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Acta Biomaterialia 2 (2006) 109–112
Acta BIOMATERIALIA
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Brief communication

The effect of oxalic acid incorporation on the setting time and strength of a glass-ionomer cement

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Received 5 April 2005; received in revised form 12 August 2005; accepted 18 August 2005

Abstract

Oxalic acid and its metal oxalate salts have been used extensively in dentistry in a range of applications: as desensitisers, in cavity preparation, and as bonding agents. This study investigated the influence of oxalic acid upon the working time, initial setting time, 24-h hardness and compressive strength of a glass-ionomer cement. Conventional glass-ionomer liquids were prepared from polyacrylic acid, tartaric acid, water, and oxalic acid at concentrations of 0–7% w/w. Liquids were dosed into capsules with a commercial glass-ionomer powder, activated and mixed. The resultant pastes were assessed for working time, initial setting time, 24-h hardness and 24-h compressive strength. Liquids containing 0.5–1% oxalic acid lengthened the working time and initial setting time. At concentrations greater than 2%, both working and initial setting times decreased with increasing oxalic acid. Surface hardness values using liquids with 3% and 7% oxalic acid were less hard than the control. Compressive strength was unchanged over the concentrations tested. Oxalic acid may be a useful reaction modifier in glass-ionomer systems. It accelerated the setting reaction without affecting strength, but was limited to low concentrations because of its relatively poor solubility in water.
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Keywords: Glass-ionomer cement; Compressive strength; Setting time; Oxalic acid; Surface hardness

1. Introduction

Oxalic acid and its metal salts have been widely used in dentistry for three main applications: as desensitisers, as dentin permeability modifiers/sealants and as cavity pretreatments to improve bonding. Some of these advantages may also be evident in glass-ionomer formulations containing oxalic acid.

As desensitisers, solutions containing oxalic acid and/or oxalates of various metals are commercially available [1]. Examples include Sensodyne Sealant (GlaxoSmithKline, Germany) and MS Coat (J. Morita Europe GmbH, Germany). These materials are recommended for use as dentin sealants, where the oxalic acid will precipitate rapidly as an insoluble salt (either calcium or ferric), block the dentinal tubules and eliminate sensitivity caused by osmotic pressure changes to the pulp [2].

Oxalates are also used in pretreatments of cavities to reduce dentin permeability [3] as a means of reducing associated sensitivity [4], though some difficulties remain in the evaluation of effectiveness [5,6]. Various materials are available, including the dentin bonding agent Tenure Quik (DenMat, Santa Maria, CA), on which several studies have been conducted [7], with some success [8].

Kanca [9] found oxalic acid and oxalate-containing bonding pretreatments to reduce bond strengths of resin-modified glass-ionomer cements (GICs) to dentin, and recommended the procedure be contra-indicated. Haveman and Charlton [10] reported similar results. The suggested mechanism for the decreased bond strengths in both studies was deposition of oxalate precipitates onto the dentin surface, which acted as a barrier to effective material penetration and bonding. It is this very mechanism that is reported to provide some sensitivity relief. Others research

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doi:10.1016/j.actbio.2005.08.007

has examined the usefulness of aluminium or ferric oxalate for dentin bonding of resin composite [11,12], with mixed results. Chohayeb and Rupp [13,14] studied the effects of oxalate pretreatments on microleakage of conventional GICs and composites, and reported lower microleakage scores when pretreatments contained aluminium or ferric oxalate. Yoshida et al. [15] found that oxalic acid bonds well to hydroxyapatite, but did not examine bond strengths of oxalic-acid containing materials.

These applications of oxalic acid may indicate its potential usefulness in glass-ionomers. In particular, the rapid precipitation of insoluble species may accelerate the reaction, while improvements to both adhesion and sealing ability of glass-ionomers would be advantageous. The role of oxalic acid in GICs has been almost ignored, although significant attention has been given to tartaric acid, which is larger only by $C(OH)_2$. Oxalic acid is the smallest dicarboxylic acid, and the strongest organic acid. It has potential in GICs to accelerate the reaction by more rapid ion leaching, strengthen the cement by improving cross-linking and improve the clinical handling of the material by reducing the initial viscosity. Given the previous literature on the use of oxalic acid in dentistry, it is likely that a GIC incorporating oxalic acid will decrease sensitivity and improve sealing of GIC restorations.

A precondition of such incorporation is the continued clinical and mechanical performance of such a material; this investigation examines the effect of oxalic acid on these properties in a GIC system, with a view to potential further investigation of the clinical effects of such incorporation. The null hypothesis is that oxalic acid incorporation into the GIC liquid has no effect on working time, initial setting time, compressive strength or surface hardness.

2. Materials and methods

Conventional glass-ionomer liquids were prepared using a polyacrylic acid (Sokalan® CP-12S), L-(+)-tartaric acid, oxalic acid and water (Table 1). Liquids were prepared at various concentrations of oxalic acid, replacing polyacrylic acid, at concentrations from 0% to 7% (Table 2). The upper limit of oxalic acid content was limited by the room temperature solubility of oxalic acid in the system, as determined by a pilot study.

Capsules from a commercial glass-ionomer capsule system (Riva SC, SDI Ltd.) were dosed with powder (Riva SC A3 Shade, SDI Ltd.) and liquid at a powder:liquid ratio of 3.0:1. Seven capsules were prepared with each liquid; three were used for working and initial setting time determination and four for 24-h compressive strength and surface hardness. Powder and liquid doses were sufficient for the manufacture of three compressive strength specimens from each capsule, as determined in a previous pilot study.

2.1. Working and initial setting times

Working time and initial setting times were determined from extruded pastes as they progressed through the setting reaction at room temperature (23 ± 1 °C). The working time of extruded pastes was determined in a manner based on that of Mount [16], as the time after which the material would no longer string cohesively to a height of 10 mm. The initial setting time was determined in a manner analogous to ISO9917:2003 [17] as the point at which no permanent indentation could be made in the surface of the cement with a 1-mm diameter amalgam plugger applied with a 300*g* force.

2.2. Compressive strength and surface hardness

Twelve cylindrical specimens of height 6.0 ± 0.1 mm and diameter 4.0 ± 0.1 mm were prepared using each powder–liquid combination, and tested for compressive strength according to ISO9917 [17], using stainless steel split moulds. Mixed capsules were extruded into the moulds, covered with mylar tape and glass slides, compressed to remove excess, and stored at >80% humidity at 37 ± 1 °C for 1 h. The specimens were removed from the moulds and immersed in water at 37 ± 1 °C for a further 23 h.

Specimens were visually examined for pores or defects prior to testing, and defective specimens rejected. Immediately prior to compressive strength testing, surface hardness data, as Vickers hardness numbers (VHN), were obtained using a micro-indentation tester (MMT-3 Digital Hardness Tester, Buehler Ltd., Lake Bluff, IL) using a 300*g* force. Specimens were mounted and loaded axially. The micro-indentations formed in the end surfaces of the specimens had been determined in a pilot study to have no significant effect on the compressive strength of the cement. For compressive strength testing, the ends of each specimen were sanded flat on 600-grit SiC paper, and the specimen axially loaded to fracture on a universal testing

Table 1
Materials

Material	Supplier	Batch	Description/ingredients
Sokalan® CP-12S	BASF, Ludwigshafen, Germany	30510968E0	Modified polyacrylic acid, 50% w/w in H_2O, MW ≈ 3000
Tartaric acid	Australian tartaric products, Red Cliffs, Australia	IJ0326	L-(+)-tartaric acid, 99.5+%
Oxalic acid	Sigma–Aldrich, St. Louis, MO, USA	102K1247	Oxalic acid, dihydrate, 99+%
Riva SC powder, A3 shade	SDI Ltd., Bayswater, Australia	040801	Strontium fluoroaluminosilicate glass, polyacrylic acid, pigments

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