid layer formed by the bonding substance or when the dentin surface has previously been in contact with eugenol-based cement (48, 49).

The SEM also reveals that many adhesives form resin tags which penetrate deep into dentinal tubuli. These terminal points are impressive in appearance, but unless tightly bonded to tubuli walls they are barely or not at all retained. Even the conventional (hydrophobic) resin which bonds to enamel will form long resin tags that infiltrate previously etched dentin, but will not produce a considerable bonding force because the resin does not moisten, that is, it does not bond adequately to the tubuli walls. Further research regarding the importance of resin tags indicated that there are only small scale bonding forces acting on deep, tubuli rich dentin (50).

Finally, resin tags that form *in vivo* are probably shorter than those which form on extracted teeth, because the dentinal tubuli are filled with a pressurized solution, which does not allow deep penetration of the resin (51, 52, 53).

SEM and TEM studies also revealed a great problem, i.e. the possibility of disproportion between the depth of demineralization and penetration of the resin through the etched dentin (54). The strength of the adhesive bond to the etched dentin could be based upon its ability to totally replace the dissolved hydroxiapatite by polymerized resin. However, if the depth of demineralized dentin is so great that the porous zone cannot be adequately impregnated by the resin, the fragile collagen layer can collapse and with time degenerate. Although contemporary primers are highly hydrophilic, adequate impregnation by resin can require the use of etching technique, which does not result in such a great depth of demineralization. A shorter treatment period or less aggressive acids could be used in the future (47).

In the future, changes and technological improvement can be expected exactly on the same level of the same total adhesive procedure. The parameters which determine the success of the adhesive process are the thickness of the hybrid layer and the strength of the bond in the interdiffusal zone. Contemporary dental adhesive systems with technological innovations, aspire to improve this segment of the whole concept of adhesive procedure.

More recent histopathological studies of the pulp and evaluation of biocompatibility of the chemical pre-treatment of dentin show that neither the use of adhesive systems nor direct overlaying of the exposed pulp, reduce the function, vitality or biological potential of the pulp in any way. There was no sign of interference with the healing process of the dental pulp or bridging of the dental orifice in the period of time defined by distinct criteria of biological testing (55, 56). Initial response of the pulp to the adhesive system is minimal. The dentinal bridge is thinner and forms slower, but consists mostly of tubulus dentin. The adhesive resin bonds strongly to the exposed pulp and dentinal bridge forms directly below the resin without leaving a visible crevice or inhibiting the healing process (57, 58).

The adhesive technique and contemporary materials, due to constant technological improve-

ts, have a broad area of indication in the whole field of dentistry. Replacement of lost hard dental tissues (due to caries or trauma), by aesthetic correction of shape, position, dimension and staining of the clinical crown of the tooth, remain the widest area of indication for composite materials and adhesive restorative process.

The concept of restorative dentistry has seen constant changes and improvements in the last few decades. In the process, the adhesive technique and the application of adhesive systems have become more significant in the clinical procedure. Adhesion to dentin is followed by adhesion to enamel. Today, universal and multipurpose adhesive systems which bond to enamel, dentin, amalgam, porcelain, metal and composites are so numerous on the dental market that it is often difficult for dentists to select a product most applicable in everyday clinical practice.

The aim of this review of the development of the strategy of adhesion to enamel and dentin, was to objectively evaluate and critically argue the basic principles of adhesive, restorative and preventive dentistry in terms of the advantages and disadvantages of contemporary clinical procedure.