journal of dentistry 41 (2013) 449–454

Available online at www.sciencedirect.com

SciVerse ScienceDirect

journal homepage: www.intl.elsevierhealth.com/journals/jden

Ion release and physical properties of CPP–ACP modified GIC in acid solutions

I. Zalizniak [a], J.E.A. Palamara [a], R.H.K. Wong [a], N.J. Cochrane [a], M.F. Burrow [b], E.C. Reynolds [a,*]

[a] Oral Health CRC, Melbourne Dental School, The University of Melbourne, Australia
[b] Faculty of Dentistry, University of Hong Kong, Hong Kong

ARTICLE INFO

Article history:
Received 25 October 2012
Received in revised form
7 February 2013
Accepted 11 February 2013

Keywords:
GIC
CPP–ACP
Hardness
Ion release

ABSTRACT

A new glass-ionomer cement (GIC) (Fuji VII^{TM} EP) includes 3% (w/w) casein phosphopeptide–amorphous calcium phosphate (CPP–ACP) to enhance ion release.

Objectives: To assess this new GIC compared with a GIC without CPP–ACP (Fuji VII^{TM}) with respect to ion release, changes in surface hardness and in mass under a variety of acidic and neutral conditions.

Methods: Eighty blocks of Fuji VII^{TM} (F7) and Fuji VII^{TM} EP (F7EP) were subjected to three acidic solutions (lactic and citric acids pH 5.0, hydrochloric acid pH 2.0) and water (pH 6.9) over a three-day period. Ion release, surface hardness and weight measurements were carried out every 24 h.

Results: Higher calcium ion release from F7EP was observed under all acidic conditions. Increased inorganic phosphate ion release was observed for F7EP in hydrochloric and citric acids. Fluoride ion release was similar between F7 and F7EP under all conditions but was significantly higher in acids compared with water. After three days there was no significant difference in surface hardness ($p > 0.05$) between the two materials under all conditions except hydrochloric acid. Minimal change in mass was observed for F7 and F7EP in water, lactic and hydrochloric acids, however citric acid caused significantly more mass loss compared with water ($p < 0.001$).

Conclusion: Incorporation of 3% (w/w) CPP–ACP into F7 enhanced calcium and phosphate ion release, with no significant change in fluoride ion release and no adverse effects on surface hardness or change in mass.

Clinical significance statement: GICs have the potential to release fluoride ions particularly under acidic conditions associated with dental caries and erosion. A new GIC containing CPP–ACP and fluoride releases not only fluoride ions but also calcium and phosphate ions under acidic conditions which should help to inhibit demineralisation associated with caries and erosion.

1. Introduction

Glass ionomer cements (GICs) are widely used for a variety of purposes such as intermediate restorations, caries stabilisation, definitive restoration of micro-cavities or non-carious cervical lesions, adhering orthodontic brackets and bands, fissure sealing erupting molars and as a surface protecting material for high risk surfaces such as root surfaces.[1,2] The main advantages of GICs are their strong

* *Corresponding author at*: Melbourne Dental School, The University of Melbourne, 720 Swanston Street, Victoria 3010, Australia. Tel.: +61 3 9351 1547; fax: +61 3 9341 1599.
E-mail addresses: e.reynolds@unimelb.edu.au, k.fletcher@unimelb.edu.au (E.C. Reynolds).
0300-5712/$ – see front matter
http://dx.doi.org/10.1016/j.jdent.2013.02.003

ability to chemically bind to dentine and their ability to release fluoride ions. This fluoride ion release has been shown to slow the progression and aid the regression of early carious lesions.[3] Tooth enamel will only demineralise when the fluid bathing the enamel crystals is undersaturated with respect to the enamel mineral (carbonated hydroxyapatite). Hence, calcium and phosphate ion activity is an important component influencing the level of enamel mineral saturation.

A number of investigators have explored the modification of dental materials in attempt to have them release calcium, phosphate and fluoride ions.[4–6] Skrtic et al.[6] explored modifying dental composites with bioactive glasses. Mazzaoui et al.[5] and Al Zraikat et al.[4] assessed the addition of casein phosphopeptide–amorphous calcium phosphate (CPP–ACP) to GICs. The casein phosphopeptides stabilise calcium and phosphate ions in a bioavailable form that allow them to inhibit demineralisation and promote the remineralisation of early lesions.[7] CPP–ACP is stable in the presence of fluoride and has been shown to work synergistically with fluoride.[8] This makes CPP–ACP a promising additive to dental products and restorative materials. The study by Mazzaoui et al.[5] was a proof of concept study that showed that CPP–ACP could be added to GIC. Al Zraikat et al.[4] later explored the effect of CPP–ACP concentration on GIC mechanical properties. Additionally, both of these of studies showed that the addition of CPP–ACP improved calcium and phosphate ion release in lactic acid.

The tooth surface can be exposed to a variety of demineralisation challenges including: acids formed from metabolic processes of oral bacteria (predominantly lactic acid); food acids such as citric and phosphoric acid that are commonly found in soft drinks[9]; and hydrochloric acid from the regurgitation of stomach contents. If these acids overwhelm saliva's protective functions mineral may be lost from the teeth.[10] Therefore, the overall aim of this study was to determine how GIC with CPP–ACP performed under a variety of acidic conditions compared with GIC without CPP–ACP in terms of ion release, change in hardness and change in mass. Fuji VII (F7) is a low viscosity strontium glass based ionomer cement and is commonly used as a surface protectant, providing effective wetting and intimate adhesion to tooth surfaces, as well as enhanced remineralisation capabilities. It has higher fluoride release than most other GICs which makes it a good candidate to establish if there is an additional benefit through the incorporation of CPP–ACP as a source of calcium and phosphate ions together with the fluoride ions. This study will establish whether F7 plus CPP–ACP has the potential to further protect surrounding hard tissue and enhance the remineralisation of demineralised tissue by additional ion release.

The research questions were: what effect on the GIC would the addition of CPP–ACP have on; (1) surface hardness; (2) change in mass under neutral or acidic environments and (3) calcium, phosphate and fluoride ion release under neutral or different acidic environments.

2. Materials and methods

Fuji VII GIC (F7) and F7 with added 3% (w/w) CPP–ACP (F7EP) from the same batch were provided by GC Corporation (Japan) in capsule form. Polyvinyl siloxane impression material (eliteHD + light body, Zhermack SpA, Badia Polesine, Italy) moulds were used to create standardised GIC blocks measuring 3 mm × 6 mm × 6 mm (thickness × width × length). 40 blocks of each GIC were prepared by placing the materials in the mould with the top and bottom surfaces covered by plastic strips, which was held between two glass slides. The glass slides were gently pressed together to extrude any excess material. The specimens were allowed to set inside the moulds for 24 h in an incubator (37 °C, ~100% relative humidity). After cooling to room temperature the blocks were removed from the moulds, and the two major parallel surfaces of the blocks were lapped with 600 grit paper (Norton Tufbak, Saint-Gobain Abrasives Ltd., Auckland, NZ).

Four different solutions were prepared to expose the blocks to a variety of acidic and neutral environments. The three acidic solutions were formulated to simulate a gastric erosive challenge (50 mM NaCl adjusted to pH 2.0 with HCl), a dietary erosive challenge (50 mM citric acid at pH 5.0) and a cariogenic acid challenge (50 mM lactic acid at pH 5.0) (this concentration was selected based on values found previously in plaque fluid).[11] Ionic strength of all the acidic solutions was made up to 50 mM including the HCl solution which was modified with NaCl to approximate that found in stomach acid.[12]

The neutral solution was distilled deionised water at pH 6.9 (Millipore Corporation, Victoria, Australia).

Ten blocks of each type of GIC were exposed to 5 mL of one of the four solutions. Solutions were changed every 24 h and the samples were measured for change in mass, surface hardness and the solutions were analysed to determine ion release of calcium, phosphate and fluoride every 24 h over three days.

The mass of each block was measured every 24 h before surface hardness measurements were performed. Blocks were taken out of solution, and then weighed using a microbalance (Precisa XT 120A, Dietikon, Switzerland). Mass loss measurement was performed under the same conditions for each sample. Blocks were gently pat dried in the same manner and weighed immediately to ensure consistent treatment before testing. All mass loss measurements were obtained under ambient conditions of $60 \pm 5\%$ relative humidity and 23 ± 2 °C. The percentage change in mass was a combination of water uptake (absorption) and dissolution (solubility) of the GIC.

Vickers microhardness measurements were determined from indentations on the lapped GIC surface using a Microhardness tester (MHT-10, Anton Paar GmbH, Graz, Austria) attached to a microscope (Leica DMPL, Leica Microsystems Wetzlar GmbH, Germany). Two indentations were made on each block (force, 1.0 N; dwell, 6 s; rate, 0.99 N/min). The indentations were separated by a distance of at least three times the indentation size. Images of the indentations were acquired through a calibrated digital camera (Leica DFC320) mounted on the microcope (Leica DMLP, Leica Microsystems Wetzlar GmbH, Germany) and distance measurements made using Image Tool software (Version 3.0, UTHSC, San Antonio, TX) which were then converted into Vickers hardness values. Blocks were then placed into fresh batch of solution and returned to the incubator.

The ion release of calcium, phosphate and fluoride after each 24 h period of storage was determined using atomic