

available at [www.sciencedirect.com](http://www.sciencedirect.com)journal homepage: [www.intl.elsevierhealth.com/journals/dema](http://www.intl.elsevierhealth.com/journals/dema)

# Enhanced resin-composite bonding to zirconia framework after pretreatment with selected silane monomers

Jukka P. Matinlinna<sup>a,\*</sup>, Lippo V. Lassila<sup>b</sup>

<sup>a</sup> Dental Materials Science, Faculty of Dentistry, The University of Hong Kong, Hong Kong SAR, PR China

<sup>b</sup> Department of Biomaterials Science, Institute of Dentistry, The University of Turku, Turku, Finland

## ARTICLE INFO

### Article history:

Received 25 February 2010

Accepted 1 November 2010

### Keywords:

Primers

Resin bonding

Silica-coating

Shear bond strength

Silanization

Zirconia

## ABSTRACT

**Objective.** To evaluate the effect of five experimental silane monomer primers in vitro on the shear bond strength of a phosphate ester resin-composite cement bonded to a silicized zirconia framework.

**Methods.** A total of 144 planar zirconia (Procera AllZircon) specimens were subjected to tri-bochemical silica treatment, randomly divided into 12 sub-groups ( $n=12$ ), and silanized with 1.0% (v/v) activated solutions of 3-acryloxypropyltrimethoxysilane, 3 glycidoxypropyltrimethoxysilane, 3-methacryloxypropyltrimethoxysilane, styrylethyltrimethoxysilane, and 3-isocyanatopropyltriethoxysilane, which had been prepared in 95% ethanol (pH 4.5). A ready-to-use 3-methacryloxypropyltrimethoxysilane (RelyX<sup>TM</sup> Ceramic Primer) was used as the control. One resincomposite cement (RelyX<sup>TM</sup> Unicem) stub was bonded to each silicized and silanized zirconia specimen. Half of the specimen groups were dry-tested and half were thermo-cycled at 6000 cycles between 5 °C and 55 °C, with a constant dwelling time of 30 s. The shear bond strengths of the cement stubs bonded to zirconia were measured using a universal testing machine using a constant cross-head speed of 1 mm/min. The silane primer activation was evaluated using Fourier-transform infrared spectroscopy.

**Results.** The highest shear bond strength was obtained for 3-acryloxypropyltrimethoxysilane in dry storage, 11.7 MPa (SD, 2.3 MPa) and after thermo-cycling 17.6 (4.1) MPa for glycidoxypropyltrimethoxysilane. The lowest shear bond strength values were obtained with control silane: in dry storage, 4.5 (1.3) MPa, after thermo-cycling 6.5 (2.6) MPa. Thermo-cycling increased the bond strengths significantly (ANOVA,  $p < 0.001$ ) and differently for each type of silane (ANOVA,  $p < 0.001$ ).

**Significance.** Silanization with five experimental silane primers in vitro produced significantly greater shear bond strengths than the ready-to-use control silane.

© 2010 Academy of Dental Materials. Published by Elsevier Ltd. All rights reserved.

## 1. Introduction

Biocompatible yttrium-stabilized tetragonal zirconia (ZrO<sub>2</sub>) is structurally stable and stiff and thus suited as a framework material. It has no known adverse tissue reactions and serves

at high simulated masticatory loads [1]. However, the reliable and durable clinical cementing of CAD/CAM-zirconia remains a clinical concern [2]. Pretreatment methods that modify the surface texture or chemistry to enhance durable adhesion have thus been investigated or proposed [3–7]. Some special resin-composite cements have also been developed. These

\* Corresponding author. Tel.: +852 2859 0380; fax: +852 2548 9464.

E-mail address: [jpmat@hku.hk](mailto:jpmat@hku.hk) (J.P. Matinlinna).

0109-5641/\$ – see front matter © 2010 Academy of Dental Materials. Published by Elsevier Ltd. All rights reserved.

doi:10.1016/j.dental.2010.11.002

cements may contain molecules with acidic phosphate ester groups, as in 10-methacryloyloxydecyl-dihydrogenphosphate in Panavia™ (Kuraray, Osaka, Japan) [8], or other multifunctional molecules with methacrylate and phosphoric ester groups, as in RelyX™ Unicem (3M ESPE, Seefeld, Germany) [5]. Both of these products have demonstrated durable bonding [9,10]. The bond strengths of other commercially available adhesive systems to zirconia have also been documented but vary in magnitude [5].

Silane coupling agents are widely used in the silanization of fillers in resin-composite. Furthermore, dual-functional silane monomers can be used as coupling agents to promote adhesion between dissimilar matrices in composites and coatings, such as silica-coated materials in dentistry, because their organofunctional group polymerizes with hydrophobic resin-composite monomers while three hydrolyzable alkoxy groups bond with hydrophilic silica and silica-coated surfaces [11]. Recent studies have shown that experimental silane monomer primers can significantly increase the microtensile bond strength between resin and zirconia [4,7]. However, inert zirconia itself is not amenable to silane-based methods of adhesion promotion [3–5]. A pretreatment method is thus widely used—namely, tribochemical silica-coating in the Rocatec™ system, in which silica-coated alumina particles are blasted onto the zirconia surface [12–14]. This method removes impurities and creates a highly microretentive surface with partial silicon dioxide coverage [15], all without any weakening in its flexural properties [16]. After this pretreatment step, immediate silanization can promote bonding to resin composite [11].

The rationale behind applying experimental silane primers is that the presence of specific organofunctional groups, such as an aromatic reactive styryl [17], isocyanato [18], glycidoxo (with epoxy ring), or acrylate [19,20], may improve the spatial compatibility of the silane molecule. This steric improvement may also increase the reactivity of silane monomers by enabling polymerization reactions between methacrylate, acrylate and styryl groups and, on the other hand, with methacrylate and phosphate ester groups in resin-composite. Now, adhesion would be promoted by the formation of new covalent bonds and a 3-dimensional network of siloxane bonds. The resin-composite cement (RelyX Unicem) is initially acidic (pH 2) owing to its phosphate groups but the pH rises within a couple of minutes. On the other hand, silanes need an acidic environment to catalyze their hydrolysis (activation) and allow them to polymerize with the monomers of the resin-composite cement [11].

This study tested the hypothesis that experimental primers of silane monomers with isocyanato, styryl, glycidoxo, methacrylate or acrylate functional groups significantly enhance bonding (shear bond strength) between an acidic resin-composite and silica-coated zirconia, when compared with a commercially available control silane with methacrylate functionality.

## 2. Materials and methods

All experimental materials of this study are listed in Table 1; all manufacturers' instructions were followed and all tests

were performed by one operator. The zirconia specimens and resin-composite cement stubs were prepared in a standardized way. First, 144 planar zirconia specimens (surface area, 10 mm × 10 mm; thickness, 3 mm) were embedded in acrylic denture resin blocks to leave one surface free. The surface was kept intact and free of any acrylic resin remains (visual check). Uniform abrasion pretreatment with Rocatec™ Plus sand (grain diameter, 110 μm) was performed for 60 s with an operational pressure of 300 kPa at a perpendicular position approximately 10 mm away and with slow rotation of the exposed surface of ca. 1.0 cm<sup>2</sup>. The specimens were cleaned in ethanol for 10 min in an ultrasonic bath, air dried, and protected from dust. The specimens were divided randomly into two storage groups: (a) to be kept dry and (b) to be thermo-cycled.

### 2.1. Activated trialkoxysilanes and silanization

The five experimental silane primers were used at 1.0% (v/v) in a standard solution of 95.0% (v/v) ethanol and deionized water (Milli-Q purification system, Millipore) that had been adjusted to pH 4.5 with 1 M acetic acid. The solution was first allowed to stabilize for 24 h and were then silane monomer was added and allowed to activate for 1 h at room temperature [4,7,18–20]. Silica-coated zirconia samples were randomly divided into 12 sub-groups ( $n=12$ ) for silanization by experimental or commercially available ready-to-use primer. Each silane primer coating was applied with a new, clean brush. The silane was allowed to dry and react for 3 min, and then gently dried with oil-free compressed air (Fig. 1).

### 2.2. Fourier-transform infrared spectroscopic analysis

Hydrolysis of each silane monomer was observed analytically for up to 60 min using reflectance-absorbance Fourier-transform infrared spectroscopy (Spectrum One spectrometer; Perkin-Elmer, Beaconsfield, UK) to detect molecular bending, vibration, wagging, and rocking of functional groups [21]. The surface analysis of a silane primer film layer was conducted throughout the spectral range 3800–600 cm<sup>-1</sup> with a specular reflectance monolayer and grazing angle accessory in which the primer film was placed against a cleaned, planar, inert Ge crystal. The resolution was 2 cm<sup>-1</sup> and 32 scans were taken [18,19].

### 2.3. Bonding of the resin-composite cement and testing

RelyX™ Unicem cement stubs were carefully prepared and bonded to the silica-coated silanized zirconia specimens using polyethylene molds (diameter, 3.6 mm; height, 5.0 mm). The cement was carefully packed against the substrate and the stubs were light-polymerized for 40 s (light-intensity, 470–520 mW/cm<sup>2</sup>; wavelength, 490 nm) from the top of the stub and also from two lateral directions at the contact area. The mold was gently removed and the specimens in the dry group were kept in a desiccator for about 2 h. One half of the specimens were subjected to thermo-cycling in de-ionized water for 6000 cycles between 5 °C and 55 °C. The dwelling time at each temperature was 30 s and the transfer time was 2 s (ISO Standard 10477), and one half, six groups, to dry storage in desiccators [22].

Download English Version:

<https://daneshyari.com/en/article/1421909>

Download Persian Version:

<https://daneshyari.com/article/1421909>

[Daneshyari.com](https://daneshyari.com)