



## Effect of different surface treatment techniques on the repair strength of indirect composites



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### ARTICLE INFO

#### Article history:

Received 14 November 2016

Received in revised form 20 January 2017

Accepted 29 January 2017

#### Keywords:

Dental materials  
Intra-oral repair  
Dental restorations  
Thermal cycling  
Dental adhesion  
Indirect composite resin  
Air-abrasion  
Etching

### ABSTRACT

**Objectives:** Composite resin restorations present high survival rates and when a failure occurs repair is often possible. The aim of this study was to assess the effect of various repair techniques on indirect restorations.

**Methods:** LAVA Ultimate (3M), and Clearfil Estenia blocks (Kuraray) were repaired with or without surface roughness treatments, silane application and artificial ageing. Micro-shear bond stress tests were performed, while cohesive strength served as positive control. ANOVA was used for cohesive strength and effect of ageing, and linear mixed models to evaluate the effect of treatment variables on repair strength. **Results:** Both materials reacted differently on surface treatments. Untreated (no treatment, no silane) repair strength was  $16.3 \pm 6.3$  MPa for LAVA Ultimate and  $19.0 \pm 4.3$  MPa for Estenia. Thermal cycling resulted in a 14–58% reduction of cohesive strength. Without cycling, all treatments resulted in a significant increase of bond strength in LAVA Ultimate ( $p < 0.003$ ). After cycling use of air-abrasion showed a positive trend for both substrates, significantly effective for LAVA Ultimate ( $p < 0.04$ ), and silane and CoJet for Estenia ( $p < 0.024$ ). The positive effect of HF treatment disappeared after cycling.

**Conclusion:** It may be concluded that (1) the effect of surface treatment procedures on the repair bond strength of indirect composites is depended on the substrate and ageing. (2) Silane did not have a clear overall positive effect on bond strength and (3) artificial ageing had a strong negative influence on the stability of the adhesive interface and on the cohesive strength of one indirect composite resin material, but not the other.

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### 1. Introduction

Improvements in quality of materials and placement techniques made the clinical application of resin-based composites feasible and predictable even in complex clinical situations such as cusp replacing restorations or rehabilitation of severely worn dentitions [1–6]. Even though the annual survival rates of composites are satisfactory, the restorations may also present failures that can be inherent to the materials, (e.g. material fracture) [7], to the operator (e.g. improper placement technique and individual criteria for failure assessment) [8–10], or to the individual risk factors of the patient (caries risk or bruxism) [11,12]. Overall, secondary caries and fracture are the predominant failure

reasons of composite restorations [12]. Repairing failed restorations may be more advantageous and less invasive than replacement [13–15]. However, when repairing a restoration, one must often obtain adhesion to different substrates such as composite resins, metals, ceramics and tooth materials at the same time. Therefore, it is important to understand the possibilities of restoration repair, where only the missing or defect part is replaced [16–18].

Numerous in vitro studies have reported on the effect of different surface treatments on repair strength of composite restorations [19–25]. Unfortunately, no conclusion could be drawn on which (universal) repair technique was the best, as the used materials and methods of all these studies varied strongly. Overall, these studies confirmed a positive role of air abrasion, but still only scarce information of repair on indirect restorations is available.

Therefore, the aim of this in vitro study was to investigate the effect of different surface roughening treatments, silane application and artificial ageing on the repair bond strength of two

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indirect composite resin-based systems. The hypotheses of this study were: (i) different surface treatment techniques will result in different repair strengths, (ii) application of a separate silane layer will enhance the repair strength and (iii) artificial ageing will decrease the stability of the repair adhesive interface.

## 2. Materials and methods

### 2.1. Production of samples

To obtain standardized samples of indirect composite resin, industrial pre-polymerized blocks of LAVA Ultimate for CAD-CAM use (color A3-LT, 3M ESPE Dental Products, St. Paul, Minnesota, USA) were cut with a diamond blade (Buehler Ltd., Lake Bluff, IL, USA) of 0.4 mm thick, resulting in 96 cubical samples of 6 × 6 mm in width and 4 mm in height. Additionally, using a custom Teflon mold, 96 similar sized cubical samples were obtained of Clearfil Estenia C&B (color DA3, Kuraray, Okayama, Japan). This composite was applied in two layers of 2 mm thick and each layer was separately photocured for 20 s with a Bluephase 16i LED polymerization unit (Ivoclar Vivadent, Liechtenstein; light intensity >1200 mW/cm<sup>2</sup>), then blocks were complementary cured with Palatray CU (wavelength 400–500 nm; Heraeus Kulzer, Hanau, Germany) for 12 min. Finally, samples were covered with glycerin-based Air Barrier paste (Kuraray) and exposed to thermal curing in a heat curing oven (Multimat Mach 3, Dentsply De Trey, Hanau-Wolfgang, Germany) at 110 °C for 15 min according to the manufacturers' instruction. All blocks of LAVA Ultimate and Clearfil Estenia were immersed in 70% ethanol for 5 min for cleaning and then air-dried. To obtain a standardized surface roughness, similar to a rough dental diamond bur, samples were grinded by hand for 10 s with a dry 150-grit silicon carbide grinding paper (Siawat, Frauenfeld, Switzerland). Finally, samples were

cleaned ultrasonically for 15 min in distilled water and stored dry at room temperature.

### 2.2. Surface treatment procedure and groups of treatment

Samples of LAVA Ultimate and Clearfil Estenia were assigned to six surface treatment protocols (n = 16): (1) no treatment, serving as negative control, (2) air-abrasion with CoJet (30 μm Al<sub>2</sub>O<sub>3</sub>-particles coated with SiO<sub>2</sub>, 3M ESPE) for 10 s at a distance of 10 mm in a circular motion with a pressure of 3 bar (MicroEtcher II, Danville Materials, San Ramon, CA, USA), (3) air-abrasion with SilJet (30 μm Al<sub>2</sub>O<sub>3</sub>-particles coated with SiO<sub>2</sub>, Danville Materials) with the identical procedure, (4) air-abrasion with SilJet Plus (silanized silica-coated 30 μm Al<sub>2</sub>O<sub>3</sub>-particles, Danville Materials) with the identical procedure, (5) air-abrasion with Aluminum Oxide particles (50 μm, Rønvig Dental, Daugaard, Denmark) with the identical procedure and (6) etching with hydrofluoric acid (Porcelain Etch Gel, 9.6% (Pulpdent Co., Watertown, MA, USA)) for 10 s. All materials used in this study are listed in Table 1. After the surface treatment, all blocks were assigned into different subgroups, identified with a waterproof mark and stored dry at 37 °C (Fig. 1a).

### 2.3. Repair procedure

Half the number of samples per group (n = 8) received a separate silane application (Relyx Ceramic Primer, 3M ESPE) before application of adhesive resin Scotchbond Multipurpose (3M ESPE) without using the primer. The other half of the samples (n = 8) was only treated with adhesive resin without silane. The adhesive was gently air-dried and photopolymerized for 10 s using the LED-polymerization unit (Fig. 1b and c). Using a silicone mold, 'fresh' direct composite resin (Filtek Supreme XTE, color A1D, 3M ESPE) was placed in 2 layers of 2 mm thick and each layer was

**Table 1**  
Materials used in this study.

| Materials                                     | Category                   | Manufacturer                 | Origin            | Lot code(s)                                    | Composition  |
|---|----------------------------|------------------------------|-------------------|--|--|
| LAVA Ultimate CAD/CAM Restorative Estenia C&B | indirect resin composite   | 3M ESPE                      | St. Paul, USA     | N490227 A3-LT Shade                            | Hybrid composite with nanoceramic compounds (ZrO <sub>2</sub> /SiO <sub>2</sub> nanoparticles) embedded in highly cross-linked polymer matrix  |
|   | Indirect resin composite   | Kuraray Noritake Dental Inc. | Okayama, Japan    | Lot n. BS0007; DA3 Shade                       | UTMA, aromatic dimethacrylate, aliphatic dimethacrylate, di-camphorquinone. Alumina microfiller, silanized glass filler, pigments, 92% colloidal silica spheres, 16 wt% microfillers, grain size 0.02 μm, 76% wt microfillers, grain size 2 μm   |
| Air-barrier paste                             | Insulating                 | Kuraray Noritake Dental Inc. | Okayama, Japan    | 6A0001   | Glycerin paste   |
| CoJet Sand                                    | Air abrasion               | 3M ESPE                      | Seefeld, Germany  | 520953   | 30 μm Al <sub>2</sub> O <sub>3</sub> particles coated with SiO <sub>2</sub>  |
| Siljet  | Air abrasion               | Danville Materials           | San Ramon, USA    | 31624  | 30 μm Al <sub>2</sub> O <sub>3</sub> particles coated with SiO <sub>2</sub>  |
| Siljet Plus                                   | Air abrasion               | Danville Materials           | San Ramon, USA    | 116-168B                                       | Pre-silanized 30 μm Al <sub>2</sub> O <sub>3</sub> particles coated with SiO <sub>2</sub>  |
| Aluminium oxide                               | Air abrasion               | Rønvig Dental                | Daugaard, Denmark | Not informed                                   | 50 μm Al <sub>2</sub> O <sub>3</sub>   |
| Porcelain etch gel                            | Etchant                    | Pulpdent Co.                 | Watertown, USA    | 100825   | Hydrofluoric (3 ppm) 9.6%  |
| RelyX Ceramic primer                          | Silane                     | 3M ESPE                      | St. Paul, USA     | N417664  | Silane stabilized alcohol solution   |
| Adper Scotchbond Multipurpose Plus            | Adhesive (bonding)         | 3M ESPE                      | St. Paul, USA     | N421442  | Bis-GMA, HEMA, tertiary amines (light-cure and self-cure initiators), photoinitiator   |
| Filtek Supreme XTE                            | Nanofiller resin composite | 3M ESPE                      | St. Paul, USA     | N272867; N433402; N437301; N4522993; A1D Shade | Combination of aggregated ZrO <sub>2</sub> /SiO <sub>2</sub> cluster filler with primary particle size and a SiO <sub>2</sub> filler (nano agglomerated/nano aggregated), BisGMA, Bis-EMA, UDMA, TEGDMA, hydrophilic dimethacrylate, di-camphorquinone, N,N-Diethanol-p-toluidine, water |

Al<sub>2</sub>O<sub>3</sub> (aluminum trioxide); Bis-GMA (bisphenolA glycidyl dimethacrylate); Bis-EMA (bisphenol-A ethoxylated dimethacrylate); HEMA (2-hydroxyethyl methacrylate); SiO<sub>2</sub> (silicon dioxide); TEGDMA (triethyleneglycol dimethacrylate); UDMA (urethane dimethacrylate); UTMA (urethanetetramethacrylate); ZrO<sub>2</sub> (zirconium dioxide).

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