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The development and testing of glaze materials for application to the fit surface of dental ceramic restorations

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ABSTRACT

Objectives. The aims of the study were to develop and test overglaze materials for application to the fit surface of dental ceramic restorations, which could be etched and adhesively bonded and increase the flexural strength of the ceramic substrate.

Methods. Three glaze materials were developed using commercial glass powders (P25 and P54, Pemco, Canada). P25 (90 wt%) was mixed with P54 (10 wt%) to produce (P25/P54). P54 (90 wt%) was mixed with P25 (10 wt%) to produce (P54/P25). P25 (90 wt%) was mixed with 10 wt% of an experimental glass powder (P25/frit). Eighty-two disc specimens (14 mm × 2 mm) were produced by heat pressing a leucite glass–ceramic and were sandblasted with 50 μm glass beads. Group 1 control specimens (10) were sandblasted. Groups 2–4 (10 per group) were coated using P25/frit (Group 2), P25/P54 (Group 3) and P54/P25 (Group 4) overglazes before sintering. Groups 1–4 were etched for 2 min using 9.5% HF (Gresco, USA). Composite cylinders (Marathon[®] v, Den-Mat) were light cured and bonded to the glazed and prepared disc surfaces and groups water stored for 8 days. Groups were tested using shear bond strength (SBS) testing at 0.5 mm/min. Disc specimens (42) were tested using the biaxial flexural strength (BFS) test at a crosshead speed of 0.15 mm/min. Group 1 was tested as sandblasted (21) and Group 2 (21) after coating the tensile surface with P25/frit. Xrd, Eds and Sem analyzes were carried out.

Results. Mean SBS (MPa ± S.D.) were: Group 1: 10.7 ± 2.1; Group 2: 9.8 ± 1.9; Group 3: 1.8 ± 1.0 and Group 4: 2.6 ± 1.7. Groups 1 and 2 were statistically different to Groups 3 and 4 ($p < 0.001$), but there was no difference between Groups 1 and 2 and 3 and 4 ($p > 0.05$). The mean BFS (MPa ± S.D.) of the overglazed Group 2 (200.2 ± 22.9) was statistically different ($p < 0.001$) to Group 1 (150.4 ± 14.3).

Significance. The P25/frit overglaze significantly ($p < 0.001$) increased the biaxial flexural strength of the leucite glass–ceramic substrate and produced comparable shear bond strengths to an etched and bonded control. The application of etched overglaze materials to dental glass–ceramic and ceramic substrates may be useful in adhesive dentistry.

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1. Introduction

The processing of all ceramic materials to construct dental restorations can result in a wide variety of flaw populations [1] due to the process of machining [2], sintering [3] or heat pressing [4]. Removal of investments and preparation for bonding by sandblasting can modify this defect population and its geometry. This can result in a population of flaws in the material which can induce strength reductions and variability [5]. Bhamra et al. [6] associated a reduction in the coarseness of surface flaws with increased flexural strength, Weibull modulus and survival probability in aluminous core materials. The surface preparation of glass–ceramic materials associated with clinical/laboratory procedures including sandblasting, polishing and grinding, have been linked to the reliability of these materials [7]. Glazing of external dental ceramic surfaces has traditionally been recommended using auto or overglaze techniques, to counteract the effects of coarse surface finish [8] and to obtain a sealed hygienic surface. Auto-glazing utilizes the further advancement of the sintering process in air, whilst overglazing techniques rely on the outer surface application and firing of thermally compatible low fusion glasses. Overglazing of ceramics has in many instances increased the strength of ceramic materials because of the glaze's ability to reduce surface flaws and the creation of surface compression upon cooling [9]. Overglazing of dental ceramics has also yielded higher flexural strength values than auto-glaze techniques [10,11], when these treatments were placed in tension. Fairhurst et al. [12] reported auto-glazing as detrimental to the biaxial flexural strength of some leucite containing porcelains, due to the introduction of larger intrinsic flaws during glaze firing. Glaze strengthening may depend on whether the ceramic substrate has a strength dependence on microstructure rather than the surface [13]. Lüthy et al. [14] applied overglaze to the tensile test surface of a leucite-glass ceramic and reported a significant flexural strength increase from 159 ± 28 (no overglaze) to 220 ± 34 MPa $\pm$ S.D., associated with the glaze layer addition. Transverse strength increases were similarly noted when overglaze was applied to the tensile surface of magnesia core ceramic beam specimens [15]. Overglazing the interior of all ceramic restorations may therefore be useful, since reported clinical failures are related to the propagation of flaws on the inner restoration surface [16], in areas of high tensile stress [17] and accelerated by the presence of water [18]. Although glaze derived strength increases are advantageous in these brittle systems, the application of a single-phase glass on the inner glass–ceramic restoration surface should also be considered in terms of its ability to be efficiently etched and adhesively bonded.

Currently leucite glass–ceramics are retained by acid etching and bonding via silane coupling agents and composite resins to etched tooth structure. Acid etching of the two-phase glass–ceramic surface creates micromechanical retention to promote the mechanical interlocking of the composite luting cement [19]. The application of silane bonding agents (organosilanes hydrolyzed to form organosilanols) wet the ceramic surface. There is initial hydrogen bond formation between the silane bonding agent and surface hydroxyl groups, and on drying the formation of covalent bonds,

through a condensation reaction [20]. The organo-functional group in the silane can next form a bond with the composite lute enhancing the bond strength [21,22]. Ceramic strengthening is also promoted by this procedure [23], which was thought to be associated with reduction of ceramic surface flaws by the resin [24]. The interaction of the resin with the entire flaw population, creation of an interfacial resin–ceramic composite hybrid layer [25] and transfer of the fracture origins form the ceramic/resin interface [26], more fully explains ceramic strengthening. The creation of surface compression by the polymerization shrinkage of composite luting cements [23] and the ability of the composite lute to act as a shock absorber and transfer the occlusal load to the supporting tooth structure may be other factors [27]. Six-year clinical trials for adhesively bonded leucite glass–ceramic restorations indicated Kaplan Meier survival rates of 94.9% for inlays and onlays [28] and 89.2% for full crowns [29]. These values decreased to 91.1% [30] and 85.5% [31] respectively, for a 7-year period. The application of a glaze layer on the interior of these restorations may be useful in terms of the ability of the glaze to wet the surface, eliminate flaws and produce surface compression. The aims of the study were therefore to develop and test thermally compatible overglaze materials for application to the fit surface of dental ceramic restorations, which could be etched and adhesively bonded and increase the flexural strength of the ceramic substrate.

2. Materials and methods

2.1. Preparation of the experimental overglaze materials

Three overglaze materials were developed. The two commercially available glass powders selected were P25 and P54 glasses (Pemco Corp., Baltimore, Canada, Table 1). The glasses were weighed using an electronic balance (Mettler ME-33360, Mettler Instruments, High Wycombe, Bucks., UK). The P25 glass powder (90 wt%) was mixed with 10 wt% of P54 glass powder to produce the first glaze material (P25/P54). The P54 glass powder (90 wt%) was mixed with 10 wt% of P25 glass to produce the second glaze material (P54/P25). The third glaze material was produced by mixing (90 wt%) of P25 glass powder with 10 wt% of an experimental aluminosilicate glass powder (P25/frit). The experimental glass powder was produced by mixing glass reagents in a baffle jar on a jar roll for 2 h. The

Table 1 – Chemical composition of the glaze materials.

Glass composition	P25 glass weight (%)	P54 glass weight (%)
SiO ₂	49.7	46.3
Al ₂ O ₃	12.1	0
B ₂ O ₃	16.9	23.3
Na ₂ O	14.7	10.4
K ₂ O	5.40	0
CaO	0.50	20.0
ZnO	0.70	0
F	1.80	0

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