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Fluoride containing bioactive glass composite for orthodontic adhesives – ion release properties

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ABSTRACT

Objective. Dental materials that release calcium, phosphate and fluoride ions could prevent demineralisation and/or enhance remineralisation of enamel. The objective was to develop a novel bioactive glass (BAG) resin and investigate pH changes and ion release in 3 immersion media.

Methods. Quench melt derived BAG (35.25% SiO₂, 6% Na₂O, 43% CaO, 5.75% P₂O₅, and 10% CaF₂) was incorporated into a resin (42.25% BisEMA, 55% TEGDMA, 0.25% DMAEM, 0.5% camphorquinone and 2% 4-Meta), with a filler load of 80% by weight. Ninety composite disks for each BAG loading of 80%, 60%, 50%, 40%, 20%, and 0% were made and each disk was immersed in 10 ml of either tris buffer (TB), or artificial saliva at pH=7 (AS7) or pH=4 (AS4), n=30 for each solution. Three disks of each loading were taken from each of the solutions, at ten time points (6 h–6 months), for measurement of pH, fluoride, calcium and phosphate.

Results. The BAG adhesive raised the pH in all the solutions, release Ca, PO₄ and F ions especially in AS4. The rise in pH and the release of Ca and F are directly related to the BAG loading and the time of immersion. The pH and the ion releases were maintained and continued over 6 months.

Significance. Unlike glass ionomer resins, favourable ions F, Ca and PO₄ releases were maintained over a long time period especially in acidic condition for this novel BAG-resin composite. This indicates the resin has the potential to prevent formation and progression of early caries lesions.

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1. Introduction

Bioactive glass (BAG) is a material that degrades and dissolves upon contact with a physiological fluid to allow controlled release of therapeutic ions and the formation of an apatite like surface layer [1,2]. It was first developed by Hench in 1969 [3] and was called Bioglass® (BAG 45S5), which has been in clinical use since 1985. The BAG 45S5 is composed of 46.1

SiO₂, 2.6 P₂O₅, 24.4 Na₂O and 26.9 CaO (mol%). The composition of BAGs strongly influences their dissolution kinetics and their ability to form apatite (bioactivity), whereby the concentration of the glass formers (silica and phosphate) and the degree of polymerisation (network connectivity) are the main determinants of its bioactivity [4]. The effect of phosphate is attributed to the formation of orthophosphate phase which is more reactive than the silica phase, therefore, an increase in phosphate content allows for a faster apatite deposition and

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thereby enhances bioactivity [5–7]. It was found that the network connectivity (NC) remains constant when the addition of P_2O_5 is accompanied with the addition of network modifiers to charge balance the orthophosphate in the glass. Furthermore, increasing the P_2O_5 content reduces the rise in pH upon dissolution of bioactive glasses, which is beneficial since optimal activity and apatite formation is around a pH of 7.3. Hence, combining the basicity from the silicate of the bioactive glass with the acidity of the orthophosphate seems to have a good effect in maintaining the pH around neutral [8]. The effect of fluoride in BAGs is to allow for fluorapatite (FAP) formation, which in turn is more resistant to acid dissolution than hydroxyapatite [9–12].

The incorporation of BAG into the resin based composites is a relatively new approach in dentistry [13,14]. Partial or full replacement of the inert glass filler particles in composite resins with BAG particles were implemented to develop a composite resin that can release therapeutic ions. Pit and fissure sealants containing BAG 45S5 fillers were studied by Yang et al. and found to have a neutralizing effect in acidic environments [14]. These authors also reported similar neutralising effect with three other different BAGs [15]. However, these studies only contained reports on the impact of pH only, with no data on ion release from the BAGs. Incorporating a sol gel BAG in an orthodontic adhesive was shown to result in significantly higher calcium release and buffering ability, with a potential in prevention of white spot lesions (WSL) [16–18]. However, these studies found limited fluoride release, which is probably attributed to the potential loss of fluorine during the sol-gel glass production route.

Most of the above mentioned studies used the BAG 45S5 or one of its modified versions where the sodium contents was around 20 (mol%). Although sodium is theoretically important for the BAG reaction and dissolution [2], high sodium concentration could lead to excessive water exchange between the glass particles and the media, which in turn might result in swelling of the glass particles and consequent adverse effects on the physical properties, survival and longevity of the composite resin. Decreasing sodium concentration in BAG would enable incorporating more advantageous elements like calcium, phosphate and fluorides which are essential for apatite formation.

No studies were found on the impact of the different BAG filler loadings (especially for those manufactured by the melt quench route) within the resin in ion releases and their potential neutralising effect. Furthermore, apart from deionised water, the effect of immersion on other media was not known. Hence, the aim of the present study was to incorporate a novel BAG (by the melt quench route) with low sodium but high fluoride, phosphate and calcium contents, into a specially designed resin and to characterise the behaviour of the resin with different BAG loadings in three immersion media.

2. Materials and methods

2.1. Synthesis of BAG

The strategy of BAG design is to decrease the sodium with an increase in calcium and fluoride content in order to allow for

Table 1 – (a) BAG batch composition in mol%. (b) BAG composition in mol%, determined by X-ray fluorescence spectrometry (XRF).

BAG	SiO ₂	CaO	Na ₂ O	P ₂ O ₅	CaF ₂
(a)	35.25	43.00	6.00	5.75	10.00
(b)	35.30	44.41	5.76	5.52	8.67

more beneficial therapeutic ion release. Phosphate concentration was also increased, since Eden has stated that “bioactivity increases monotonically with increasing phosphorus content of the BAG” [7]. Therefore, 5 glasses with variable concentrations of phosphate and fluoride were manufactured and tested for the most appropriate BAG as the replacement fillers in the resin. The glasses were characterised using X-ray diffraction (XRD) and Fourier transform infrared spectroscopy (FTIR) followed by ion release tests after immersion in tris buffer solution (TB). The test revealed that the BAG with the composition listed in Table 1(a) was the most suitable in terms of bioactivity. The NC of this BAG calculated according to Hill and Brauer [4] was 2.19.

The BAG was manufactured via the melt quench route. A 200 g batch of analytical grade SiO₂ (Prince Minerals Ltd., Stoke-on-Trent, UK), Na₂CO₃, CaCO₃, P₂O₅ and CaF₂ (all Sigma-Aldrich, Gillingham, UK) were mixed and transferred into a platinum-rhodium (80/20) crucible for melting in an electric furnace (EHF 17/3, Lenton, Hope Valley, UK) at 1480 °C for 1 h. The melted glass was then quenched quickly in distilled water. The glass frit was milled in a gyro-mil (Gyro mill, Glen Creston, London, UK) and sieved using a 38 μm sieve (Endecotts Ltd., London, UK). The average particle size for the BAG glass powder was approximately 10 μm. To ensure that the experimental composition is similar to the theoretical composition, which is particularly important for the F-containing BAG as some F may be lost on melting, the BAG powder (20 g of coarse powder, >38 μm in size) was sent to a commercial laboratory (Lucideon limited, Queens Road, Penkhull, Stoke On Trent, United Kingdom) for chemical analysis using X-ray fluorescence spectrometry (XRF). The results (Table 1(b)) show that the BAG composition was largely as expected, based on its nominal composition. The decrease of CaF₂ and the corresponding increase in CaO indicated that there was a slight loss of F in the melting process, as shown by Brauer et al. [19].

2.2. Preparation of the resin

The resin was prepared using 42.25% bisphenol A ethoxylate dimethacrylate (BisEMA), 55% tri(ethylene glycol) dimethacrylate (TEGDMA), 0.25% 2-(dimethylamino)ethyl methacrylate (DMAEM) and 0.5% camphorquinone (all Sigma-Aldrich Gillingham, UK). 2% 4-Meta (First Scientific GmbH, Elmshorn, Germany) was added to enhance the bond with the teeth and the brackets.

2.3. Preparation of adhesive paste and disks

The adhesive paste was prepared using a glass to resin ratio of 80:20% by weight. The BAG powder load was replaced incrementally by a highly inert glass (CDL Ltd., England) that

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