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Shear bond strengths of various luting cements to zirconia ceramic: Surface chemical aspects

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ABSTRACT

Objectives: To measure the shear bond strengths of various luting cements to a sandblasted zirconia ceramic and to determine the surface energy parameters of the luting cements.
Methods: Two conventional glass ionomer cements, two resin-modified glass ionomer cements, two compomer cements, and two adhesive resin cements were prepared and bonded to sandblasted zirconia (Lava). All bonded specimens were stored in water at 37 °C for 48 h and then half of them additionally thermocycled 10,000 times prior to the shear bond strength test ($n = 10$). Surface roughness (R_a) values and surface energy parameters of the eight luting cements and polished zirconia ceramic were evaluated using a profilometer and contact angle measurements, respectively ($n = 10$). The bond strength and surface roughness data were statistically analysed using non-parametric and parametric procedures, respectively ($\alpha = 0.05$). Relationships between surface energy parameters and measured shear bond strengths were investigated using the Spearman rank correlation test.
Results: Panavia F 2.0 and Principle produced higher bond strengths than the other cements, with no significant changes before and after thermocycling. Fuji I, Ketac Cem Easymix, and Ionotite F yielded near-zero or zero values after thermocycling. All debonded specimens showed adhesive failure. Mean R_a values ranged from 0.104 to 0.167 μm. We found the base (hydrogen bond accepting) components of the luting cements significantly affected the bond strengths both before and after thermocycling.
Conclusion: It is recommended that the surface energy parameters of luting cements be considered in evaluating their adhesive properties with zirconia ceramic.

1. Introduction

Modern all-ceramic dental restorations provide cosmetically good and metal-free treatment options. Amongst various ceramic systems, zirconium-oxide ceramics based on yttria-stabilized polycrystalline tetragonal zirconia (Y-TZP) have become favoured, especially where high functional demands must be met.[1]

However, the surface characteristics of the zirconia ceramic make it difficult to establish a durable mechanical or chemical bond in zirconia-based restorations.[2,3] To use the durable siloxane bond between silica and a silane coupling agent, tribochemical silica coating followed by silanization

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was introduced to improve bond strength with zirconia ceramic. As several studies using this method have yielded inconsistent bond strength data,[4,5] the method seems unlikely to provide a reliable durable bond with zirconia ceramic. In contrast, it has been shown that a combination of sandblasting and resin luting agents containing organophosphate ester monomers, such as 10-methacryloyloxydecyl dihydrogenphosphate (10-MDP), may result in a relatively high and durable bond to zirconia ceramic.[2,3] However, there are insufficient data on the actual bonding mechanism of the 10-MDP monomer to zirconia ceramic and the long-term *in vivo* performance of the 10-MDP-containing resin luting cements.[6,7]

In clinical practice, full-coverage zirconia ceramic restorations and fixed partial dentures not requiring high retention may be luted using various luting cements, whether resin-based or non-resin-based.[8,9] Nonetheless, a 3-year clinical follow-up study suggests that loss of retention of zirconia ceramic restorations is related to the cement used.[10] Except for zinc phosphate cements,[11] most of these luting cements are expected to adhere to zirconia ceramic due to the presence of functional polymers or monomers in their composition.[12] As the luting cements containing functional polymers or monomers seem to have differing surface energies, there may arise different interfacial chemical interactions and bonding potentials with zirconia ceramic. Asmussen and Peutzfeldt[13] demonstrated that the surface energy parameters of composite and adhesive-treated dentine influence bond strength. In their study, bond strength increased with the ''base'' component of the surface energy of the resin composites and dentine treated with a higher surface energy adhesive yielded higher bond strengths. However, these features have not been extensively researched in the field of adhesion to zirconia ceramic.

The purpose of this study was thus to evaluate the shear bond strengths of various luting cements to a sandblasted zirconia ceramic and to test their bonding durability through thermocycling. The chemical composition of the luting cements was analysed using Fourier transform infrared (FTIR) spectroscopy. The surface roughness of luting cements and zirconia ceramic was measured using a profilometer. Surface energy parameters were determined by measuring contact angles on the eight luting cements and zirconia ceramic surface. Relationships between surface energy parameters and measured shear bond strengths were investigated using the Spearman rank correlation test.

2. Materials and methods

2.1. *Shear bond strength test*

Two conventional glass ionomer cements (GICs) (Fuji I, FI; Ketac Cem Easymix, KC), two resin-modified glass ionomer cements (RMGICs) (Fuji Plus, FP; RelyX Luting, RL), two compomer cements (Principle, PR; Ionotite F, IT), one adhesive resin cement (Panavia F 2.0, PV), and one self-adhesive resin cement (RelyX Unicem, UC) were selected for this *in vitro* study. Their codes, manufacturers, and compositions are summarized in Table 1.

Thirty-two cylinder-shaped (20 mm diameter and 1.5 mm thickness) commercial zirconia ceramic discs (Lava, 3 M ESPE, Seefeld, Germany) were prepared according to the manufacturer's instructions and embedded in round silicone rubber moulds (25.4 mm in inner diameter, 19.0 mm in height) using poly(methyl methacrylate) resin, ensuring that one surface of the zirconia disc remained uncovered for adhering to cement. The exposed surface of each specimen was sandblasted with 110 μm Al_2O_3 from a distance of 10 mm perpendicular to the specimen surface at a pressure of 0.25 MPa for 13 s and ultrasonically cleaned in isopropyl alcohol for 3 min.[2,14] The sandblasted and ultrasonically cleaned zirconia ceramic surfaces were examined using an optical microscope (MM-40, Nikon, Tokyo, Japan) at a magnification of 100× and 500×.

To prepare hollow cylinders for bonding, clear and flexible polyvinyl chloride tubing (CFT-02-C, Small Parts Inc., Miami Lakes, FL, USA) with an inner diameter of 3.175 mm and an outer diameter of 6.35 mm was used. The tubing was inserted inside a long, clear acyclic split mould (inner diameter: 6.5 mm) so that 1.0 mm height of the tubing protruded perpendicularly from one surface of the mould. The protruding portion was cut off by moving a sharp razor blade (ST-300, Dorco Co., Ltd., Seoul, Korea) along the surface. In this manner, precisely cut tubings (approximately 1.0 mm in height) were obtained. The luting cements were mixed following manufacturers' instructions and carefully inserted into the tubing lumens, ensuring that the tubing did not move or slip on the surface by pressing the tubing with a custom-made wire loop tool during the procedure. The materials were allowed to self-cure. The PR, PV, and UC cements were additionally irradiated for 20 s by holding the tip of the light guide of a light-curing unit (Elipar TriLight, 3 M ESPE; standard mode, output intensity = 750 mW/cm^2) approximately 1 mm above the cement-filled tubing. All specimens were left undisturbed for 30 min in 100% humidity[15] and the tubing around cement cylinders was removed by vertically cutting the tubing into two or more fragments using a sharp blade (KB, Olfa Corp., Osaka, Japan). Special care was taken not to apply any stress or damage to the boned material. Cement cylinders that showed any apparent defects under an optical microscope (SMZ800, Nikon Corp., Tokyo, Japan) were excluded from this study and replaced. In this manner, five bonded cement cylinders of each luting cement were arranged in a circle on one zirconia surface (n = 10). Prior to debonding, all bonded specimens were stored in distilled water at 37 °C for 48 h. Half of them were additionally thermocycled 10,000 times between 5 °C and 55 °C waterbaths with a dwelling time of 30 s and an exchange time of 5 s between each bath (n = 10).[16]

The bonded specimens were mounted in a jig attached to a universal testing machine (3343, Instron Inc., Canton, MA, USA). A stainless steel wire loop (0.41 mm diameter; G&H Wire Company, Greenwood, IN, USA) was placed around the cement cylinder so that it made contact with the lower half-circle of the cylinder and touched the zirconia surface.[17] Each cylinder was stressed one by one by turning the specimen at a crosshead speed of 1 mm/min and the maximum load at failure was recorded, then converted to MPa. Following debonding, all fractured interfaces were examined under an optical microscope (SMZ800) to determine