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Alternatives to amalgam: Is pretreatment necessary for effective bonding to dentin?

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ABSTRACT

Objective: The aim of this study was to determine whether pretreatment of the dentin surface is beneficial or not by analysis of the bond strengths of four self-adhesive restoratives and four restoration materials where pretreatment of dentin was necessary.

Methods: Bovine incisors (n = 160) were ground flat on the labial surfaces to expose dentin using a grinder and silicon carbide (SiC) abrasive papers under running water. Between preparation and bonding procedures, the crowns were stored in Chloramine-T solution at 4 °C. Eight different restorative materials were studied: Activa BioActive (**ABA**), Cention Forte (**CNF**), Ceram.x Spectra ST (**CXS**), Riva self-cure (**RSC**), Equia Forte (**EQF**), Fuji II LC (**FJI**), Ketac Molar (**KTM**), Surefil one (**SFO**). Four materials required pretreatment of the dental hard tissue before placement, whereas the other four were self-adhesive (no pretreatment). The specimens were mounted vertically in plaster. A preload of 5 N was applied and the subsequent cross-head speed was 0.8 mm/min. Shear bond strengths (MPa) were calculated as the failure load divided by the bonding area. Failure modes were recorded as adhesive, cohesive or pretest. Data were statistically analyzed via ordinal regression for inference and Tukey's method to adjust for multiple comparisons. All computations were done using R version 4.1.2 (R Core Team 2021).

Results: S_{max} (failure stress in MPa) of the combined groups with pretreatment were significantly higher than the self-adhesive materials. The highest frequency of pretest-failure was seen with **FJI**. Glass-ionomer cements without pretreatment were the only restoratives with pretest failures. Amongst materials without pretreatment, **SFO** had the highest bond strengths.

Significance: The further reduction of the placement steps for materials used as an amalgam alternative, namely the omission of pretreatment of the dentin, results in these self-adhesive materials having lower bond strengths than materials that require pretreatment of the dentin.

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1.   Introduction

The search for reliable amalgam alternatives is continuing as the European Union changed its regulation to impose a stepwise reduction of amalgam use for children under 15 years and pregnant women. Strict regulation now exists regarding the disposal of waste amalgam (2017/852 article 10).

Although modern restorative materials, particularly resin composites, are reasonable alternatives to amalgam, it is difficult for them to compete with several beneficial characterics of amalgam such as easy handling and extended durability. The clinical and mechanical properties of composite restoratives are often compromised by complicated handling procedures and unfavorable cavity conditions. The benefits of resin composite such as excellent esthetics, conservation of tooth structure, longevity, and reparability stand in contrast to their technique sensitivity, placement time and affordability. Especially for the group of patients affected by the above-mentioned regulation, children and pregnant women, placement time should be shortened to a minimum. This is sometimes impossible with the complicated handling procedures of composites.

Another group of dental restoratives, glass-ionomer cements (GIC) are often used instead of amalgam because they are easier to handle. But GIC fails to be considered as a proper alternative to amalgam along with resin composites due to its poor physical and chemical properties.

New materials with simplified handling, and shorter processing times compared to composites, are being developed by several major manufacturers. In some cases, they are rapidly introduced to the market. There is often insufficient time for prior long-term clinical studies. It is therefore important to conduct standardized laboratory investigations. These can be used to compare such new materials with conventional composite materials.

One such material is Surefil one, a self-adhesive hybrid composite, which is claimed to combine resin composite strengths with the easy handling of GIC-based materials. It is light cured, but depth-of-cure is not an issue because it is dual-activated. Cross-linking of structural monomers, combined with self-adhesive properties of GIC polyacids, is proposed to obviate the need for etching, bonding or layering.

Cention Forte is a self-curing radiopaque material for direct treatment of anterior and posterior cavities. Cention Forte has been termed an Alkasite and is claimed [1] to be bioactive and suitable for cavities where amalgam is contraindicated or not desired.

The aim of the study was to evaluate whether or not pretreatment of the dentin substrate is desirable. To address this question, bond strengths were measured of eight materials that are intended as amalgam alternatives. Four of these require pretreatment and four are specified as self-adhesive. Our null hypothesis is therefore that there is no difference in performance between the pretreatment and self-adhesive categories.

2.   Materials and methods

2.1.   Sample size

160 bovine incisors were used as substrates to measure the bond strengths of eight dental restoratives. A handsaw was used to separate the crowns from the roots. Buccal surfaces of the crowns were ground using a universal grinder (Metaserv 2000, Buehler, Düsseldorf, Germany) and silicon carbide (SiC) abrasive papers (grit 80, 400 and 1000, Buehler, Düsseldorf Germany) under running water. Grit 80 SiC paper was used to obtain a flat dentin surface, followed by grit 400 for 1 min. Between preparation and bonding procedures, the crowns were stored in Chloramine-T solution at 4 °C. Immediately before bonding, grit 1000 SiC paper was applied for 10 s.

Eight different restorative materials were each randomly placed on 20 dentin substrates. Four materials required pretreatment of the dental hard tissue before placement, whereas the other four were self-adhesive (no pretreatment) (Table 1).

2.2.   Pretreatment and application

For the two resin-based composite restoratives (**CXS** & **ABA**) a universal self-etching adhesive material (Prime&Bond active (**PBA**), Dentsply, Konstanz, Germany) was applied with a microbrush and agitated lightly on the surface for 20 s. The adhesive was dispersed with water- and oil-free compressed air until a shiny and immobile film had formed and was light-cured for 10 s with a light-curing unit (SmartLite Pro (SLP), Dentsply, Konstanz, Germany; Irradiance: 1250 mW/cm²).

For the self-curing Alkasite (**CNF**), a two-component self-etching and self-curing primer (Cention Primer, Ivoclar Vivadent, Schaan, Liechtenstein) was applied to the dentin surface with a single-use applicator. After coating and scrubbing for 10 s, the primer was dispersed with water- and oil-free compressed air until a thin and shiny film had formed.

For the glass ionomer (**RSC**), the dentin surface was etched with Riva Conditioner (SDI, Victoria, Australia) – a polyacrylic acid conditioner and left on the surface for 10 s. The etching agent was rinsed, followed by gentle air-drying of the dentin surface.

After completion of the pretreatment steps, cylindrical specimens of the restorative pastes were produced in self-dissolving gelatin capsules and bonded perpendicularly to the dentin surface. Any excess was removed and the materials were cured according to the manufacturers' recommendations. For the light-activated materials, the light curing unit was used from 3 different directions for 10 s each (see Table 2).

For self-adhesive materials the dentin area was subject to gentle air-drying. Cylindrical specimens of the restorative in self-dissolving gelatin capsules were bonded perpendicularly

Table 1 – Materials.

Code	Material	Type	Pretreatment Y/N	Curing mode	Manufacturer	Formulation	Lot numbers
ABA	Activa BioActive	Resin-based "bioactive" composite	Yes	Light	Pulpdent, Watertown, MA, USA	Powder: silanated bioactive glass and calcium, silanated silica, sodium fluoride; Liquid: diurethane modified by the insertion of a hydrogenated polybutadiene and other methacrylate monomers, modified polyacrylic acid	210305
CNF	Cention Forte	Self-curing "bioactive" Alkasite	Yes	Dual	Ivoclar Vivadent, Liechtenstein	Powder: barium aluminum silicate glass, ytterbium trifluoride, isofiller, calcium barium aluminum fluorosilicate glass, calcium fluoro silicate glass; Liquid: urethane dimethacrylate, tricyclodecandimethanol dimethacrylate, tetramethyl-xylylene diurethane dimethacrylate, polyethylene glycol 400 dimethacrylate, ivocerin, hydroxyperoxide	Z02CMP
CXS	Ceram.x Spectra ST	Resin-based composite (RBC)	Yes	Light	Dentsply, Konstanz, Germany	Ethoxylated Bisphenol A Dimethacrylate, Urethane modified Bis-GMA dimethacrylate resin, 2,2′-ethylenedioxydiethyl dimethacrylate, ytterbium trifluoride, 2,6-di-tert-butyl-p-cresol	2103000526
RSC	Riva self-cure	Bulk fill glass-ionomer	Yes	Self	SDI, Victoria, Australia	Powder: fluoroaluminosilicate glass, polyacrylic acid, iron oxide; Liquid: polybasic carboxylic acid, water	A2104058EA
EQF	Equia Forte HT	Glass-ionomer	No	Self	GC, Tokyo, Japan	2-hydroxyethyl methacrylate, polybasic carboxylic acid, urethane dimethacrylate, dimethacrylate, calcium-aluminum-fluoro-silicate glass, others	210202A
FJI	Fuji II LC	Resin-modified Glass-ionomer	No	Light	GC, Tokyo, Japan	Powder: Ca, La, Al-fluorosilicate glass, pigments; Liquid: Polycarbonic acid, tartaric acid, water	210524B
KTM	Ketac Molar	Glass-ionomer	No	Self	3 M, Neuss, Germany	Powder: Radiopaque fluroaluminosilicate glass, polyacrylic acid, pigments; Liquid: Polyacrylic acid, tartaric acid, and water	8048452
SFO	Surefil one	Self-adhesive composite hybrid	No	Light	Dentsply, Konstanz, Germany	Powder: silanated aluminum-phosphor-strontiumsodium-fluoro-silicate glass, dispersed silicon dioxide, ytterbium fluoride, pigments; Liquid: acrylic acid, polycarboxylic acid, bifunctional acrylate, self-cure initiator, camphorquinone, stabilizer	2104000853

Table 2 – Study variables. Modified after and [2]		
Tooth substrate	Type and age of teeth	Bovine incisors of the lower jaw; average age 60 months
	Selected region	Superficial layers of facial incisal dentin
	Condition	Only sound dentin
	Collection and storage	Stored at − 20 °C; thawed 24 h before experiments; stored at 4 °C in 0.5% chloramine-T solution
Restorative material		Ceram.x Spectra ST, Activa BioActive, Cention Forte, Riva self-cure, Surefil one, Ketac Molar, Fuji II LC, Equia Forte HT
Specimen preparation and pre-testing conditions	Method	Grinder (Metaserv 2000, Buehler, Düsseldorf, Germany) and SiC abrasive paper (grit 80, 400 and 1000, Buehler, Düsseldorf, Germany). Grit 80 used to create appropriate bonding dentin surface; grit 400 for 1 min; grit 1000 for 10 s right before bonding procedures.
	Restorative shape and dimensions	Cylindrical (internal diameter 4 mm, height 8 mm)
	Restorative application and curing conditions	Restorative material was inserted into self-dissolving gelatin capsules, pressed against the dentin surface at 90°. If material is light-activated, light-curing from 3 different directions for 10 s each (total 30 s).
	Bonding and testing environment	20 °C, 60% relative humidity
	Post-bonding storage	24 h, water, 37 °C
	Potting material	Tooth specimens were mounted vertically in hard plaster, type 3, color blue (Heraeus Kulzer, Hanau, Germany)
Data reporting and analyses	Descriptive statistics parameters	Results were expressed as means and with exact confidence intervals.

to the dentin surface. The excess was removed and the materials were cured if required, according to the manufacturers' recommendations. **SFO** and **FJI** were light-cured from 3 different directions for 10 s each.

For encapsulated materials a Rotomix device was used (3 M, Neuss, Germany). All specimens were stored in distilled water at 37 °C for 24 h.

2.3. Shear bond strength measurements

The specimens were mounted vertically in plaster. Specimen cylinders were aligned horizontally, parallel to the surface of the hard plaster. A preload of 5 N was applied and the subsequent cross-head speed was 0.8 mm/min, using a Universal testing machine (Zwick/Roell, Ulm, Germany). The shear bond strength was calculated as the load at failure divided by the bonding area and was expressed in MPa. The mode of failure was recorded as adhesive, cohesive or pretest failure. The data were statistically analyzed.

2.4. Statistical analysis

Because of concerns with normality of residuals, ordinal regression was used for inference and Tukey's method was used to adjust for multiple comparisons. All computations were done using R version 4.1.2 (R Core Team 2021).

3. Results

S_{max} (failure stress in MPa) for all material are depicted in Fig. 1. Pooled data are shown in Fig. 2. Types of failure are shown in Fig. 3. Descriptive statistics are summarized in
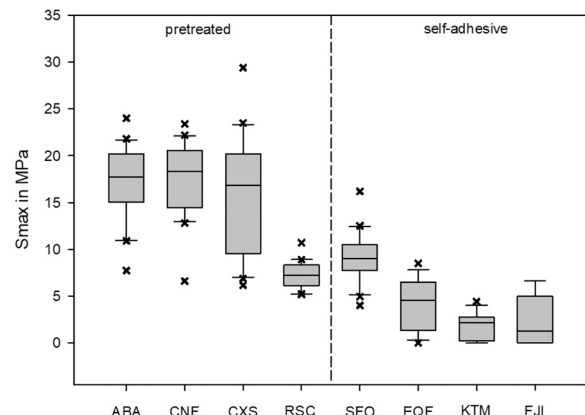

Fig. 1 – SBS Boxplot.

Table 3. Fig. 2 shows that the combined groups with pretreatment had a significantly higher S_{max} compared to the self-adhesive materials. As shown in Figs. 1 and 2, three of the four materials with pretreatment outperformed all the others. Mean S_{max} ranged from 1.7 MPa for **KTM** to 17.4 MPa for **CNF**.

As shown in Fig. 3, the highest frequency of pretest-failure was seen with **FJI**. The glass-ionomer cements without pretreatment were the only restoratives with pretest failures.

Except for **RSC**, the materials with pretreatment showed the highest S_{max} (Fig. 1). All three glass-ionomer cements showed the lowest S_{max}. The greatest range of S_{max} was seen in **CXS**. For materials without pretreatment, **SFO** showed the highest bond strengths.
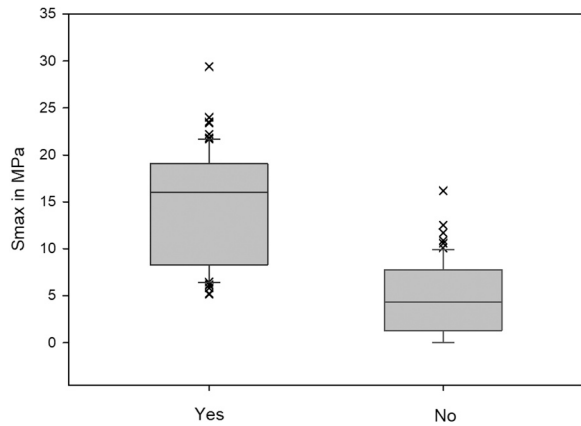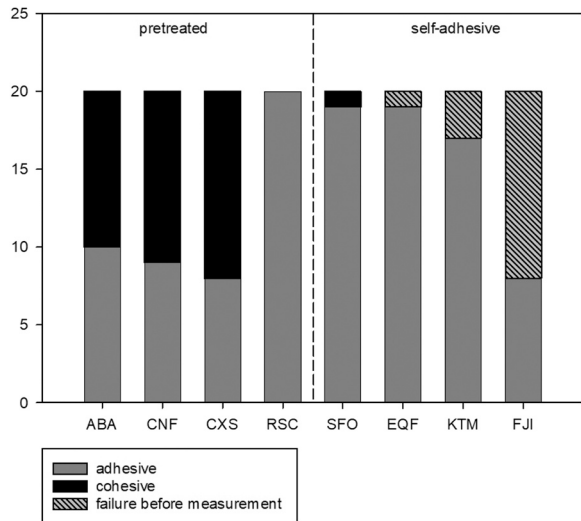
Fig. 2 – Pretreatment.

Fig. 3 – mode of failure.

Table 3 – Descriptive statistics. Missing values = failure before measurement.				
Material	Mean	SD	N	Missing
ABA	17.1	4.0	20	0
CNF	17.4	4.0	20	0
CXS	16.1	6.1	20	0
EQF	4.2	2.7	20	1
FJI	2.2	2.6	20	12
KTM	1.7	1.5	20	3
RSC	7.3	1.5	20	0
SFO	9.1	2.6	20	0
All	10.2	7.0	20	16

4. Discussion

This study compared the SBS of eight different materials to bovine dentin. Four materials required pretreatment and four were self-adhesive. In the case of **ABA** and **CXS** the pretreatment entailed the use of a bonding agent (**PBA**) upon the dentin surface. The dentin surface was treated with a primer before placing **CNF** and for the placement of **RSC** a conditioner was used.

The SBS of materials with pretreatment were significantly higher than self-adhesive materials. This is in accordance with previous studies where SBS to pretreated surfaces were compared with self-adhesive materials [3–7]. **ABA** showed significantly higher SBS than conventional glass-ionomer materials, which is in agreement with previous studies [8]. Etch and rinse with a phosphoric acid was used for **RSC**, a glass ionomer. The use of a dentin conditioner prior to glass-ionomer placement proved to be an important step in improving the bond strength [9].

ABA, CNF and **SFO** were introduced recently. Previous measurements have shown that specimens without pretreatment had lower SBS than after applying Scotchbond Universal adhesive [10]. Although the pretreatment agents in our study were **PBA** for **ABA**, Cention Primer for **CNF** and no pretreatment for **SFO**, our mean bond strengths (Table 3) for these three materials correlate with the previous study [10], but are clearly 10–30% lower. If no adhesive was applied, SBS for **SFO** was highest compared to the other two materials [10].

The manufacturer's recommendation is to wait for the **ABA** material to self-cure for 20–30 s before proceeding with low-intensity light curing. This is to reduce polymerization stress and exothermic reaction. However, in our in vitro study, both polymerization stress and exothermic reaction can be neglected. Therefore, the same irradiance was used for all materials in order to obtain comparable results. The manufacturer of **ABA** does not give a concrete indication of how much mW/cm² energy output can be considered "low intensity".

Specimens used in this study were stored in water at 37 °C for 24 h. Although the storage time of specimen after bonding procedures is considered clinically relevant [11], it has been shown that long-term storage had only a small effect on bonding [12].

Amongst the self-adhesive group of materials, **SFO** had the highest shear strengths: S_{max} of 9.1 MPa (Table 3). Previous research showed poor bond strengths with GICs including **EQF, FJI** and **KTM** [13,14]. Incorporating light-activated monomers to GIC improved their physical and mechanical properties – now known as resin-modified glass ionomer cements (RMGIC) [13–16]. Other formulations exist that either include reactive glass fillers but set by photo-polymerization (polyacid-modified resin-composites: PMRCs or compomers) or that incorporate prereacted GIC filler in a photocured resin matrix (GIOMER) [10]. Bond strengths for PMRCs are up to 12 MPa [16,17] in contrast to conventional GICs which range between 3 and 7 MPa [14,18] or, as shown in the present study, ranging between 1.7 and 4.2 MPa (Table 3). The application of conditioner for the three materials **EQF, FJI** and **KTM** is described by the manufacturer as "optional" or "recommended". Since this study deals with amalgam alternatives, rapid treatment is essential. Therefore, in this study, the quickest possible handling procedure was applied, which is also applied by most dental practitioners.

Conventional polyacids lack polymerizable groups, therefore they cannot form a polymerized network [19,20]. **SFO** has been formulated as a modified polyacid system of high molecular mass to merge the classical self-adhesive feature of glass-ionomer cement with a structural polymerized network [19–21]. Recent 1-year clinical recall results of 41 patients suggest that **SFO** gave a clinically satisfactory performance in load-bearing class I, II and non-retentive class V cavities [21].

CNF is presented with a two-component self-etching and self-curing primer. **CNF** had the highest mean shear strengths of the investigated materials (Table 3).

A previous in-vitro study [22] showed that in terms of marginal quality, Surefil one behaves similar to conventional resin composite bonded with self-etch adhesives. The wear behavior of Surefil one outperformed other amalgam alternatives, such as **ABA, Equia Forte Fil** and **FJI** [22].

A recent clinical trial [21] showed that 84% of **SFO** restorations (class I, II and V cavities in permanent teeth) were satisfactory after 1 year with a recall rate above 80% and an annual failure rate (AFR) of 2%. Results were evaluated at baseline using the USPHS criteria, developed by Cvar and Ryge in 1971. Moderate hypersensitivity and partial fracture of the restoration were recorded as failures [21]. In another clinical study postoperative sensitivity (POS) was shown for **ABA (4.2%)**, **EQF (12.5%)** and **CNF (29.2)** 24 h after placement in class I maxillary and mandibular premolars and molars, which decreased significantly after 1 month (**ABA (0%)**, **EQF (4.2%)** and **CNF (10.4%)**) [22].

Two-year clinical performances of a high viscosity GIC (Equia, GC) and a nanohybrid resin composite (GrandioSO, Voco) were compared in a randomized, split-mouth study. A total of 112 restorations were done on both mandibular second molars (class I). GICs showed a success rate of 96%, while all resin composite restorations succeeded after 2 years. Significantly higher wear was observed for Equia [23]. Restorations were evaluated using modified USPHS criteria. An in-vitro study on localized and generalized wear showed that self-cured **SFO** in its experiment stage (ASAR-MP4) showed similar results to **ABA, FJI** and **EQF** [24].

The link between appropriate in-vitro properties and long term in-vivo success is the clinician's ability to handle and manipulate the material (technique sensitivity), which has an enormous impact on the quality and on the longevity of dental restoratives [25]. Several dental materials for posterior restoration have failed, either because of their technique sensitivity, or pure mechanical properties to withstand load in large posterior cavities [25,26]. Self-adhesive restoration materials are preferred as amalgam alternatives because no technique-sensitive and time-consuming bonding procedure is required [27].

Laboratory measurements on bond strength remain a useful tool to compare systems. However, no correlations between in vitro performance and clinical success can be demonstrated and if so, they are poor at best [28]. The absence of direct correlation to clinical outcomes, however, should not invalidate in-vitro trials [29]. Clinical studies remain the ultimate tool to evaluate the performance of new materials shown in this study.

5. Conclusion

1. In this study, the bond strength of materials requiring pretreatment of dentin is superior to that of self-adhesive materials.
2. Pre-test failures were only observed with glass-ionomer cements.

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