Development and Assessment of New Bioactive Glass Fiber Post. Part II: Ion's Release

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Abstract

Objectives: This study was designed to develop and assess the ions release property of an innovative experimental bioactive fiber-reinforce composite as a new material for an intra-canal post.

Study Design: An in vitro study

Materials and Methods: Surface treated unidirectional E-glass fiber were impregnated and implanted with a triple cure, self-adhesive ACTIVA BioACTIVE resin cement to fabricate an experimental bioactive glass fiber post using hand lay-up moulding technique and polytetrafluoroethylene (PTFE) moulds, The cylindrical and rectangular form specimens were prepared, for each specimen, ensure 3min for self-cure setting, then light-cured for the 40s. The specimens were individually immersed in a deionized (DI) water, and sodium chloride solution to assess the fluoride ion release, and calcium-phosphate ions release sequentially. Virgin ACTIVA BioACTIVE cement was used as a reference group for all investigations.

Statistical analysis used: Statistical analysis was achieved by means of independent variable t-test, one-way ANOVA, and Bonferroni Pairwise comparisons utilizing IBM-SPSS software.

Results: Experimental material recognized a significant reduction in $F^{-,}$ $Ca^{+2,}$ and PO_4^{3-} release after reinforcement compared to virgin ACTIVA BioACTIVE.

Conclusions: The ACTIVA BioACTIVE cement's reinforcement process reduced the ion release intensity of the material but not affected the releasing pattern.

Keywords*: Dental fiber post, Fiber-reinforced composite, Ions release.*

Introduction

The rising popularity and demand for aesthetic fiber posts are predictably shifting the endodontically treated tooth rebuilding to a more uncomplicated technique. A similar elastic modulus of fiber post to dentine is considered the main advantage by producing a distribution of stress close to normal teeth [1]. Other advantages include better biocompatibility, aesthetic, and corrosion resistance, which gives the fiber post its priority [2].

Compared to traditional metallic cast posts, glass fiber posts minimize the risk of irreversible root fractures. [3]. Laboratory studies have confirmed the desirable mechanical and physical properties of fibre posts, and a broad variety of failure mechanisms have been documented clinically in the literature.

Barfeie et al. (2015) reported adhesive failure in 16 of the 19 clinical trials, which was the most common cause of failure [4]. Failure was attributed to the components from which the post-core system is constructed, notably the resin matrix. The results of different studies show that the lower elastic modulus reduces the cause of failure of the root fracture but increases the debonding of the glass fiber post, which is the most typical post-failure mode [5].

The vulnerability of glass fiber posts to chemical, mechanical and thermal fatigue leads to an irreversible decline of mechanical properties, enhancing the likelihood of debonding. In a wet environment, the polymer matrix, glass fibers, and the interface between them will deteriorate [6].

Tooth remineralization can be assisted by a restoration that releases ions into the mineral content of the tooth structure surrounding it [7]. Fluoride supports remineralization by transporting calcium and phosphate ions together and incorporating them into the remineralized surface [8], as calcium is an element of the mineral content of the dental system. Numerous new dental restorative materials have been developed with the release of calcium.

ACTIVA BioACTIVE is a modern bioactive, flowable, resin-based composite comparable to RMGICs, present as base/liner, restorative, cement. ACTIVA BioACTIVE contains a high molecular weight polyacrylic acid, Urethane dimethacrylate monomers, dimethacrylate phosphate (acids), Fluoro-alumino-silicate glass, silanated nonreactive fillers, water, initiators (chemical and light). Such composition permits the release of fluoride, calcium, phosphate. Also enhance protects against microleakage, and antibacterial properties. [9, 10].

Saunders et al. (2018) found that ACTIVA BioACTIVE cement remineralized the enamel near to orthodontic brackets compared to the non-fluoride release cement [11].

Cross-linked ones (thermoset) matrix, such as epoxy resin and bisphenol A- urethane di-methacrylate (UDMA), are the most used matrices in fiber-reinforced composite posts. Even though cross-linked network thermosetting polymers are better in mechanical properties, thermal stability, durability, and chemical resistance than thermoplastics (linear), They exhibit insufficient surface adhesive properties [12].

Therefore, substituting the resin matrixes of commercial glass fiber posts by bioactive resin cement (ACTIVA BioACTIVE) with ionic resilient resin and ion release property may enhance adhesion durability employing ions release and reduce microleakage.

This study aimed to create a bioactive fiber post by reinforcing the ACTIVA BioACTIVE cement with E-glass fibers and testing the ion release property of the new fiber post material.

Materials and Methods

Preparation of the experimental specimens

The fabrication procedure started with the surface treatment for the E-glass fiber (Mayitr company, China). Unidirectional E-glass fibres bundles with 8-15μm filament diameter and 2,58 g/cm3 density which their chemical composition mention in (Table1) was treated by 99.9% acetic acid (Chem-Lab NV, Belgium) for 2h at 55 °C, washed three times with purified water and dried in the incubator for 8 h at 50 ° C. A part of the acetic acid-treated glass fiber was further exposed to wet salination by means of 2% of 3-methacryloxyproyltrimethoxysilane silane coupling agent A-174 (Sigma-Aldrich, Germany) in an ethanol-water mixture (90/10 w/w) acidified to pH 3.8 and maintained at this level with glacial acetic acid. The glass fiber was processed for 1 h in this solution and then dried for two h at 110 ° C before usage, as in previous studies [13, 14].

The fiber's surface texture and morphology were examined by scanning electron microscopy (TESCAN VEGA III, TESCAN, Czech Republic) with an accelerating voltage of 5KeV as well as a magnification of (1Kx, 2Kx, and 5Kx). Untreated glass fiber was used as a reference.

For the ion release investigation, a cylindrical and rectangular shape specimen (n=8) were prepared by the incorporation of one bundle of treated glass fiber consisting of sub-bundles of 1000 filaments with a constant amount of ACTIVA BioACTIVE cement (Pulpdent Corporation, Watertown, USA.) Using PTFE mould with a cylindrical and rectangular shaped designed according to previous study [15].

The fiber value in percentage volume was determined using the formula. [13, 14].

$$Vk(\%) = \frac{Ds-Dr}{Df-Dr} \, x \, 100 \quad \text{(Eq 1)}$$

where Vk is the vol.% of the GF, Dr is the density of resin matrix, Df is the GF density (2,58 g/cm3), and Ds is the density of the FRC sample.

The fiber content in percentage volume for each sample was 40%.

The impregnation procedure of the fiber to resin cement base on hand lay-up moulding technique was the first step in fabrication [13], in which the PTFE mould fixed to a glass slide and Mylar strip, then the resin cement and the treated fiber added and painting layer by layer until the mould filling.

Table 1, Composition of the materials used for this study

Material	Manufacture	Composition	Batch
E-glass fibers bundles	Mayitr company, China	55%SiO2, 11%Al2O3, 18%CaO, 6%B2O3, 5%MgO, 5%Other	13723
ACTIVA BioACTIVE Cement	Pulpdent Corporation, Watertown, MA USA	Blend of diurethane and other methacrylates with modified polyacrylic acid. 47%wt Bioactive Glass with Sodium Fluoride	180129

Later, another mylar strip and glass slide were positioned over the mould and pressed with (1 Kg) weight to level up the specimen surface and prevents the development of bubbles and an oxygen-inhibited layer. Each specimen waiting for 3 minutes to ensure self-cure setting, then cured by visible light for the 40s from each side with Paradigm™ DeepCure L.E.D. (3m, USA.).

Each specimen was polished with silicon carbide paper (grit 800 and 1200). Sixteen specimens of virgin ACTIVA BioACTIVE without glass fiber were prepared as reference samples.

The fluoride ion release assessment

Sixteen disc-shaped specimens (8 mm x 3 mm, 175.85 mm overall surface area) were fabricated from experimental and virgin ACTIVA. Each sample was soaked in polypropylene vials comprising 4 mL (37°C ± 1°C) of deionized water. [10, 16]. Fluoride release was measured at four-time intervals (1, 7.14, 28 days). For each time point, the specimens were separated from polypropylene vials approximately 30 min before each read-out to yield similar temperature conditions for the deionized water that used to measure the quantity of released fluoride ions (ppm) by ion chromatography (88 Compact Ic pro, Metrohm, Herisau, Switzerlan).

The specimens removed from the solution were rinsed with 1 mL deionized water. To eliminate excess water, blotting paper was used, then again placed in a polypropylene vial tube comprising 4 mL deionized water and placed in the incubator to the next evaluation period [10, 16].

Calcium and phosphate ions release assessment

Sixteen rectangular bar-shaped (2 mm×2 mm×12 mm) specimens were also fabricated of the experimental and virgin material. Sodium chloride solution (133 mmol/L) was formulated and buffered to pH values: pH 4 with 50 mmol/L lactic acid [17]. Every three specimens were soaked in 50 mL of the solution, yielding 2.9 mm^3/mL of sample volume/solution.

Calcium and phosphate ions release was also measured at four-time interval (1, 7, 14, and 28 days); aliquots of 0.5 mL of sodium chloride solution were removed and substituted by a fresh solution. The concentrations of Ca^{2+} and Po_4^{3-} ions were measured in ppm at each time point using inductively coupled plasma-optical emissions spectroscopy. (ICP-OES) (Shimadzu ICPE-9000 SHIMADZU Corporation, Japan) using commonly known standards [17].

Result

Fluoride ion release

The outcome of this study revealed that the virgin material (ACTIVA BioACTIVE) generally, recorded a higher mean value of fluoride ion release at all time points as compared to experimental material. In addition, ion release deceased with the time for both materials, reaching the lowest value after 28-day, with statistically highly significant differences as shown by the student's t-test, Table (2).

Table (2): Student's t-test for comparison of the significance of fluoride ion release between two groups at different points time.

Time	T	df	P.value
1-day	-21.247	14	.000HS
7-days	-5.354	14	.000HS
14-days	-4.073	14	.001HS
28-days	-51.815	14	.000HS

Calcium and phosphate ions release

The highest cumulative calcium ion release was scored after one week of immersion of both materials, with a value of (9.52±0.06) ppm for the experimental material and (9.94±0.01) ppm for the virgin one. Also, calcium ion release gradually decreased with time.

The comparison outcome displayed highly significant differences between the two materials in 7-days and 14-days' time points, Table (3).

Regarding phosphate (PO_4^{3-}) ions, it is clear that two materials release the highest cumulative phosphate ion with the first week about (464.5±9.6) and (491.87±3.2) ppm for experimental and virgin materials consecutively, then ion release was reduced with time (Fig.1).

Table (3): Student's t-test for comparison of the significance of calcium ion release between two groups at different points time

Time	T	df	P.value
1-day	-1.812	14	0.091NS
7-days	-17.277	14	0.000HS
14-days	-8.910	14	0.000HS
28-days	-1.419	14	0.178NS

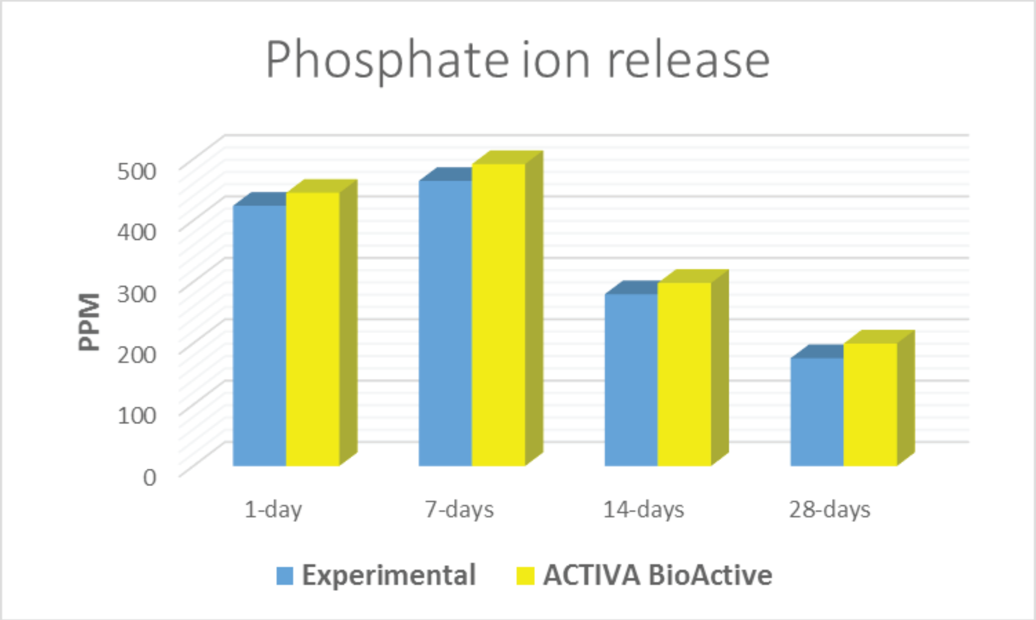

Figure (1): Mean of phosphate ion release in (ppm) for the two groups within each period.

Discussion

Microleakage and secondary caries are known to be the incredibly significant cause for failure of the direct and indirect dental restorations, including the fiber post [9]. The cariostatic properties of the restorative products are related to the volume of fluoride emitted and incorporated into the neighbouring tooth structure [18]. In order to allow the best of these advantages, the ions should be placed as near as possible to the surface of the tooth.

In addition, the rate of mineral reduction may be twice as fast from the root dentine as from the enamel, so fluoride-releasing bioactive cement may be useful in avoiding or reducing secondary caries along the edges of dental restoration and the fabrication of the experimental fiber post with such property may accomplish this critical purpose. Consequently, A substrate that may produce both fluoride and calcium ions will be required to improve the production of fluorapatite on the surface of the tooth [9, 18].

Specimens were soaked in distilled water to act as a baseline for fluoride release in an unstimulated environment, no organic molecules or minerals that might affect the results, and more fluoride is released in water than in synthetic saliva [10, 18, 19]. Ion chromatography was used to evaluate the fluoride release owing to its sensitivity to measure accurately even the low concentration of fluoride [19, 20].

The results of this research demonstrated a highly statistically significant difference between the virgin and experimental material, and such findings may be related to the amount of fluoride content that is already reduced after the reinforcing of with 40% of glass fibers [21].

Both materials are showed a similar pattern of fluoride release; on the 1st day period assessment, major fluoride ion release was detected. This can be clarified by the accelerated elution of fluoride produced due to the acid-base reaction, which takes place on the glass particle surface, as one of the hardening mechanisms of this cement is the acid-base reaction. This was agreed with many in vitro experiments that also demonstrated a higher release of fluoride in the first two days [10, 18, 22].

This elevated level of fluoride emitted on the first day is called “Initial Burst Effect”, As the release of fluoride depends on its concentration and diffusion restriction in the matrix and the particles, and the surface was rinsing effect. In the earlier point, a significant volume of fluoride has been part of the reaction matrix.

Then, fluoride spreads promptly from the exposed matrix on the surface of the specimens during the early acid breakdown of powder element surfaces for short-term release and is periodically replaced by fluoride disperse from the matrix below the surface showing slower or prolonged release. [21]. A reduction in the release of fluoride over the next days can be due to the slower dissolution of glass particles through the pores of the bioactive composite over time; Another explanation might be the hydrophobic nature of embrace resin in the virgin and experimental materials that might reduce water diffusion and the following discharge of fluoride from the substance [18].

The amount and the pattern of fluoride release from ACTIVA BioACTIVE in present study are comparable to that result of other studies [10, 18, 19, 23]; in fact, most of the polymer resins have developed very low fluoride ions (within 30–60 days, less than 0.02–2 ppm) [23], which has been supported by our result.

A study by Claussen et al. (2017) compared ACTIVA cement with well-established self-adhesive resin cement RelyX Unicem (RU) regarding ion release. The first demonstrated higher fluoride release, while the second released a higher amount of calcium [24]. Anyway, There is also a promising study by Huang et al. (2020) reported that both bioactive cement ACTIVA and RelyX Unicem revealed netting areas of demineralization inhibition when evaluated regarding the prevention of root dentin demineralization; they propose that bioactive cement could be recommended for patients at risk of secondary caries along the crown margins [9].

Calcium and phosphate ions release material could encourage apatite precipitation and provide a tooth seal to avoid marginal gaps [9, 25]. Consequently, the use of ion releasing bioactive posts may improve the sealing and adhesion of the post to the dentine structure and reduce the occurrence of secondary caries.

Our findings revealed that there was a statistically significant difference regarding Ca^{+2}, PO_4^{3-} ions release between the virgin ACTIVA and experimental materials. Such findings may be related to the number of ions comprise after reinforcement modification of cement composition.

The findings of experimental and virgin ACTIVA showed an increase in the Ca^{+2-} release with time to reach the highest value of ion release after one week, and the differences were statistically highly significant. These observations might indicate that there was a release of calcium from the bulk of ACTIVA containing the sample to the immersed fluid, as the matrix comprises calcium alumino fluorosilicate glass, to deliver calcium and fluoride ions, then the concentration of released ions decreased significantly to get the lowest value after 28 days.

Such reduction can be explained by the fact that many Ca^{+2} ions are dissolved from glass particles in the solution, and the frequency of the attraction of Ca^{+2} ions in the surface of the samples is more than that released to the solution. Another expected explanation may be the presence of an acid phosphate group within the resin that has Ca^{2+} binding affinity [26].

Regarding the PO_4^{3-} ion, there is a highly significant change in the concentration of release, especially after one week of immersion in sodium chloride solution. This due to the release of PO_4^{3-} from ACTIVA containing specimens, as the ionic resin of ACTIVA portion consists of phosphate acid groups, which strengthen the interaction with the resins and extend the bonding with the tooth structure. Through ionization, which is water-dependent, hydrogen ions break out of the phosphate groups and are replaced by calcium in the tooth structure. This ion interface binds the resin to the minerals in the tooth, providing a strong resin-hydroxyapatite complex and a supporting microleakage seal [27].

In addition, various acid monomers have applied different efficiencies to boost the release of Ca^{2+} and PO_4^{3} ions; remember that the Ca^{2+} ions are expected to disperse and transport more readily across the resin than the phosphate ions [17].

Another critical factor that may affect the ion release is the pH. According to the manufacturer, ACTIVA responds to pH cycles in the mouth, so the specimens were immersed in solution with a pH4 in the current study as an accelerated experiment; this may explain the releases of more phosphate especially with the first week of immersion.

During the later immersion period, the concentration of phosphorous in sodium chloride solution slowly decreased, and these changes were due to the formation of calcium phosphate layers [17, 28, 29].

One of the limitations of this experiment was it's not considered the ions recharging capacity of the experimental material.

Conclusions

With the limitations of this study, the reinforcement of the ACTIVA BioACTIVE cement with E-glass fibers reduced the ion release intensity of the material but not the releasing pattern.

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