



ELSEVIER

Available online at www.sciencedirect.com

SciVerse ScienceDirect

journal homepage: www.intl.elsevierhealth.com/journals/dema

A randomized controlled 5-year prospective study of two HEMA-free adhesives, a 1-step self etching and a 3-step etch-and-rinse, in non-carious cervical lesions

Jan W.V. van Dijken*

Department of Odontology, Faculty of Medicine, Umeå University, Umeå, Sweden

ARTICLE INFO

Article history:

Received 6 April 2013

Accepted 9 August 2013

Keywords:

Adhesion

Cervical

Clinical

Composite resin

Dental resin

Restoration

Self-etch

ABSTRACT

Objective. The aim of this study was to evaluate the 5 year clinical dentin bonding effectiveness of two HEMA-free adhesives in Class V non-carious cervical lesions.

Material and methods. A total of 169 Class V restorations were placed in 67 patients with a self-etching adhesive (G-Bond; 67), a 3-step HEMA and TEGDMA free etch-and-rinse (cfm; 51) and a control HEMA-containing etch-and-rinse adhesive (XP Bond; 51) in non-carious cervical lesions without intentional enamel involvement. The restorations were evaluated at baseline and yearly during a 5 year follow-up with modified USPHS criteria. Dentin bonding efficiency was determined by the percentage of lost restorations.

Results. During the 5 years, 159 restorations could be evaluated. Good short time dentin retention was observed for the three adhesives, there all adhesives fulfilled at 18 months the full acceptance ADA criteria. At 5 years a cumulative number of 22 lost restorations (13.8%) was observed. The HEMA-free adhesives showed significantly higher dentin retention compared to the HEMA-containing one. Loss of retention was observed for 5 G-Bond (7.9%), 4 cfm (8.3%) and 13 XP Bond (27.1%) restorations ($p < 0.05$). No post-operative sensitivity was reported by the participants. No secondary caries was observed.

Significance. The durability in non-carious cervical lesions of the HEMA-free adhesives was successful after 5 years. Despite concerns which have been raised, showed the 1-step SEA one of the best reported clinical dentin bonding effectiveness.

© 2013 Academy of Dental Materials. Published by Elsevier Ltd. All rights reserved.

1. Introduction

Adhesive systems have revolutionized and are routinely used in operative dentistry to improve retention, sealing

and esthetics of resin-based materials. The interaction with the tooth substrates is today based on the etch-and-rinse or the non-rinse self-etch approach. Self-etching adhesives (SEA) contain acidic monomers which simultaneously condition and prime the smear layer

* Correspondence to: Department of Odontology, Dental School Umeå, Umeå University, 901 87 Umeå, Sweden. Tel.: +46 90 7856034/7856226.

E-mail address: Jan.van.Dijken@odont.umu.se

0109-5641/\$ – see front matter © 2013 Academy of Dental Materials. Published by Elsevier Ltd. All rights reserved.

<http://dx.doi.org/10.1016/j.dental.2013.08.203>

and underlying tooth tissues. Clinical advantages suggested are its decreased technique sensitivity, decreased application time and decreased risk for re-contamination of the etched tooth surfaces and/or collapse of the collagen network after air drying. Disadvantages reported are that 1-step SEA's are more hydrophilic and can absorb rapidly water which result in higher solubility and water uptake. This may result in polymer swelling, plasticization and weakening of the polymer network [1,2]. One-step SEA's may act as semi permeable membranes, permitting water movement through the layer even after polymerization [3].

Diffusion of monomers into the demineralised tooth tissues to create a hybrid layer is considered to be the essential mechanism of adhesive bonding. HEMA (2-hydroxyethyl methacrylate), an effective hydrophilic methacrylate primer monomer, is frequently present in dental adhesives. It improves dentin bond strength due to its wetting enhancement effect and promotes diffusion of co-monomers by expanding the demineralised collagen [4–6]. In 1-step SEA adhesives HEMA maintain the resin monomers and water in one solution and prevent phase separation [6,7]. However, high HEMA content promotes water uptake and subsequent gradual hydrolytic degradation of the polymers, swelling and staining [8]. Increased water uptake might accelerate the reduction of mechanical properties of the SEA [9]. Omission of HEMA in adhesives leads to phase separation between water and the adhesive monomers, which requires strongly air blowing to remove the water-containing droplets from the interface [6,10,11].

Methacrylate monomers are potent contact allergens and especially the low weight monomer HEMA is considered as one of the most potent ones [12,13]. Fast penetration of non cured monomers through the skin and gloves cause contact dermatitis in dental personal [14–16]. In addition another commonly used low viscous monomer TEGDMA has been associated with cytotoxic reactions [17,18]. Unpolymerized HEMA remain chemically and physically unchanged and can leach up to 30 days [19]. Organic solvents can solve higher amounts compared to water or saliva only [19].

Assessing the bonding effectiveness of adhesives in vitro showed that 3-step etch-and-rinse adhesives performed best, irrespective of bond strength test [20]. Two-step SEA conducted better than the all-in-one systems [21,22]. However, laboratory tests cannot predict the clinical situation and Class-V clinical trials remain therefore the ultimate studies to test adhesives [20,23]. In an earlier review of clinical Class V studies it was concluded that etch-and-rinse adhesives were more efficient than SEA's [6]. Lower microtensile bond strength have been reported for SEA especially to enamel [20,24]. However, recent Class V clinical trials showed that 1-step SEA's substantially improved with annual failure rates in line with the etch-and-rinse adhesives [20,25].

The disadvantages of HEMA have led to the introduction of HEMA-free less hydrophilic adhesives which may show reduced water sorption, higher stability of mechanical properties, stability of the interfacial bond, improvement in bonding durability and reduced allergenic potential [19,26]. Short time evaluation of HEMA-free adhesives showed a satisfactory performance [27–30].

Table 1 – Baseline data, distribution and lesion characteristics, of the lesions included.

	G-Bond	cfm	XP Bond
Teeth			
Incisor/cuspidate	25	16	23
Premolar	30	19	20
Molar	12	16	8
Jaw			
Maxilla	34	40	37
Mandible	33	11	14
Lesion size			
Small	11	7	18
Medium	23	33	19
Large	33	11	13
Lesion depth			
Superficial	24	28	33
Medium	27	17	13
Deep	16	6	5
Degree of sclerosis			
0%	19	25	26
<50%	6	8	10
>50%	42	18	15

The purpose of this study was to determine the long term clinical bonding durability of a 1-step HEMA-free SEA, a 3-step HEMA/TEGDMA-free etch-and-rinse and a 3-step HEMA containing etch-and-rinse adhesive in Class V non-carious cervical lesions without using retention of external lesion surface area. The null hypothesis tested was that there is no difference in durability of the clinical dentin bond formed with the HEMA-free and HEMA-containing adhesives.

2. Material and methods

During the period May 2006–October 2007, all patients attending the author's PDHS clinic at the dental school Umeå, for who treatment of non-carious cervical lesions was indicated were requested to participate in the study. No patient was excluded because of caries activity, periodontal condition or parafunctional habits. All participants received informed consent and the study was approved by the commission for medical ethics of the University of Umeå. A total of 169 Class V restorations were placed in 67 patients, 34 men and 33 women with a mean age of 64.7 year (min–max 39–84), who needed treatment of non carious cervical lesions. All restorations were placed by one experienced operator, familiar with adhesive dentistry, in dentin lesions without any intentional enamel involvement. Pre-operatively, the lesions were categorized by the operator compared to lesion models in terms of depth (shallow, moderate, large) and size (small, moderate, large) of the lesion, the area of the dentin surface estimated as sclerotic tissue (0, <50%, >50%) (Table 1) [31].

A single-step, self-etching HEMA-free primer (G-Bond, GC Corp, Tokyo, Japan), a 3-step HEMA/TEGDMA free etch-and-rinse (cfm, Saremco AG, Rebstein, Switzerland) and a 2-step HEMA-containing etch-and-rinse adhesive (XP Bond, Dentsply/DeTrey, Konstanz, Germany) were evaluated in combination with two restorative resinous materials (Table 2). The resin composite Gradia Direct (GC Corp) was used in combination with G-Bond, and els (extra low shrinkage; Saremco) in combination with the two other adhesives. After the

Table 2 – Composition and application methods of the adhesive systems tested.

Adhesive	Composition	Manufacturer	Application
G-Bond 1-step SEA	4-MET, UDMA, TEGDMA, phosphoric acid monomer, photo-initiator, stabilizer, fumed silica filler, acetone, water pH= 2.0	GC Corp, Tokyo, Japan lot 0506141	Apply adhesive on the air dried dentin surface. Agitating for 5 s and reapply if necessary. Leave undisturbed for 10 s. Dry strongly with air with maximum pressure for 5 s.
cfm adhesive system	<i>cmf etch</i> : buffered phosphoric acid (pH= 1.5) <i>cmf primer</i> : metacrylated phosphoric salt, alcohol, acetone, CQ, co-initiator <i>cmf bonding</i> : hydrophilic ethoxylated Bis-GMA, silanized barium glass, CQ, co-initiator.	Saremco AG, Rebstein, Switzerland primer lot 06.2011.01 bonding lot 06.2011.05	Etch for 15 s enamel and dentin Rinse for 30 s Air dry for 5 s Apply <i>cmf primer</i> using a rubbing motion for 30 s, dry for 5 s and light cure for 20 s Apply <i>cmf bonding</i> using a rubbing motion for 20 s Light cure for 30 s
XP Bond 2-step etch-and-rinse	Conditioner: 36% phosphoric acid: primer: TCB, PENTA, UDMA, TEGDMA, HEMA, butylated benzenediol (stabilizer), ethyl-4-dimethylaminobenzoate, CQ, amorphous silica, tertiary butanol	DeTrey Dentsply, Konstanz, Germany lot 0701000	Apply conditioner to the lesion surface for 15 s Rinse thoroughly for 10 s and dry but do not desiccate Apply adhesive and leave undisturbed for 20 s Air blow for min 5 s. Light cure for 20 s.
els (extra low shrinkage) resin composite Gradia Direct resin composite	Bis-GMA, Bis-EMA, silanized barium glass, catalysts, inhibitors, pigment	Saremco AG, Rebstein, Switzerland lot 10.2010-006 GC Corp, Tokyo, Japan lot 121222	
Abbreviations: HEMA, 4-MET 4-methacryloxyethyl trimetellitic acid; PENTA, phosphoric acid modified acrylate resin; TCB, resin carboxylic acid modified dimethacrylate; TEGDMA, triethyleneglycol dimethacrylate; Bis GMA, bisphenol A-glycidyl methacrylate; Bis EMA, Bisphenol A ethoxylate dimethacrylate; UDMA, urethane dimethacrylate; CQ, camphorquinone.			

operative procedure decision, the lesions were filled in randomly order by three adhesive systems. In subjects with two or more lesions, different adhesives were applied in the lesions to make intra-individual comparison possible.

2.1. Operative procedure

After randomization, the lesions were slightly roughened by a diamond bur before application of the adhesive systems to create a surface smear layer. The operative field was isolated with cotton rolls and a saliva suction device. The adjacent gingiva was retracted by gingival retraction instruments when necessary to secure unrestricted contamination free access to the field. In order not to enlarge the retention area, according to the ADA guidelines, no enamel bevels were placed or enamel etched, nor were other ways used to get extra mechanical retention. The materials were applied on the dentin lesions according to the manufacturer's instructions (Table 2). After cure of the adhesive, the lesions were in many cases filled with an oblique incremental layering technique there the first oblique layer was placed in the incisal/occlusal part of the lesion. Each layer was cured for 20–40 s using a well controlled light-curing device (Astralix 7, Vivadent, Schaan, Liechtenstein; Demetron light meter, Kerr, Orange, CA, USA). After polymerization, the restorations were finished with fine diamond burs (DZ, Berlin, Germany) and polishing stones under water spray (Brownie, Shofu Dental Co, Ratingen, Germany).

2.2. Evaluation

The restorations were evaluated at baseline, and then blindly at, 6, 12, 18 and 24 months and then yearly during the 5 years by the operator and at regular intervals by two calibrated evaluators (inter- and intra-examiner Cohen's Kappa values of loss of retention criteria were >0.94). During evaluation, the dental assistant wrote the scores in the participants form. The evaluator(s) had no knowledge of the materials to be evaluated or earlier evaluation scores. Slightly modified USPHS criteria were used (Table 3) [32]. Postoperative sensitivity was registered by questioning the participants at the evaluation visits or by self-reporting in between the visits.

2.3. Statistical analysis

The IBM SPSS (Statistical Package for the Social Sciences) statistics version 19 was used to process the data. The characteristics of the restorations were described by descriptive statistics using frequency distributions of the scores. Cumulative retention failures were calculated by dividing the number of lost restorations at the recalls by the total number evaluated at each of the recalls. Survival functions were given by descriptive statistics. Differences in loss of retention between the adhesives were tested with Friedman two-way analysis of variance test. The null hypothesis was rejected at 5% level.

Table 3 – Criteria for direct clinical evaluation with slightly modified USPHS criteria (van Dijken, 1986).

Category	Score		Criteria
	Acceptable	Unacceptable	
Marginal adaptation	0		Restoration is contiguous with existing anatomic form, explorer does not catch
	1		Explorer catches, no crevice is visible into which explorer will penetrate
	2		Crevice at margin, enamel exposed
		3	Obvious crevice at margin, dentin or base exposed
		4	Restoration mobile, fractured partially or totally
Color match	0		Very good color match
	1		Good color match
	2		Slight mismatch in color, shade or translucency
	3		Obvious mismatch, outside the normal range
	4		Gross mismatch
Marginal discoloration	0		No discoloration evident
	1		Slight staining, can be polished away
	2		Obvious staining can not be polished away
		3	Gross staining
Surface roughness	0		Smooth surface
	1		Slightly rough or pitted
	2		Rough, cannot be refinished
		3	Surface deeply pitted, irregular grooves
Caries	0		No evidence of caries
		1	Caries is evident contiguous with the margin of the restoration

Table 4 – Relative cumulative frequencies (%) of lost restorations during the 5 year follow-up.

	6 m	12 m	18 m	2 yr	3 yr	4 yr	5 yr
G-Bond	0	0	1.5	1.6	3.2	6.3	7.9
cfm	2.0	2.0	4.0	4.0	4.2	6.3	8.3
XP Bond	4.0	4.0	8.0	8.0	16.7	22.9	27.1

3. Results

During the five year evaluation, 4 patients with 10 restorations (4 G-Bond, 3 cfm, 3 XP Bond) could not be evaluated at all recalls because of extraction, crown treatment or death of the patient. A cumulative number of 22 lost restorations (13.8%) was observed during the follow-up. The loss rates were 5 G-Bond (7.9%), 4 cfm (8.3%) and 13 XP Bond (27.1%) restorations ($p < 0.05$). Loss of retention was the only reason of failure observed during the follow up. Cumulative absolute and relative loss rate frequencies for the 3 adhesives at the different recalls are shown in Table 4. No post-operative sensitivity was reported by the participants.

The relative frequencies of the evaluated variables marginal adaptation, color match and marginal discoloration at 18 months, 3 and 5 years are shown in Table 5. All acceptable restorations, except for two restorations (1 cfm, 1 XP Bond) with score 1, received at 5 years a score zero for surface roughness. No secondary caries was observed contiguous the restorations during the follow up. A slightly, but not significant, higher loss of retention was observed in medium sized lesions (14.7%) compared to small (11.1%) and large (10.5%) lesions. Almost double as many lost restorations were found in the superficial and medium depth lesions, 15.5% and 12.3% respectively compared to the deep lesions (7.4%). Significant less lost restorations were found in the lesions with >50%

degree of sclerosis compared to the other groups: 0% 15.7%, <50% 16.7%, >50% 9.3% ($p < 0.05$). A higher loss rate was found in the anterior teeth lesions (15.6%) compared to posterior teeth lesions (premolar 11.6%, molar 11.1%). Lesions in the maxilla showed a 15.3% and in the mandible a 10.4% loss rate frequency.

4. Discussion

Adhesive technology is rapidly evolving and adhesive formulations are replaced frequently by new ones. The latest innovations have been based on reducing the handling steps and improving biocompatibility. Unfortunately, many new systems have no independent clinical evaluations and especially, like for the tested adhesives, long term studies are missing. Most bond strength test are performed immediately after bonding and only a few studies mimic, and then only partially, the chemical and physical stress factors occurring within the oral environment like chewing loads, pH- and temperature changes [33]. To evaluate durability of the interfacial bond and the effect of exposure to oral fluids over longer time periods the ultimate proof of performance should be observed in Class V non-cariou lesion studies as recommended by the ADA [34–37]. Peumans et al. described these clinical studies as time consuming and difficult to obtain adequately high recall rates in order to obtain sufficient clinical validation [34]. The

Table 5 – Relative frequencies of the evaluated scores for marginal adaptation, color match and marginal discoloration at baseline, 18 months, 3 and 5 years (%).

		0	1	2	3	4
Marginal adaptation	G-Bond baseline	100	0	0	0	0
	cfm baseline	98	2	0	0	0
	XP Bond baseline	96	4	0	0	0
	G-Bond 18 mth	81.8	16.7	0	0	1.5
	cfm 18mth	63.2	32.6	0	0	4.2
	XP Bond 18mth	63.3	28.5	0	0	8.2
	G-Bond 3 yr	92.2	4.7	0	0	3.1
	cfm 3 yr	64.6	31.3	0	0	4.2
	XP Bond 3 yr	56.3	27.1	0	0	16.7
	G-Bond 5 yr	71.5	20.6	0	0	7.9
	cfm 5yr	60.4	29.2	2.1	0	8.3
	XP Bond 5 yr	47.9	25.0	0	0	27.1
Color match	G-Bond baseline	64.2	34.3	1.5	0	0
	cfm baseline	62.0	36.0	2.0	0	0
	XP Bond baseline	58.0	38.0	4.0	0	0
	G-Bond 18 mth	38.5	53.9	9.2	0	0
	cfm 18 mth	63.8	29.8	6.4	0	0
	XP Bond 18 mth	44.4	46.7	8.9	0	0
	G-Bond 3 yr	32.3	50.0	17.7	0	0
	cfm 3 yr	28.3	54.3	17.4	0	0
	XP Bond 3 yr	35.0	45.0	15.0	0	0
	G-Bond 5 yr	27.6	53.4	19.0	0	0
	cfm 5 yr	27.3	45.4	27.3	0	0
	XP Bond 5 yr	37.1	45.7	17.1	0	0
Marginal discoloration	G-Bond baseline	100	0	0	0	
	cfm baseline	100	0	0	0	
	XP Bond baseline	100	0	0	0	
	G-Bond 18 mth	87.7	7.7	4.6	0	
	cfm 18mth	93.6	4.3	2.1	0	
	XP Bond 18 mth	88.9	4.4	6.7	0	
	G-Bond 3 yr	87.1	6.5	6.5	0	
	cfm 3yr	76.1	21.7	2.2	0	
	XP Bond 3 yr	77.5	15.0	7.5	0	
	G-Bond 5 yr	72.4	10.4	17.2	0	
	cfm 5 yr	81.8	13.6	4.6	0	
	XP Bond 5 yr	85.7	5.7	8.6	0	

long term recall rates in Umeå are in all studies >80%, in contrast to the low figures wrongly referred by Peumans et al., while the recall rate in the present study was 94% [31,36–38].

The adhesives tested were launched only recently and therefore only few short or medium-long clinical reports are available. The one step self-etch adhesive showed satisfactory short time performance (1–3 year) investigated by 4 research groups [28–30,39]. In three of these studies the retention surface area of the lesion was increased by preparing an enamel bevel and/or etching of the enamel incisal of the lesion margin with phosphoric acid [27–29]. Because bonding to dentin remains more challenging, an additional enamel bond will decrease the information on dentin bonding of the adhesive. It has been claimed that the reduction in bond strength over time may not be as great in restorations there an enamel seal can be maintained [40]. However, the combined bond to enamel and dentin will camouflage the role of the dentin bond in the clinical effectiveness. Since many years we chose in the Umeå biomaterial research group to investigate the more degradation sensitive dentin-only design according to the ADA guidelines for clinical evaluation of bonding systems [34]. These lesions will consist of dentin in more than 85% of the lesion surface area and are therefore a unique dentin

bonding substrate. In the first enamel–dentin bonding study Van Landuyt et al. reported after 3 year similar success in clinical performance for G Bond as the so called “golden standard” Optibond FL with retention rates of 94.7% and 94.0%, respectively [29]. The majority of these restorations were lost after 2 or 3 years of clinical service. The second study found after 2 years a 2% loss of retention [28]. This can be compared with the 2 and 3 year retention rates of 98.4% and 96.8%, respectively, observed in this study for the SEA. Burrow and Tyas [30], who used as in the present study lesions without increasing the retention area, reported in a small restoration sample 100% retention for G-Bond after 3 years. Deterioration of the enamel margins bonded with G Bond, as small marginal defects have been reported in the enamel–dentin design studies, which were easily removed by polishing [29]. Blunck et al. [41] reported no retention failures for the 2-step etch-and-rinse XP Bond after only 6 months. No other clinical evaluation of XP Bond and cfm have been reported.

These short time figures indicated that the interfacial bond of the adhesives was strong enough to withstand the immediate physical forces and stress formation during and post shrinkage as well as the first year's intra oral expansion and contraction stresses by temperature and chewing

Table 6 – Published annual failure rates of in Umeå tested adhesive systems in similar dentin-only cervical non-carious lesion studies after 5 year follow up periods. AFR = annual failure rate. For etch-and-rinse systems 35–37% phosphoric acid has been used for most systems, in other cases the acid is given in parenthesis.

Classification	Adhesive system (reference)	Lost restorations after 5 years (%)	AFR (%)	Manufacturer
4 step etch & rinse	Syntac classic [37]	23.4	4.7	Ivoclar/Vivadent, Schaan, Liechtenstein
3 step etch & rinse	cfm (buffered phosphoric acid)	8.3	1.7	Saremco AG, Rebstein, Switzerland
	Optibond [37]	13.8	2.8	Kerr Corp, Orange, USA
	Clearfil LB [36]	14.0	2.8	Kurary Co. Ltd., Osaka, Japan/Cavex, Holland
	Allbond 2 [36]	14.6	2.9	Bisco, Schaumburg, IL, USA
	XP Bond	27.1	5.4	DeTrey/Dentsply, Konstanz, Germany
	Scotchbond MP (Maleic acid) [37]	40.0	8.0	3M, St. Paul, MN, USA
	Permagen [37]	52.3	10.5	Ultradent Prod Inc, South Jordan, Utah, USA
	Dentesive(EDTA) [36]	89.5	17.9	Hereaus–Kulzer GmbH, Wehrheim, Germany
2 step etch & rinse	PQ 1 [72]	37.7	7.5	Ultradent Prod Inc, South Jordan, Utah, USA
	Dentesive 2 (Maleic acid) [36]	68.6	13.7	Hereaus–Kulzer GmbH, Wehrheim, Germany
	Gluma 2000 (Oxalic acid) [36]	73.0	14.6	Bayer Dental, Leverkusen, Germany
2-step self etch	Clearfil SE [72]	12.7	2.5	Kurary Co. Ltd., Osaka, Japan/Cavex, Holland
	ART [36]	16.7	3.3	Colténe, Altstätten, Switzerland
	PUB3 [36]	38.1	7.6	DeTrey/Dentsply, Konstanz, Germany
1-step self etch	G-Bond	7.9	1.6	GC, Tokyo, Japan
	Xeno III [42]			DeTrey/Dentsply, Konstanz, Germany
	Tetric Ceram	9	1.8	
	Dyract	19.1	3.8	
	PSA [37]	16.0	3.2	DeTrey/Dentsply, Konstanz, Germany
RMGIC	Fuji Bond LC [38]			GC International, Tokyo, Japan
	Tetric Ceram	5.9	1.2	Vivadent, Schaan, Liechtenstein
	Hytac	21.2	4.2	ESPE, Seefeld, Germany
RMGIC	Vitremer [36]	16.3	3.3	3M, St. Paul, MN, USA

forces. It is well known that factors like further water sorption, influence of chemical agents and bacterial products in the mouth challenge the hybrid layer and the resin bond. This will result in continuous degradation of the bonding effectiveness of adhesives resulting in increasing failure rates [36,37,42]. The decrease in bonding effectiveness of the 2-step HEMA-containing etch-and-rinse adhesive was higher and started earlier, while it increased for the HEMA-free adhesives first after 3 years indicating again the necessity of long time evaluations [43]. After 5 years, the clinical success of both HEMA-free adhesives was significantly higher than for the HEMA-containing 2-step etch-and-rinse adhesive. The null hypothesis was therefore rejected. Annual failure rates (AFR) in the 5-year follow up for G-Bond, cfm and XP Bond were 1.6%, 1.7% and 5.4%, respectively. These numbers can be compared with AFR's of other adhesives, earlier tested in a similar way in 5 year evaluations as in the present study, published (Table 6) [38–40,42]. The best performance was shown for a resin modified glass ionomer bonding system (Fuji Bond LC), closely followed by two 1-step SEA investigated (G Bond and Xeno III), and a 3-step etch-and-rinse system (cfm). A slightly higher AFR was shown for the two golden standard adhesives, the 2-step SEA Clearfil SE and the 3-step etch-and-rinse adhesive Optibond. The high variation in clinical effectiveness within each of the different adhesive classes has been shown earlier [36,37].

The need of HEMA-free adhesives has been indicated in many studies. Clinical reports of allergic responses of resin-based restorative materials showed that HEMA is the most common sensitizer to induce hypersensitivity in dental

personal. In vitro, HEMA has shown to be a potent inducer of apoptotic cell death [44]. Cell mutation has been observed after exposure to TEGDMA or HEMA [45,46] as well as increased intracellular concentrations of reactive oxygen species (ROS) ([47,48]. Exposure to low concentrations of the monomer for a prolonged time reduced the rate of cell proliferation possibly as result of DNA damage [49]. Reichl et al. showed that that unpolymerized HEMA and TEGDMA remained physically and chemically unchanged and could leach up to 30d from adhesives [19]. This means that these monomers can not only cause effects as potent allergic methacrylates during the first days but also for a longer time. Apart from the initial elution of residual monomers, wear and chemical processes like erosion, enzymatical hydrolysis and alcoholysis cause a constant disintegration and dissolution of the resin-based system [19]. In vitro studies showed cytotoxic and mutagenic effects of HEMA containing systems, but Reichl and et al showed that the eluted concentrations from resin composites are far below the cytotoxic concentrations described [19,50].

During aging in the oral environment, HEMA will enhance water sorption resulting in increased deterioration of the bond and shorter durability of the restoration. G-Bond showed shear bond strength values even after 16 weeks comparable to initial values [51]. To exclude HEMA may have several advantages like improved mechanical strength and lower hydrolytic degeneration. Takahashi et al. showed recently in vitro that HEMA containing SEA significantly decreased in microtensile strength after 180 and 360 days of water storage [26]. The HEMA-free adhesive G-Bond did not change water sorption and ultimate tensile strength even after 360-days water

storage [26]. An elution of hydrolytically degraded poly-HEMA of the HEMA-containing adhesives was speculated to cause the bond strength reduction over time in combination with less hydrolytic degradation of G-Bond. Good bond strength after 24 h and after 1 year water storage has also been reported by others for HEMA-free adhesives [52,53]. Lower microtensile bond strength values for G Bond have been reported recently [54,55]. The Leuven group showed that the adhesive solution of several 1-step SEA's including G-Bond were unstable after exposure to air and exhibited a phase separation reaction [56]. With time, the opaque droplets observed disappeared and the clinical implication was unclear. They compared the microtensile bond strength of nine 1-step SEA, including G-Bond, with the 3-step etch-and-rinse Optibond FL. G-Bond showed similar mean micro tensile bond strength values to dentin and enamel (24.5 and 23.9 MPa, respectively). Slightly but not significantly lower compared to the 38.1 and 31.6 Mpa for the 3-step etch-and-rinse control adhesive Optibond FL, but significantly higher as two other HEMA-containing SEA's (16.6/11.1 and 18.2/14.5 Mpa, respectively). For G-Bond a 0.8–1 µm adhesive layer was observed. It was interesting that only very high amounts of HEMA replacing the majority of acetone were able to prevent phase separation, while these higher concentrations resulted on the other hand in the lowest bond strength results [57]. The Leuven group investigated recently in another study 11 contemporary adhesives including G Bond and XP Bond [55]. Rather different results compared to the earlier reported bond strength values were observed. After 24-h storage in water mean microtensile bond strength to dentin were reported of 20.4 Mpa for G-Bond and a 52.2 Mpa for XP Bond. Optibond FL showed the highest values. They concluded that 1-step SEA underperformed compared to conventional 3-step adhesives. However, the clinical results of the 1-step SEA referred above and found in the present study contradict the laboratory findings. The differences found in the literature concerning bond strength values of the 1-step SEA reported are difficult to explain. Scherrer et al. showed after pooling of 147 studies, a high scatter in the bond strength data regardless of which adhesive or bond test was used [58]. The ranking of adhesives seemed to be dependent on the bond test used, if laboratory results can be predictive of clinical outcome remains dubious and even if weak relationships have been reported the correlation between *in vitro* and *in vivo* data are inconclusive [25,59].

Fukuoka et al. observed that G Bond interacted only superficially with dentin to a depth of approximately 300–400 nm, by which the surface remained rich in hydroxyapatite [60]. Sarr et al. [55] speculated that since the mild and ultra-mild SEA do not remove all hydroxyapatite, much calcium is available for an additional chemical interaction with functional monomers, suggesting a two-fold bonding mechanism. These bonds may be better withstand hydrolytic breakdown and explain a better durability of the adhesives. The long term stability of these bonds is not known. Observing the good long term clinical results it seems very well possible that these additional bonds complete the micromechanical retention of the SEA. Removal of the solvent has been suggested to be crucial for the adhesive [24]. To omit HEMA from the adhesive, a higher concentration of solvent is necessary. G Bond contains 40% solvent [53] and to obtain complete water/solvent evaporation strong air blow

blowing was necessary but remaining solvent may hamper conversion and enhance plasticizing of the resin [61].

Air drying may therefore have a significant effect on the removal of the solvents included in 1-step SEA's. When HEMA-free SEA's are not air-dried strong enough and long enough to remove droplets, which result by the phase separation between water and the other adhesive ingredients, this will result in lower polymerization and mechanical properties [10,61].

The cfm adhesive is a new 3-step etch-and-rinse adhesive free of HEMA and TEGDMA, as shown by Reichl et al. [19]. Studies have shown that surfactant dimethacrylates, amphiphilic organic compounds may act as solubility enhancers, facilitate penetration of hydrophobic component in the wet demineralized dentin, reduce phase separation and increase dentin bond strength [62]. The HEMA substitution for Bis EMA which represents high molecular weight may result in reduced toxicity. Zanchi et al. showed that the type of surface active agent was a significant factor for bond strength. Of five experimental HEMA free adhesive systems, the two Bis EMA containing adhesives showed the lowest microtensile bond strength values. The adhesive showed good microtensile bond strength to enamel but significantly lower to dentin compared to the golden standard 3-step etch-and-rinse Optibond FL (enamel: 30.7 and 26.7 Mpa; dentin 25.7 Mpa and 45.6 Mpa respectively) (Mine et al., 2008). Bonded to dentin, cmf showed a 1.5–3 µm thick, completely demineralized and acid-resistant hybrid layer. Incomplete resin infiltration was observed as a pattern of silver deposition resembling nanoleakage but this was not different in form or extent to that observed for Optibond FL. Omitting smaller hydrophilic monomers in the cfm adhesive resulted in a more hydrophobic resin layer, which was less prone to water absorption and hydrolytic degeneration. A clinical disadvantage of the adhesive was the long application times used to compensate for the absence of the small diluting monomers. The bond strength to dentin was enough to withstand early debonding stress as well as the following intra oral stress as shown by the good retention rate found after 5 years.

XP Bond was the first etch-and-rinse adhesive using *t*-butanol as solvent and is relatively densely filled with silica. The manufacturer suggested that the solvent with equal vaporization as ethanol, should improve polymerization of the adhesive and to be less technique sensitive due to an improved diffusion through partially collapsed demineralized dentin [55,63]. A competitive initial marginal adaptation to enamel and dentin was observed compared to other solvents containing adhesives [63]. Saboia et al. showed complete infiltration of the demineralized dentin by the resin monomers and revealed additional chemical bonding as formation of calcium phosphate complexes derived from the phosphate esters and the mineral apatite in dentin [33]. Under dry conditions, the adhesive exhibited higher bond strength than did two other etch-and-rinse systems [64]. Of 11 contemporary adhesives, XP Bond scored in a study by the Leuven group the second highest microtensile bond strength values to dentin, directly after Optibond FL [64]. A 3–5 µm completely demineralized, hydroxyapatite-free hybrid layer has been reported [55,65,66]. In addition a chemical interaction was suggested by Latta et al. between the mineral apatite in the dentin and phosphate esters in the adhesive [66]. They reported that the adhesive

showed shear bond strength values to enamel and dentin similar to two other etch-and-rinse adhesives and significantly better than the classic Syntac adhesive. Kimmes et al. showed good enamel shear bond strength for XP Bond but in contrast the lowest dentin shear bond strength of 11 adhesive systems (8 SEA and 3 etch-and-rinse) [67]. Saboia et al. [33] reported only a slight decrease in bond strength after storage in artificial saliva, but the bond strength decreased significantly after 6 months, as also shown for other etch-and-rinse adhesives [11]. Because the artificial saliva did not contain bacteria, they concluded that the reduced bond strength should be ascribed to water sorption and the following decrease in mechanical properties of the hydrolytic adhesive resins and hybrid layer degradation. Another mechanism suggested is the degradation of exposed collagen by dentin endogenous collagenolytic factors that are activated during the etching procedure leaving the collagen fibrils unprotected. The initial good in vitro dentin bond strength values and the decrease in bond strength to dentin after aging reported confirm the clinical retention picture observed. The adhesive showed despite good initial bond strength and clinical retention non-acceptable high failure rates starting already after the second clinical year.

One step SEA tended to have lower hybrid layer (HL) values than the etch-and-rinse systems. Supkien et al. observed a very rudimentary hybridized zone with G-Bond which was explained by the fact that the SEA incorporated the smear layer into the hybrid layer [68]. It has been suggested that shallow hybrid layers formed by adhesives with functional monomers capable of interacting chemically with hydroxyapatite can protect collagen and show therefore higher durability in the long run which is in line with the clinical findings for G-Bond [69,70]. On the other hand, etch-and-rinse adhesive hybrid layers which do not have a high portion of not by minerals covered unprotected collagen, may show a hydrolytic breakdown accelerated by acids and enzymes produced by bacteria and/or enzymes found in the dentin [71]. The by hydroxyapatite crystals covered collagen may have the possibility to react chemically with the functional monomer of the mild or ultra-mild SEA. In the present study a slightly roughening of the lesion surface was performed by a diamond burr, creating a smear layer as is the case in prepared cavities.

No clinical significant differences were seen between the different sizes of the lesions which confirmed earlier reported findings [72]. The relative frequency loss of retention in deep lesions was twice as less as for the other depth lesions. In earlier studies we found no such differences, however, the frequency of deep lesions was low in both studies [72]. The highest frequency lost restorations was found in the shallow lesions as also shown earlier for a 2-step SEA [72]. In earlier Class V studies of dentin bonding durability, no difference in lost restorations was found in sclerotic compared to non-sclerotic lesions [31,37], while in a recent study no difference was found for an etch-and-rinse adhesive and a significant higher loss rate in sclerotic lesions for a 2-step SEA [72]. In the present study we found the opposite, significant less lost restorations were observed in the lesions with the highest degree of sclerosis. Van Landuyt et al. observed in enamel-dentin bonded restorations that irrespective of the adhesive, 75% of the lost restorations were bonded to lesions exhibiting severe sclerosis [29].

It can be concluded that the durability in non-carious cervical lesions of the HEMA-free adhesives was successful after 5 years. Despite concerns which have been raised, the 1-step SEA showed one of the best clinical dentin bonding effectiveness.

Acknowledgements

The support from the Swedish Dental Association and the County Council of Västerbotten is gratefully acknowledged.

REFERENCES

- [1] Malacarna J, Carvalho RM, de Goes MF, Svizero N, Pashley DH, Tay FR. Water sorption/solubility of dental adhesive resins. *Dent Mater* 2006;22:973–80.
- [2] Ito S, Hashimoto M, Wadgaonker B, Svizero N, Carvalho RM, Yiu C, et al. Effects of resin hydrophilicity on water sorption and changes in modulus of elasticity. *Biomaterials* 2005;26:6449–59.
- [3] Tay FR, Pashley DH, Yoshiyama M. Two modes of nanoleakage expression in single-step adhesives. *J Dent Res* 2002;81:472–6.
- [4] Nakabayashi N, Watanabe A, Gendusa NJ. Dentin adhesion of modified 4-META/MMA-TBB resin: function of HEMA. *Dent Mater* 1992;8:259–64.
- [5] Nakaoki Y, Nikaido T, Pereira PN, Inokoshi S, Tagami J. Dimensional changes of demineralized dentin treated with HEMA primers. *Dent Mater* 2000;16:441–6.
- [6] 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;28:3757–85.
- [7] Van Landuyt KL, De Munck J, Snauwaert J, Coutinho E, Poitevin A, Yoshida Y, et al. Monomer-solvent phase separation in one-step self-etch adhesives. *J Dent Res* 2005;84:183–8.
- [8] Moszner N, Salz U, Zimmermann J. Chemical aspects of self-etching enamel-dentin adhesives: a systematic review. *Dent Mater* 2005;21:895–910.
- [9] Salz U, Zimmermann J, Zeuner F, Moszner N. Hydrolytic stability of self-etching adhesive systems. *J Adhes Dent* 2005;7:107–16.
- [10] Ikeda T, De Munck J, Shirai K, Hikita K, Inoue S, Sano H, et al. Effect of air-drying on the strength of HEMA-rich versus HEMA-free one-step adhesives. *Dent Mater* 2008;24:1316–23.
- [11] De Munck J, Ermis RB, Koshiro K, Inoue S, Van Landuyt K, Lambrechts P, et al. NaOCl degradation of a HEMA-free all-in-one adhesive bonded to enamel and dentin following two air-blowing techniques. *J Dent* 2007;35:74–83.
- [12] Kanerva L, Estlander T, Jolanki R. Occupational skin allergy in the dental profession. *Dermatol Clin* 1994;12:517–32.
- [13] Kanerva L, Jolanki R, Leino T, Estlander T. Occupational allergic contact dermatitis from 2-hydroxyethyl methacrylate and ethylene glycol dimethacrylate in a modified acrylic structural adhesive. *Contact Dermatitis* 1995;33:84–9.
- [14] Wallenhammer LM, Örtengren U, Andreasson H, Barregegård L, Björkner B, Karlsson S, et al. Contact allergy and hand eczema in Swedish dentists. *Contact Dermatitis* 2000;43:192–9.
- [15] Lönnroth EC, Wellendorf H, Ruyter E. Permeability of different types of medical protective gloves to acrylic monomers. *Eur J Oral Sci* 2003;111:440–6.

- [16] Goon AT, Isaksson M, Zimmerman E, Goh CL, Bruze M. Contact allergy to (meth)acrylates in the dental series in southern Sweden: simultaneous positive patch test reaction patterns and possible screening allergens. *Contact Dermatitis* 2006;55:219–26.
- [17] Geurtsen W, Leyhausen G. Chemical–biological interactions of the resin monomer triethylglycol-dimethacrylate (TEGDMA). *J Dent Res* 2001;80:2046–50.
- [18] Volk J, Leyhausen G, Dogon S, Geurtsen W. Additive effects of TEGDMA and hydrogen peroxide on the cellular glutathione content of human gingival fibroblasts. *Dent Mater* 2007;23:921–6.
- [19] Reichl F-X, Löhle J, Seiss M, Furche S, Shehata MM, Hickel R, et al. Elution of TEGDMA and HEMA from polymerized resin-based bonding systems. *Dent Mater* 2012;28:1120–5.
- [20] Poitevin A, De Munck J, Cardoso MV, Mine A, Peumans M, Lambrechts P, et al. Dynamic versus static bond-strength testing of adhesive interfaces. *Dent Mater* 2008;26:1068–76.
- [21] Sidhu SK, Omata Y, Tanaka T, Koshiro K, Spreafico D, Semeraro S, et al. Bonding characteristics of newly developed all-in-one adhesives. *J Biomed Mater Res B Appl Biomater* 2007;80:297–303.
- [22] Burrow MF, Kitasako Y, Thomas CD, Tagami J. Comparison of enamel and dentin microshear bond strengths of a two-step self-etching priming system with five all-in-one systems. *Oper Dent* 2008;33:456–60.
- [23] Ferracane J. Resin-based composite performance: are there some things we can't predict. *Dent Mater* 2013;29:51–8.
- [24] Söderholm K-JM, Soares M, Argumosa M, Loveland C, Bimstein E, Guelmann M. Shear bond strength of one etch-and-rinse and five self-etching dental adhesives when used by six operators. *Acta Odontol Scand* 2008;66:243–9.
- [25] Van Meerbeek B, Peumans M, Poitevin A, Mine A, Van Ende A, Neves A, et al. Relationship between bond-strength tests and clinical outcomes. *Dent Mater* 2010;26:e100–21.
- [26] Takahashi M, Nakajima M, Hosaka K, Ikeda M, Foxton RM, Tagami J. Long-term evaluation of water sorption and ultimate tensile strength of HEMA-containing/-free one-step self-etch adhesives. *J Dent* 2011;39:506–12.
- [27] Burrow MF, Tyas MJ. Clinical trial of G-Bond all-in-one adhesive and Gradia Direct resin in non-carious cervical lesions—results at 1 year. *J Dent* 2007;35:623–5.
- [28] Kubo S, Yokota H, Yokota H, Hayashi Y. Two-year clinical evaluation of one-step self-etch systems in non-carious cervical lesions. *J Dent* 2009;37:149–55.
- [29] Van Landuyt KL, Peumans M, De Munck J, Cardoso MV, Ermis B, Van Meerbeek B. Three-year clinical performance of a HEMA-free one-step self-etch adhesive in non-carious cervical lesions. *Eur J Oral Sci* 2011;119:511–6.
- [30] Burrow MF, Tyas MJ. Comparison of two all-in-one adhesives bonded to non-carious cervical lesions—results at 3 years. *Clin Oral Investig* 2012;16:1089–94.
- [31] van Dijken JWV. Durability of three simplified systems in Class V non-carious cervical dentin lesions. *Am J Dent* 2004;17:27–32.
- [32] van Dijken JWV. A clinical evaluation of anterior conventional, microfiller and hybrid composite resin fillings. A six year follow up study. *Acta Odont Scand* 1986;44:357–67.
- [33] Saboia VPA, Silva FCFA, Nato F, Mazzoni A, Cadenaro M, Mazzotti G, et al. Analysis of differential artificial ageing of the adhesive interface produced by a two-step etch-and-rinse adhesive. *Eur J Oral Sci* 2009;117:618–24.
- [34] ADA Council on acceptance program guidelines: dentin and enamel adhesive materials. *J Am Dent Assoc* 2001;142:12.
- [35] Peumans M, De Munck J, Van Landuyt KL, Poitevin A, Lambrechts P, Van Meerbeek B. Eight-year clinical evaluation of a two-step self-etch adhesive with and without selective enamel etching. *Dent Mater* 2010;26:1176–84.
- [36] van Dijken JWV, Sunnegårdh-Grönberg K, Lindberg A. Clinical long term retention of etch-and-rinse and self-etch adhesive systems in non-carious cervical lesions. A 13 years evaluation. *Dent Mater* 2007;23:1101–7.
- [37] van Dijken JWV, Pallesen U. Long term dentin retention of etch-and-rinse and self-etch adhesives and a resin modified glass ionomer cement in non-carious cervical lesions. *Dent Mater* 2008;24:915–22.
- [38] van Dijken JWV. Retention of a resin-modified glass ionomer adhesive in non-carious cervical lesions. A 6-year follow-up. *J Dent* 2005;33:541–7.
- [39] Zhou Z, Yu S, Jiang Y, Lin Y, Ni L. A randomized controlled clinical trial of one-step self-etching adhesive systems in non-carious cervical lesions. *Am J Dent* 2009;22:235–40.
- [40] De Munck J, Van Meerbeek B, Yoshida Y, Inoue S, Vargas M, Suzuki K, et al. Four-year water degradation of total-etch adhesives bonded to dentine. *J Dent* 2003;82:136–40.
- [41] Blunck U, Knitter K, Jahn K-R. Six-months clinical evaluation of XP Bond in non-carious cervical lesions. *J Adhes Dent* 2007;9:265–8.
- [42] van Dijken JWV, Pallesen U. A 7-year randomized prospective study of a one-step self-etching adhesive in non-carious cervical lesions. The effect of curing modes and restorative material. *J Dent* 2012;40:1060–7.
- [43] van Dijken JWV, Sunnegårdh-Grönberg K. Fiber-reinforced packable resin composites in class II cavities. *J Dent* 2006;34:763–9.
- [44] Paranjpe A, Bordador LCF, Wang M-Y, Hume WR, Jewett A. Resin monomer 2-hydroxyethyl methacrylate (HEMA) is a potent inducer of apoptotic cell death in human and mouse cells. *J Dent Res* 2005;84:172–7.
- [45] Schweikl H, Schmalz G, Rackebrandt K. The mutagenic activity of unpolymerized resin monomers in *Salmonella typhimurium* and V79 cells. *Mutat Res* 1998;415:119–30.
- [46] Schweikl H, Schmalz G, Spruss T. The induction of micronuclei in vitro by unpolymerized resin monomers. *J Dent Res* 2001;80:1615–20.
- [47] Schweikl H, Spagnuolo G, Schmalz G. Genetic and cellular toxicology of dental resin monomers. *J Dent Res* 2006;85:870–7.
- [48] Stanislawski L, Lefeuvre M, Bourd K, Soheili-Majd E, Goldberg M, Perianin A. TEGDMA-induced toxicity in human fibroblasts is associated with early and drastic glutathione depletion with subsequent production of oxygen reactive species. *J Biomed Mater Res A* 2003;66:476–82.
- [49] Samuelsen JT, Hole JA, Becher R, Karlsson S, Morisbak E, Dahl JE. HEMA reduces cell proliferation and induces apoptosis in vitro. *Dent Mater* 2008;24:134–40.
- [50] Geurtsen W, Lehmann F, Spahl W, Leyhausen G. Cytotoxicity of 35 dental resin composite monomers/additives in permanent 3T3 and three human primary fibroblast cultures. *J Biomed Mater Res* 1988;41:474–80.
- [51] Salz U, Bock T. Adhesion performance of new hydrolytically stable one-component self-etching enamel/dentin adhesives. *J Adhes Dent* 2010;12:7–10.
- [52] Mine A, De Munck J, Van Landuyt KL, Poitevin A, Kuboki T, Yoshida Y, et al. Bonding effectiveness and interfacial characterization of a HEMA/TEGDMA-free three-step etch&rinse adhesive. *J Dent* 2008;36:767–73.
- [53] Torkabadi S, Nakajima M, Ikeda M, Foxton RM, Tagami J. Bonding durability of HEMA-free and HEMA-containing one-step adhesives to dentine surrounded by bonded enamel. *J Dent* 2008;36:80–6.
- [54] Hass V, Folkuenig MS, Reiss A, Loguercio AD. Influence of adhesive properties on resin-dentin bond strength of one-step self etching adhesives. *J Adhes Dent* 2011;13:417–24.

- [55] Sarr M, Kane AW, Vreven J, Mine A, Van Landuyt KL, Peumans M, et al. Microtensile bond strength and interfacial characterization of 11 contemporary adhesives bonded to bur-cut dentin. *Oper Dent* 2010;35:94–104.
- [56] Van Landuyt K, Mine A, De Munck J, Jaecques S, Peumans M, Lambrechts P, et al. Are one-step adhesives easier to use and better performing? Multifactorial assessment of contemporary one-step self-etching adhesives. *J Adhes Dent* 2009;11:175–90.
- [57] Van Landuyt KL, Snauwaert J, Peumans M, De Munck J, Lambrechts P, Van Meerbeek B. The role of HEMA in one-step self-etch adhesives. *Dent Mater* 2008;24:1412–9.
- [58] Scherrer SS, Cesar PF, Swain MV. Direct comparison of the bond strength results of the different test methods: a critical literature review. *Dent Mater* 2010;26:e78–93.
- [59] Heintze SE, Thunpithayakul C, Armstrong SR, Rousson V. Correlation between microtensile bond strength data and clinical outcome of Class V restorations. *Dent Mater* 2011;27:114–25.
- [60] Fukuoka A, Koshiro K, Inoue S, Yoshida Y, Tanaka T, Ikeda T, et al. Hydrolytic stability of one-step self-etching adhesives bonded to dentin. *J Adhes Dent* 2011;13:243–8.
- [61] Hiraishi N, Breschi L, Prati C, Ferrari M, Tagami J, King NM. Technique sensitivity associated with air-drying of HEMA-free, single-bottle, one-step self-etch adhesives. *Dent Mater* 2007;23:498–505.
- [62] Zanchi CH, Münchow EA, Ogliaari FA, de Carvalho RV, Chersoni S, Prati C, et al. A new approach in self-etching adhesive formulations: replacing HEMA for surfactant dimethacrylate monomers. *J Biomed Mater Res Part B* 2011;99B:51–7.
- [63] Manhart J, Trumm C. Marginal adaptation of an etch-and-rinse adhesive with a new type of solvent in class II cavities after artificial aging. *Clin Oral Investig* 2010;14:699–705.
- [64] Hegde M, Manjunath J. Bond strength of newer dentin bonding agents in different clinical situations. *Oper Dent* 2011;36:169–76.
- [65] Margvelashvili M, Goracci C, Beloica M, Papacchini F, Ferrari M. In vitro evaluation of bonding effectiveness to dentin of all-in-one adhesives. *J Dent* 2010;38:106–12.
- [66] Latta MA. Shear bond strength and physicochemical interactions of XP Bond. *J Adhes Dent* 2007;9:245–8.
- [67] Kimmes NS, Barkmeier WW, Erickson RL, Latta MA. Adhesive bond strengths to enamel and dentin using recommended and extended treatment times. *Oper Dent* 2010;35:112–9.
- [68] Skupien JA, Susin AH, Angst PDM, Anesi R, Machado P, Bortolotto T, et al. Micromorphological effects and the thickness of the hybrid layer – a comparison of current adhesive systems. *J Adhes Dent* 2010;12:435–42.
- [69] Breschi L, Mazzonni A, Ruggeri A, Cadenaro M, Di Lenarda R, De Stefano Dorigo E. Dentin adhesion review: aging and stability of the bonded surface. *Dent Mater* 2008;24:90–101.
- [70] Inoue S, Koshiro K, Yoshida Y, De Munck J, Nagakana K, Suzuki K, et al. Hydrolytic stability of self-etch adhesives bonded to dentin. *J Dent Res* 2005;84:1160–4.
- [71] Hanabusa M, Mine A, Kuboki T, Momoi Y, Van Ende A, Van Meerbeek B, et al. Bonding effectiveness of a new multi-mode adhesive to enamel and dentin. *J Dent* 2012;40:475–84.
- [72] van Dijken JWV. A prospective 8-year evaluation of a mild two-step self-etching adhesive and a heavily filled two-step etch-and-rinse system in non-carious cervical lesions. *Dent Mater* 2010;26:940–6.