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Evaluation of microtensile bond strength on ceramic-resin adhesion using two specimen testing substrates

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ABSTRACT

Objectives: The objective of this study was to compare two bonding models using a microtensile bond test and evaluate the effect of two surface treatments on lithium disilicate ceramics using two resin cements.
Methods: Ceramic blocks (e.max CAD®) were sectioned, polished and fired for final crystallization. The blocks were treated with one of two surface treatments: (1) hydrofluoric acid (HF) (IPS Ceramic Etching gel) etched followed by silane (Monobond-S) application; (2) HF etched, silane applied, followed by hot air drying and rinsed with hot water, dried and an unfilled resin (Heliobond) applied. Ceramics without surface treatment were the control. Two bonding substrates were used: resin composite and ceramic with the same surface treatment and the corresponding groups were divided into two bonding models: ceramic to ceramic (C–C) and ceramic to resin composite (C–R). Two resin cements, Variolink II® and Clearfil SA Cement, were tested. Each group ($n=30$) was stored in distilled water for 7 days at 37 °C, then subjected to a tensile force until failure. Failure modes were determined with stereomicroscope and SEM. ANOVA, Bonferroni tests and Weibull analysis were used for statistical analysis ($p<0.05$).
Results: All the control groups experienced spontaneous debonding during preparation. The C–C groups showed significantly higher bond strength than the C–R groups ($p<0.05$). Failure mode in the C–R groups was dominated by cohesive failure in resin cement while in the C–C groups was mostly mixed failure. Ceramic treated with HF etching and silanization and luted with Variolink II showed the highest bond strength (53.5 ± 6.6 MPa) while ceramic treated with HF etching, silanization and hot treatment and luted with Clearfil SA Cement showed the lowest bond strength (35.4 ± 7.0 MPa) in the C–C groups. Weibull analysis showed that Weibull modulus in the C–C model was higher than the C–R model.
Conclusions: Ceramic-bonded to ceramic model is recommended for evaluating the microtensile bond strength of ceramic-resin cement-adhesion. Variolink II showed better bonding than Clearfil SA Cement.

1. Introduction

Nowadays glass ceramics are widely used in dental restorations because of their excellent esthetic performance. However, a major limitation for clinical use of these materials is their comparatively low strength which may lead to fracture and restoration failure [6,7]. IPS e.max Press (Ivoclar Vivadent AG, Schaan, Liechtenstein), a lithium disilicate ceramic, has been introduced with improved mechanical properties compared with other glass ceramics [1]. Recently this system was further enhanced and has exhibited a higher strength and better processing technology than IPS e.max Press. This newer ceramic, IPS e.max CAD, can now be clinically used for 3- to 4-unit bridges and for constructing milled restorations.

The bond to ceramics is of great importance to the long-term success of glass ceramic restorations. Nowadays, maximal preservation of dental hard tissue is advocated in dental treatment [23]. There are increasing cases where the retention of restorations is chiefly reliant on adhesion with the tooth preparation having minimal traditional retentive form, e.g. short crown height. Hence, a durable and reliable bond is required for success of such ceramic restorations. Compared with traditional 'adhesive' luting cements such as zinc polycarboxylate or glass ionomer cement, resin composite luting cements have been introduced to overcome retention problems of all-ceramic restorations. They not only provide a stronger and more durable bond between ceramic materials and tooth structure, but are also more esthetic. Furthermore, it has been found that the ceramic strength is enhanced by the use of resin cements.

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The adhesion of ceramic restorations has two interfaces: the ceramic-resin cement interface and resin cement-tooth interface. A number of studies have been performed evaluating the ceramic-resin bond with the aim of increasing the adhesive strength [10,18,20]. It is well recognized that bonding between ceramics and resin cements can be achieved by two mechanisms: micromechanical attachment and a chemical bond between the ceramic and resin cement. Micromechanical adhesion is created by HF etching [12] and/or grit blasting while a chemical bond is achieved with a silane coupling agent [14]. However, no wide agreement has been reached for the optimal bonding methodology. The traditional method of surface treatment is HF etching followed by silane application to the fitting surface of the ceramic. Nevertheless, a study has shown various bonding procedures to a leucite reinforced ceramic showed an 'optimal' method which was a particular silane treatment with heating of the silanated ceramic [11]. Due to the different chemical composition and microstructure between leucite reinforced and lithium disilicate ceramics, this method may not be the best choice for lithium disilicate ceramics. So far, no study has been performed on testing the bond between lithium disilicate ceramics and resin cement using this 'optimal' method mentioned above.

Of the various laboratory bond test methods available, the microtensile bond strength test is a relatively reliable method which has the potential to reveal a "true" bond strength as failure is of the adhesive. In most studies, to date, the test method employed for resin-ceramic bonding evaluation has used a bonding model that consists of a ceramic block bonded to a resin composite block with a resin luting cement [16,17]. In this model, the bond strength that is of interest is at the interface of the resin cement and ceramic on account that the adhesion to the resin composite is regarded as similar to the resin cement, as well as resin cement to composite is not often used clinically. However, there are cases where fracture may occur at the interface between the resin cement and resin composite or cohesively in the resin composite material. This form of failure is not at the adhesive interface of interest, therefore if such data are included in a study, it will lead to inaccurate outcomes of the test aim. An alternative model is a combination of two ceramic blocks 'luted' with a resin cement [11]. In this model, the fracture would be generated either at the interface of resin-ceramic or cohesively in the resin cement. The results are believed to be more indicative of the 'true' adhesive strength of bonded interface. However, no study has been carried out evaluating the difference of these two bonding models.

Therefore, the aim of this study was to compare the two bonding models (ceramic to resin composite and ceramic to ceramic) using a microtensile bond strength test. In addition, an evaluation of the effect of surface treatments between the traditional method and the 'optimal' method used in studies mentioned above was undertaken using two resin cements. The null hypotheses tested were: (1) there is no difference in microtensile bond strength between two bonding substrates; and (2) there is no difference in microtensile bond strength among different surface treatments and resin luting cements.

2. Materials and methods

The materials, batch number and manufacturers are listed in Table 1.

2.1. Preparation of ceramics

Ceramic blocks (e.max CAD®, Table 1) were sectioned into smaller blocks ($14 \times 12 \times 3$ mm^3 thick) with a self-assembled cutting machine (Miki pulley DC motor, Miki Pulley Co., Ltd, Japan) using an alloy blade. Each block was further polished with 180−, 400−, 600− and 1200-grit SiC papers using a polishing machine (ECOMET 5, Buehler, Düsseldorf, Germany) under running water for 30 min with light hand pressure. The polished surfaces were then ultrasonically cleansed in 95% ethanol for 5 min and air dried for 5 min. After that, the blocks were fired for final crystallization using Programat CS ceramic furnace (Ivoclar Vivadent AG, Schaan, Liechtenstein) following the manufacturer's instructions for e. max CAD.

The polished surfaces were treated using two different surface treatment methods. Specimens in the first treatment group were etched with 4.7% Hydrofluoric acid (Table 1) for 60 s and rinsed with running tap water for 60 s and ultrasonically cleaned with distilled water for 5 min. A silane coupling agent (Table 1) was applied to the etched surface for 60 s. This is the conventional surface treatment method for glass ceramics. Specimens in the other treatment group were etched following the method used in the first group, then the 'optimal' method of silane treatment was applied as described in a previous study [11]. The 'optimal' method is, the silane was applied for 60 s, then hot air dried at 50 ± 5 °C for 15 s with a hair dryer, then the dried silanated surfaces were dipped into 80 °C distilled water for 15 s, dried with hot air for 30 s, then a thin layer of unfilled resin (Table 1) was applied to the treated surface. Polished surfaces without any surface treatment were used as control groups.

2.2. Preparation of resin composite

Resin composite (Table 1) blocks were made $14 \times 12 \times 3$ mm^3 thick, the same dimensions as the ceramic block, by layering 3 mm-thick increments in a silicone mold, and light-cured for 40 s for each increment with a LED light curing unit (Elipar™ 2500, 3 M ESPE, Seefeld, Germany). The last increment was condensed using a flat glass slide to obtain a flat surface. The five surfaces of the composite that contacted with the silicone mold were further irradiated for another 40 s for each side after the removal of the specimens (total 200 s). The blocks were then taken to be heat cured at 100 °C for 15 min following the manufacturer's instructions. The to-be-bonded surface was polished with 500-grit SiC paper under running water followed by ultrasonic cleansing in distilled water for 5 min and air dried for 5 min.

2.3. Luting procedure

Two bonding models were used in this experiment. The first was two ceramic blocks 'luted' with one of the two resin cements, while the other model used a ceramic block 'luted' to a resin composite block with resin cement. The specimens were further subdivided into two groups by using two different resin cements: Variolink II® (Table 1) and Clearfil SA Cement (Table 1). The bonding procedures were carried out using a loading device that produced a constant seating load of 2 N (0.2 kg), applied for 5 min when the two blocks were bonded together. Excess cement was removed with a scalpel blade before complete setting of the cement. Specimens were photo-activated for 20 s on each side of the block with the LED curing light (total 80 s). The ceramic-to-composite luting procedures were repeated in the same manner.

2.4. Microtensile bond strength test

Each bonded block was fixed with sticky wax to the platform of the cutting machine, and was vertically sectioned into a series of 1 mm thick slices using a water-cooled rotating diamond saw blade. The block was rotated 90° and the cutting procedure repeated. Beams were obtained from each block with approximately 1.0 mm^2 cross-sectional areas. Thirty beams were prepared