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Evaluation of modern bioactive restoratives for bulk-fill placement

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ABSTRACT

Objective: To investigate the impact of storage up to one year on the micro-mechanical properties of dental bioactive restoratives (R) used for bulk-application and their intermediate layer (IL) to dentin (D).
Methods: One giomer bulk-fill resin composite (RBC) and one glass-ionomer cement (GIC) were applied in 5-mm deep Class I cavities, which were prepared in 50 sound human molars. Specimens were stored in distilled water at 37 °C for 24 h, one week, one month, three months and one year. The variation in micro-mechanical properties (indentation modulus (Y_{HU}) and Vickers hardness (HV)) were determined in 100-μm steps in line-profiles located parallel to the tooth axis, starting 0.1 mm from the filling surface, through the IL in between R and D, and ending 300 μm in D.
Results: HV and Y_{HU} were influenced by the material ($p < 0.001$, partial eta-squared $\eta^2_P = 0.011$ and $\eta^2_P = 0.395$), immersion duration ($\eta^2_P = 0.081$, $\eta^2_P = 0.081$) and depth ($\eta^2_P = 0.050$, $\eta^2_P = 0.091$). The micro-mechanical properties progressively increased with immersion time in both restoratives, with a comparable time-dependency pattern, but to a different extent. IL showed weaker mechanical properties compared to more superficial cavity areas, which were maintained in RBC and even enhanced in GIC during storage. After one year of storage HV ranked in the following sequence: GIC: D: 61.4 ± 7.25-N/mm^2, IL: 86.0 ± 23.63-N/mm^2, R: 112.3 ± 30.36-N/mm^2 and RBC: D: 67.2 ± 11.55-N/mm^2, IL: 76.7 ± 18.88-N/mm^2, R: 94.1 ± 12.28-N/mm^2.
Conclusion: No degradation with aging was identified in the giomer restorative. The gradual ascending transition in micro-mechanical properties from D through R identified in both restoratives might have a positive effect on the bond quality.
Clinical significance: Giomers represent a new category of restoratives with promising clinical behavior and good mechanical stability up to one year of aging under simulated clinical conditions.

1. Introduction

The current trend in the development of aesthetic restorative materials has been marked by the increased demand for bioactive restoratives able to prevent the recurrence of carious lesions. Though less aesthetic, glass ionomer cements (GIC) are reliable bioactive materials owing to their biocompatibility, long-term fluoride release, bulk-application prospects, and ability to adhere to moist enamel and dentin without necessitating an intermediate agent [1–5]. The susceptibility to water uptake and water loss, particularly in the initial setting reaction, however, is crucial for their clinical performance [6]. Derived from the fluoride-releasing mechanism of GICs during the acid-base reaction phase, Roberts et al. [7] developed in 1999 a pre-reacted glass-ionomer (PRG) filler that can be incorporated into resinous materials. The particles are made of fluoroaluminosilicate glass that has been reacted with a polyalkenoic acid in the presence of water to form a wet siliceous hydrogel. Freeze-dried, the desiccated xerogel is further milled and silanized [7,8]. A stable PRG filler with a trilaminar structure is consequently formed, and is described as allowing the release and recharge of fluoride by a ligand exchange mechanism within the pre-reacted hydrogel [9–16]. Besides fluoride, the release of ions such as Na^+, Sr^{2+}, Al^{3+}, BO_3^{3-}, and SiO_3^{2-} was also observed [9]. The pre-reaction can involve only the surface of the glass particle (surface pre-reacted glass ionomer, S-PRG) or the entire particle (fully pre-reacted glass ionomer, F-PRG) [7]. Materials based on PRG fillers and resins delineate a new material category, the giomers (glass ionomer + polymer), and appear in modern dentistry as restoratives [14], adhesives [17], coatings [18–20], fissure sealants [15] or endodontic sealers [16].

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Previous research has ascribed to materials based on S-PRG a certain role in preventing demineralization [19,21,22] or in inducing remineralization [23]. When used as coatings for artificial enamel lesions in primary teeth, a partial remineralization was acknowledged, but the lesion was not arrested [18]. Exposed human root dentin was effectively protected in vitro [20], while the application of an endodontic sealer with S-PRG induced a continuous release of ions, which were subsequently incorporated by the root canal dentin [16]. S-PRG fillers incorporated in a bonding agent enhanced radiopacity [17], while their use in GICs improved compressive strength and handling characteristics [24]. In experimental resin-based composites (RBC), S-PRG fillers caused a reduction in biofilm formation, but a decrease in fluoride release over time was observed as well (48 h vs. 120 h) [14].

Currently, S-PRG fillers are incorporated also in bulk-fill RBC. In previous works, it has been shown that the materials in both low and high viscosity consistencies reached adequate depths of cure (>4 mm), good micro-mechanical properties [25] and low polymerization shrinkage [26] when compared to clinically established materials. Their long-term stability, however, has not been investigated yet. Notably, previous researchers have highlighted conflicting results regarding the effect of long-term immersion in aqueous solutions on the mechanical properties of both material categories, RBCs [27,28] and GICs [29–32]. Apart from material properties, the time-dependent interaction of the restoratives with the tooth structure is of particular importance [33,34]. When using GIC as a restorative in deep Class I cavities, an intermediate dentin–GIC layer has been previously described, with weaker mechanical properties compared to more superficial material areas, which are assumed to be the result of multiple ion diffusion processes between dentin and GIC [32].

This study aims therefore to compare up to one year of immersion in water two bioactive restoratives, a GIC and a bulk-fill RBC with S-PRG fillers, placed in bulk in 5-mm deep Class I cavities. The hypotheses tested were these: (a) within one restorative, there would be no significant difference in the measured properties, neither within different depths of the restorative material nor among different regions in the restoration, assigned as restorative material (R), intermediate layer (IL) and dentin (D); (b) the immersion duration (24 h to one year) would not alter the measured properties; (c) there would be no significant difference in the measured properties between analyzed materials.

2. Materials and methods

One giomer bulk-fill RBC (BEAUTIFIL-Bulk Restorative, Shofu, Kyoto, Japan, shade Universal, Lot 11402) and one GIC (Ketac^{TM} Molar Aplicap^{TM}, 3 M ESPE, Seefeld, Germany, shade A3, Lot 532337) were selected. Ten sets of five extracted non-carious human molars were selected and stored in a 1% sodium azide aqueous solution. Teeth were well cleaned with distilled water before receiving a deep Class I cone cavity (5 mm in depth and 3 mm in diameter at ground level) standardized by using a diamond bur with the shape of a truncated cone 3 mm in diameter at its bottom. The fillings were prepared at room temperature according to the manufacturer's instructions. The cavities were cleaned with water and dried gently before the restorative material was applied. The encapsulated GIC was mixed by rotating in a RotoMix (3 M ESPE) apparatus for 10 s and applied directly in the cavity. A filled, light-cure total-etch dental adhesive (Optibond^{TM} FL, Kerr, Orange, CA, USA) was used as an intermediate agent in teeth restored with the bulk-fill RBC, while the RBC was applied in one 5-mm increment and cured for 20s (LED LCU Freelight 2, 3 M ESPE, 1200 mW/cm^2). Tooth specimens were then stored in distilled water at 37 °C for 24 h, one week, one month, three months and one year. The distilled water was renewed daily for one-week immersion and weekly for longer immersion conditions. Prior to measurement, the teeth were cut mesio-distally through the center point of the cavities by a circular saw (Isomet Low Speed Saw, Buehler, Lake Bluff, IL, USA) to obtain a cross-sectional area. The surface of the cross section was wet-grounded with 2500- and 4000-grit silicon-carbide paper (FEPA, Hermes, Hamburg, Germany). The mechanical properties Vickers hardness (HV) and indentation modulus (Y_{HU}) were determined by means of an automatic micro-hardness indenter (Fischerscope H100C, Fischer, Sindelfingen, Germany). Therefore, measurements were done in 100-μm steps starting at 0.1 mm from the surface in the middle of the filling and ending approximately 300 μm within the dentin. Two parallel line-profiles were performed in each slice approx. 500 μm from each other. The measurements allowed differentiating among the properties of the restorative material (R), the intermediate layer (IL) which was defined as the last 300 μm of the restorative material above dentin, and the dentin (D) adjacent to the restorative material at the bottom of the cavity. An integrated light-microscope was used to verify the position of the indentation after measurement and to differentiate clearly among these regions.

For statistical analysis the values measured in the restorative material at different positions were grouped in intervals as follows: starting from the top to the bottom of the filling, values measured within the depth interval (0.1–1.0) mm were assigned as 1-mm-depth; values measured within the interval (1.1–2.0) as 2 mm and so forth to 5 mm. The IL and D were considered as described above.

The test procedure was force-controlled; the test load increased and decreased with constant speed between 0.4 mN and 500 mN. The load and the penetration depth of the indenter were continuously measured during the load-unload hysteresis. The Universal hardness (Martens hardness) is defined as the test force divided by the apparent area of the indentation under the applied test force. From a multiplicity of measurements, a conversion factor between Universal hardness and Vickers hardness was calculated and implemented in the software, so that the measurement results were indicated in the more familiar Vickers hardness (HV) units. The indentation modulus (Y_{HU}) was calculated from the slope of the tangent of indentation depth-curve at maximum force (DIN-50359-1 [35]).

Table 1
Restorative material.

Restorative	Parameter	Depth		Immersion duration		Depth x immersion duration	
		p	η^2_P	p	η^2_P	p	η^2_P
Ketac Molar	HV	$\le$0.001	0.084	$\le$0.001	0.110	$\le$0.001	0.016
	Y_{HU}	$\le$0.001	0.126	$\le$0.001	0.120	0.015	0.045
Beautifil Bulk	HV	$\le$0.001	0.022	$\le$0.001	0.115	$\le$0.001	0.037
	Y_{HU}	$\le$0.001	0.057	$\le$0.001	0.113	$\le$0.001	0.070

Results of the two-way ANOVA (general linear model); the significance value, p, and the partial eta-squared value, η^2_P, are indicated.