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The effect of desiccation on water sorption, solubility and hygroscopic volumetric expansion of dentine replacement materials

Ruba Mustafa^{a,b}, Ruwaida Z. Alshali^c, Nick Silikas^{a,*}

^a School of Dentistry, The University of Manchester, Manchester, UK

^b Conservative and Department, Faculty of Dentistry, Jordan University of Science and Technology, Irbid, Jordan

^c Oral and Maxillofacial Prosthodontics Department, Faculty of dentistry, King Abdulaziz University, Jeddah, Saudi Arabia

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ABSTRACT

Objective. To evaluate water sorption, solubility and hygroscopic expansion and the effect of desiccation for a calcium silicate-based material, a conventional glass ionomer, and a resin-modified glass ionomer.

Methods. Water sorption, solubility and hygroscopic expansion of Biodentine™ (BD), GC Fuji IX GP® FAST (FJ), and Ionolux (IO) were tested under two pre-storage conditions: with desiccation and without desiccation. Disc-shaped samples (n = 5) were immersed in water and weighed at different time intervals (1 h, 24 h, 3 d, 7 d and 30 d) and hygroscopic expansion was recorded at 7 d and 30 d. Data were analysed using Factorial repeated measures ANOVA, one-way/two-way ANOVA, Independent samples t-test and Tukey's post hoc test ($\alpha = 0.05$). **Results.** With desiccation, sorption of IO and FJ was 124.33 $\mu\text{g}/\text{mm}^3$ and 79.97 $\mu\text{g}/\text{mm}^3$ respectively. Solubility was $-12.36 \mu\text{g}/\text{mm}^3$ for IO and $-20.19 \mu\text{g}/\text{mm}^3$ for FJ. Hygroscopic expansion was 3.01% for IO and -2.35% for FJ.

Without desiccation, sorption was in the order: IO \square BD \square FJ (130.35 $\mu\text{g}/\text{mm}^3$, 122.07 $\mu\text{g}/\text{mm}^3$, and 107.21 $\mu\text{g}/\text{mm}^3$ respectively), while solubility order was: BD \square FJ \square IO (154.83 $\mu\text{g}/\text{mm}^3$, 88.82 $\mu\text{g}/\text{mm}^3$, and 25.67 $\mu\text{g}/\text{mm}^3$ respectively). IO and FJ showed significant difference in sorption and solubility between the two pre-storage treatment groups ($p < 0.005$). Hygroscopic expansion was in the order: IO \square BD \square FJ.

Significance. BD had the highest solubility while IO had the least. The relatively stable polymeric resin in IO may contribute to its low solubility but high hygroscopic expansion. Desiccation had significant effect on sorption, solubility and volumetric expansion of water-based materials.

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* Corresponding author at: School of Medical Sciences, The University of Manchester, Manchester, M13 9PL, UK.

E-mail address: nick.silikas@manchester.ac.uk (N. Silikas).

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1. Introduction

Replacing lost dentine has been a major problem in clinical dentistry for several years [1]. By the end of the 1960s, Wilson and Kent introduced glass ionomer cement (GIC) which is also called glass polyalkenoate cements [2,3]. GIC have good ion migration and particle adaptation which result in an interactive zone formation and chemical bond between GIC and the tooth surface [4]. Lamination or sandwich technique using conventional glass ionomer has been introduced as a dentin replacement method [5].

With further GIC development, the resin modified GIC (RMGIC) has been marketed by the late 1980s and it has been used in the sandwich technique as a dentine replacement material [6]. Adding the hydrophilic hydroxyethyl methacrylate monomers (HEMA) to GIC results in improved chemical and mechanical properties [7,8]. A number of other materials have been suggested to be used as dentine replacement materials including bioglasses [9] and the new generation flowable bulk-fill resin-composites such as Smart Dentine Replacement (SDR) [10].

Part of this evolution process is Biodentine™ (BD), which is new water calcium-based cement, that has been developed in 2010 and offers a bioactive, biocompatible and biointeractive dentine replacement material [11]. The powder contains tricalcium and dicalcium silicate ($3\text{CaO}\cdot\text{SiO}_2$ and $2\text{CaO}\cdot\text{SiO}_2$) as a core material. Calcium carbonate (CaCO_3) makes up approximately 15% of the material (according to the manufacturer) [12,13] and it performs as a nucleation site and a filler [14]. Zirconium dioxide (ZrO_2), which makes approximately 5% of Biodentine, acts as a radiopacifier material [15–18].

Although the practice of dentine replacement using the 'liner' or 'base' approach may have negative tendency to affect the restoration longevity as has been shown by a recent systematic review [19], it may be legitimate to apply BD as a dentine replacement material compared to other materials mainly due to its effect on preserving the pulp vitality through its bioactive therapeutic effect. Furthermore, it has an antibacterial effect that makes it suitable for deep caries cavities [20] as well as an excellent bond and seal with dentine, short setting time, and good mechanical properties [21]. On the other hand, it does not have a satisfactory wear resistance, mechanical strength and aesthetic to be a definitive restoration. Therefore, BD should be covered by a resin-composite layer similar to GIC (sandwich technique) [1]. Since BD is relatively weak during its initial setting period, coverage by a resin-composite should be delayed for at least fourteen days since its first application as recommended by the recent study. This should provide adequate time for BD to mature and withstand the polymerisation contraction force of the resin-composite [22]. Thus, during the first two weeks of its application, BD is left directly exposed to oral fluids which might negatively affect the material through water sorption and solubility, especially that water sorption has been shown to be the fastest during the first 2–3 weeks before reaching the saturation phase [23].

The lifespan of a restorative material in the aggressive environment of the oral cavity is dependent on a number of clinical factors and material's properties including its resistance to

water absorption, solubility, and volumetric expansion. The hydrolytic stability of a restorative material affects its durability and the treatment success [24]. In addition, solubility and water sorption may contribute to microleakage [25] which could indirectly result in restoration failure [26]. Accordingly, it is desirable to have water sorption and solubility as low as possible [24].

Currently, there is no a specific standard for testing water sorption, solubility, and volumetric expansion of water-based materials. A number of studies aimed to mimic the *in vivo* situation by immersing the samples directly in water without desiccation [27]. However, in other studies the ISO 4049 (2009) standard for assessing polymeric materials have been followed to assess materials such as GIC [28,29] which involves desiccating the test materials in order to reach an initial stable mass after removing the free water molecules from the materials. 3D hygroscopic expansion is one of the recognised techniques for dimensional changes of materials in water [30]. In the current study, both techniques were used to assess the effect of desiccation on different properties of water-based materials. The aims of this study were to assess water sorption, solubility and volumetric expansion of water-based dentine replacement materials and the effect of pre-immersion desiccation on these measurements. The null hypotheses tested were: (i) there is no difference in water sorption, solubility and 3-D hygroscopic expansion of different materials, and (ii) there is no effect of pre-storage desiccation on water sorption, solubility and volumetric expansion, of tested materials.

2. Materials and methods

The materials investigated in this study (Table 1) were Biodentine™ (BD), GC Fuji IX GP® FAST (FJ), and Ionolux (IO) which respectively represent a calcium silicate-based material, a conventional glass ionomer, and a resin modified glass ionomer.

Disc-shaped samples of each material were prepared using PTFE moulds of 15.0 mm (± 0.1 mm) internal diameter and 2 mm (± 0.1 mm) thickness. The mould was set on a glass microscopic slide with a transparent film. The mould was slightly overfilled with the material and another film and glass slide were applied on the top with pressure. Clamps were used to maintain the pressure during materials setting.

According to ISO 6876, BD was placed in the moulds and allowed to set at 37 °C and 99% relative humidity for 9 min (70% of the final setting time). FJ moulds were also set for 15 min at 37 ± 1 °C and a relative humidity value of $95 \pm 5\%$. IO specimens were irradiated at five overlapping points on each side for 20 s using an Optilux light curing unit (Optilux 501, USA) with an irradiance of 620 mW/cm². The tip of the light curing unit was approximately 1 mm away from the specimen surface. Each sample was checked for voids and irregularity and any excess flash was removed using 1000 grit silicon carbide abrasive paper. A digital electronic calliper (Mitutoyo Corporation, Japan) was used to measure the specimen thickness and diameter at 4 and 3 points respectively. The mean values were used to calculate the specimen volume (v). Each sample was then placed in a labelled glass bottle.

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