Modification of mineral trioxide aggregate. Physical and mechanical properties

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Abstract

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Aim To evaluate the physical and mechanical properties of two cements with a similar chemical composition to mineral trioxide aggregate and to attempt to improve their properties by producing a composite material.

Methodology Two cement types were used: a mixture of calcium sulpho-aluminate cement and Portland cement (CSA) and calcium fluoro-aluminate cement (CFA) in conjunction with an admixture to improve the handling characteristics. Cements were mixed with an inert filler to produce cement composite. The setting time of the cements was evaluated using an indentation technique. The flexural and uni-axial compressive strengths and solubility of the cements and cement composites were evaluated. The properties of the materials were compared with proprietary brand glass–ionomer cement.

Results The setting time of the CSA and CFA cements was less than 6 min. The use of an admixture to improve the handling properties tended to retard setting. CSA was stronger then CFA in both compression and flexure ($P < 0.001$). Addition of granite increased the flexural strength of both cements but reduced the compressive strength ($P < 0.01$). CFA absorbed more water then CSA. Addition of granite reduced the water uptake of both cements.

Conclusions Both CSA and CFA cements had adequate setting times and compressive strength values when compared with proprietary brand glass–ionomer cement. CSA was superior to CFA and had more promise as a prospective dental material.

Keywords: accelerated cements, compressive strength, flexural strength, Portland cement, setting time.

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Introduction

Mineral trioxide aggregate (MTA) is composed of Portland cement and bismuth oxide (Torabinejad & White 1995). MTA was developed as a root-end filling material following apicectomy and to repair root perforations (Lee *et al.* 1993, Pitt Ford *et al.* 1995). The material has been used successfully in this regard and indeed, its use has been extended. For example, MTA has been reported to be suitable for use as a pulp capping agent (Pitt Ford *et al.* 1996, Bakland 2000); as a dressing over pulpotomies of permanent (Holland *et al.* 2001) and primary teeth replacing the formocresol pulpotomy procedure (Eidelman *et al.* 2001); for obturation of retained primary teeth (O'Sullivan & Hartwell 2001), and permanent immature (Hayashi *et al.* 2004) and mature teeth (Vizgirda *et al.* 2004); for single visit apexification procedures for immature teeth with necrotic pulps (Whiterspoon & Ham 2001), thus acting as an apical barrier material (Shabahang & Torabinejad 2000); and as a root canal sealer cement (Holland *et al.* 1999, Geurtsen 2000). Unfortunately, MTA has limited applications in operative dentistry because of its long setting time and low compressive strength compared

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with other materials (Torabinejad *et al.* 1995). The extended endodontic uses and its potential use in operative dentistry necessitate the development of a new formulation of the material based on Portland cement, which optimizes both its strength and its setting time without compromising its biocompatibility.

Portland cement takes a minimum of 4 h to achieve a final set (Taylor 1997). The rate of reaction is controlled by the calcium sulphate (gypsum) which is added by the manufacturer for that purpose. The calcium sulphate reacts with the tricalcium aluminate in Portland cement to produce a high-sulphate calcium sulpho-aluminate (ettringite). In the absence of calcium sulphate, the tricalcium aluminate forms a hydrated calcium aluminate of cubic structure leading to a flash set. Thus, the calcium sulphate is added by the manufacturer to avoid the formation of the hydrated calcium aluminate thus delaying the hydration mechanism (Taylor 1997).

The setting time of Portland cement can be reduced by addition of an accelerator, the removal of gypsum from the cement and addition of calcium aluminate cement (Neville 1981). Removal of gypsum results in a flash set which can be controlled by superplasticizing admixtures (Camilleri *et al.* 2005). When calcium aluminate cement is added to Portland cement the former reacts to the calcium sulphate (gypsum) in the Portland cement leaving the tricalcium aluminate free to flash set, thus resulting in a shorter setting time (Neville & Wainwright 1975). Cements produced by addition of calcium aluminate cement to Portland cement have a reduced compressive strength (Camilleri *et al.* 2006). The compressive strength of the cement can be improved by reduction in the water to cement ratio. This is achieved through the use of super-plasticing admixtures. The plasticizing action of water reducers is related to their adsorption and dispersing effects in the cement–water system (Hattori 1978).

The aim of the study was to test the suitability of two cement types based on Portland cement and to improve their properties by admixing them with an inert material. The physical and mechanical properties of these materials were evaluated and compared with proprietary brand glass–ionomer cement.

Materials and methods

Two cement types were used:.
1. CSA: calcium sulpho-aluminate cement mixed in the following proportions

- 3 parts calcium aluminate (Lafarge Special Cements, Nottingham, UK)
- 8 parts white Portland cement (Lafarge Asland, Valencia, Spain)
- 1 part synthetic anhydrite (Lafarge Special Cements, Nottingham, UK)

2. CFA: calcium flouro-aluminate cement (Italcementi SPA, Bergamo, Italy).

A super-plasticizing admixture (Degussa Construction Chemicals, Manchester, UK) was added to the mixing water to increase the workability of the mix and also to reduce the amount of water required by the cement during mixing. A cement composite made up of the cement mixed with granite (Halmann, Iklin, Malta) was also tested (CSAG, CFAG). The granite used was obtained by grinding a slab to very fine particles retained on a 150 μm sieve. The proportion of cement and granite was calculated using the Theory of Particle Admixtures (Dewar 1999). Glass–ionomer cement (GIC; Ketac Molar, 3M Seefeld, Germany) was used as the control.

Setting time of cements

The setting time of the cements with and without the addition of the super-plasticizing admixture was tested using the Vicat apparatus (British Standard Institution 2005b). Four hundred grams of the cement was mixed with water on a moistened aluminum tray with a small trowel at a water/cement ratio of 0.25 as suggested by the manufacturer. The super-plasticizing admixture was added at the end of the mixing process. The dosage was adjusted to 0.8 L per 100 kg of cement, again as suggested by the manufacturer. The mix was then compacted in a two-part brass mould and initial and final setting times were determined. The Vicat assembly was placed in an incubator at 37 °C. The test was repeated thrice.

Flexural and compressive strength

The flexural and compressive strengths of the cements were tested according to British Standard Institution (2005a). A total of 270 g of cement was mixed thoroughly with water at the pre-determined water/cement ratio for 2 min. The cement was then loaded into a prism mould $40 \times 40 \times 160$ mm in size previously coated with mould oil (Separol, Sika, Switzerland). The cement was compacted in two layers and a vibrating table was used during the compaction to avoid air entrapment in the cement mix. After casting

was complete, the moulds were covered with a plastic sheet to avoid loss of moisture and the cements were allowed to cure for 24 h. Twenty-four prisms were cast of each cement type. They were then removed from the mould and cured at 37 °C in water. Flexural strength as performed after 1 day, and then after 7, 28 and 56 days. The prisms were mounted in a flexure testing jig. This jig allowed for 3-point bending to be performed. The distance between the lower rollers was 100 mm and the upper roller was placed in the middle. The flexure jig was mounted between the platens of a compression machine (Controls spa, Bergamo, Italy). A 15 kN load cell was used and the load applied was accurate to 0.1%. The cements were loaded at 50 N s^{-1} until they failed. Flexural strength was calculated using the following formula:

$$R_f = \frac{1.5 \times F_f \times l}{b^3}$$

where R_f is the flexural strength in N mm^{-2}, F_f is the load applied to the middle of the prism at fracture, l is the distance between the supports in mm and b is the side of the square section in mm.

After flexural strength was determined the prism halves were tested in compression at 14.4 kN s^{-1}. A 250 kN load cell was used for this test with an accuracy up to 0.1%. Compressive strength was calculated using the following formula:

$$R_c = \frac{F_c}{1600}$$

where R_c is the compressive strength in N mm^{-2}, F_c is the maximum load at fracture in N, $1600 = 40$ mm $\times 40$ mm is the area of the platens in mm^2.

In addition to the cements, cement composite was also tested. The composite was produced by addition of granite to the two cement types.

Preliminary tests to establish the relative density (British Standard Institution 1999b), the loose bulk density (British Standard Institution 1998), the moisture content (British Standard Institution 1999a) and the water absorption (British Standard Institution 2000) of the granite were performed. Tests on cement were carried out to establish the relative density (British Standard Institution 1992), the moisture content (British Standard Institution 1999a) and the fineness (British Standard Institution 1992). After performing the preliminary tests on the cements and aggregate, the proportion of granite to cement was deduced using the Theory of Particle Admixtures (Dewar 1999). Quantities of each material required to achieve the best

particle packing and optimal physical properties were thus calculated. The cement composite was tested in both flexure and compression in the same way as described for the cements.

Solubility

The solubility of CSA, CFA, granite admixed cements (CSAG, CFAG) and glass–ionomer cement was calculated by measuring the water uptake of the cements. Twelve samples each 10 mm in diameter and 2 mm thick were made for each cement type. Six were cured in water and the rest were cured at 100% humidity at 37 °C. The weight of the samples was recorded after 1, 7, 28 and 56 days.

Statistical analysis

For all tests carried out, the data were evaluated using STATISTICAL PACKAGE FOR THE SOCIAL SCIENCES (SPSS Inc., Chicago Ill, USA) software. The distribution was first evaluated to determine the appropriate statistical test. The data were plotted and the distribution curve was analysed together with the Kolmogorov–Zmirnov test with $P = 0.05$. The P-value of >0.05 signified a normal distribution and thus parametric tests were performed. With normally distributed data, analysis of variance (ANOVA) with $P = 0.05$ was performed to evaluate variations between the means. Then two-tailed independent sample t-tests at 95% confidence level with $P = 0.05$ was used to analyse the data.

Results

Setting time of cements

The setting times of the cements is shown in Table 1. Both cement types had a very rapid set, with CFA setting faster than CSA; however, this difference was

Table 1 Means of initial and final setting times of different cement types $\pm$ SD ($n = 3$)

Cement type	Water %	Admixture % weight of fine material	Initial set minutes	Final set minutes
CSA	28	0.5	2.41 ± 0.37	4.05 ± 0.09
CFA	27	0.5	1.83 ± 0.29	3.08 ± 0.14
CSA and admixture	25	0.5	3.67 ± 0.14	5.25 ± 0.25
CFA and admixture	25	0.5	3.05 ± 0.18	5.42 ± 0.14

not statistically significant. The use of a super-plasticizing admixture retarded the final setting time of the cements, reduced the water required by the cements and improved the handling properties.

Flexural and compressive strength

The results for the physical properties of the cements and aggregate are shown in Table 2. The moisture content of the cements was minimal, thus they were used without desiccation. The quantities of material required to produce the cement composite as calculated by the Theory of Particle Admixtures (Dewar 1999) are shown in Table 3.

The results for the flexural strength testing of the cements are shown in Fig. 1a and for the compressive strength tests in Fig. 1b. The CSA cement was stronger than the CFA cement both in compression and in flexure at all curing times ($P < 0.001$) except for flexural strength at 1 day where no difference between the two cements was demonstrated ($P > 0.05$). Addition of granite to the CSA cement resulted in an increase in flexural strength at 28 and 56 days but a reduction in the compressive strength at all ages ($P < 0.001$). Addition of granite to CFA increased the flexural strength up to 28 days ($P < 0.01$) but the

Table 2 Results of preliminary tests carried out on (a) granite ± SD; (b) cements ± SD

Test	Result
(a)	
Relative density kg m^{-3}	2.68 ± 0.56
Loose bulk density g ml^{-1}	1.11 ± 0.1
Moisture content %	0.02 ± 0.01
Water absorption %	1.4 ± 0.19

	Cement type		
Test	CSA	CFA	OPC
(b)			
Relative density kg m^{-3}	2.95 ± 0.08	2.85 ± 0.04	2.94 ± 0.01
Moisture content %	0.08 ± 0.06	0.06 ± 0.01	0.54 ± 0.05
Fineness m^2 kg^{-1}	450	450	421 ± 0.9

The fineness of the CSA and CFA was determined by the manufacturer.

Table 3 Percentage ratio of materials used in the mix design

Cement type	% cement	% granite	% water	% admixture
CSA	100	0	25.00	0.5
CFA	100	0	25.00	0.5
CSAG	58.25	41.60	15.19	0.5
CFAG	58.64	41.36	15.28	0.5

flexural strengths of the cement and the cement composite were the same at 56 days ($P > 0.05$). The compressive strength of the calcium fluoro-aluminate cement did not change markedly with addition of granite. All the cements tested failed in the normal mode.

Solubility

The results for the water uptake and loss of the cements and cement composites is shown in Table 4. The CFA cement absorbed more water than the CSA cement at all ages ($P < 0.01$). Both cement composites absorbed less water in comparison with the parent cement ($P < 0.001$). The glass–ionomer cement did not show a progressive water uptake or loss with time with the peak water absorption being at 7 days of curing. The CSA cement and composite were quite stable and both lost minimal amounts of water when cured at 100% humidity ($P > 0.05$). Both the CFA cement and composite exhibited extensive water loss at all ages.

Discussion

Compressive strength testing of dental cements is usually performed according to British Standard Institution (2003). In a previous paper (Camilleri *et al.* 2006) testing various proprietary brands, it was concluded that compressive strength testing depends on specific test conditions. The cements were not susceptible to changes in the compressive strength testing procedure at 1 and 7 days, but at 28 days all the fast setting cements had a significantly higher strength with a particular testing mode. Abnormal failure occurred when testing according to British Standard Institution (2003) and the standard deviations for the tests carried out were large. Compressive strength testing of prototype dental cements was thus performed according to British Standard Institution (2005a).

The setting time of the cement was controlled by addition of calcium aluminate. The gypsum present in the Portland cement system reacts with the calcium aluminate to produce ettringite as the main by-product of the hydration reaction. Once the gypsum is depleted, the calcium silicate hydration reaction is accelerated. This has already been investigated in a previous paper (Camilleri *et al.* 2006) where Portland cement was admixed with calcium aluminate cement; This resulted in poor compressive strengths. In the present experiment, excess sulphate ions provided by the synthetic anhydrite reacted with the calcium hydroxide produced
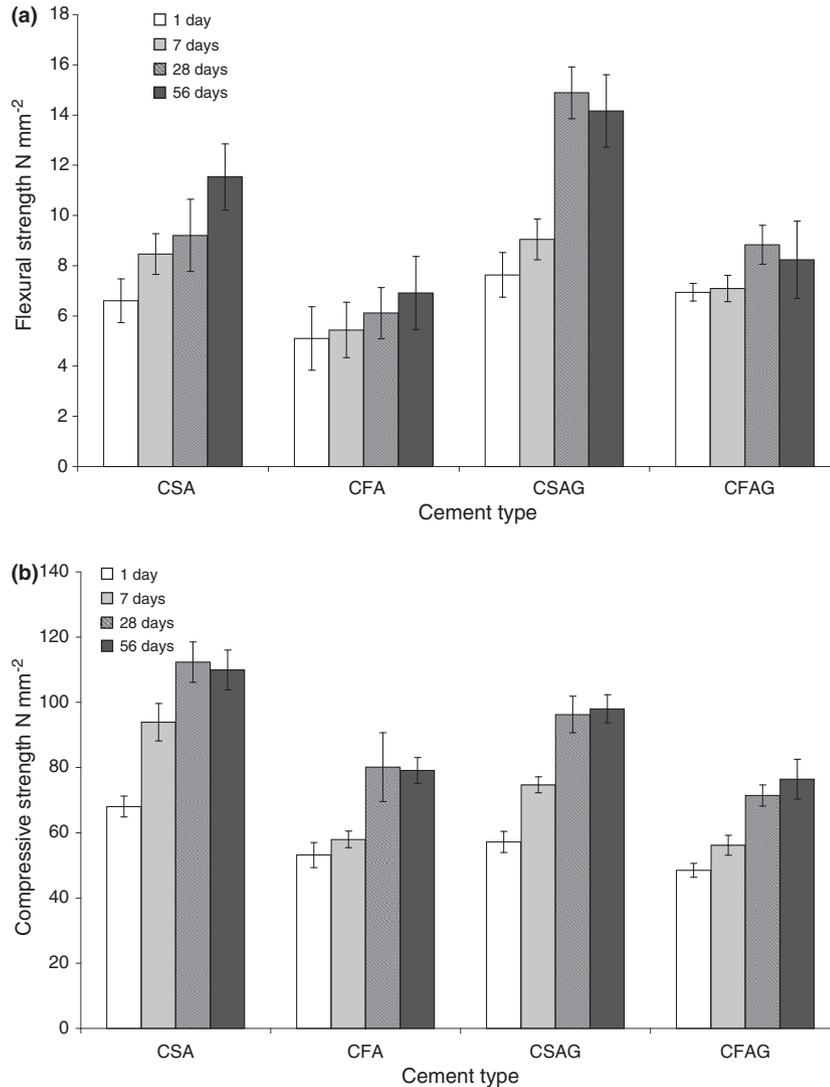
Figure 1 (a) Mean flexural strength testing of cements and cement composites over a period of 56 days ± SD (*n* = 6). (b) Mean compressive strength testing of cements and cement composites over a period of 56 days ± SD (*n* = 12).

Table 4 Percentage water absorption/shrinkage of cements cured at 100% humidity or immersed in water over a period of 56 days ± SD (*n* = 6)

Cement type	%water absorption of cements cured in water at 37 °C/days				%water absorption of cements cured at 37 °C and 100% humidity/days			
	1	7	28	56	1	7	28	56
CSA	4.16 ± 0.6	4.81 ± 0.5	5.60 ± 0.6	5.90 ± 0.8	−0.80 ± 0.1	−1.09 ± 0.1	−1.62 ± 0.2	−1.97 ± 0.3
CSAG	0.99 ± 0.1	1.29 ± 0.1	2.17 ± 0.1	2.72 ± 0.1	−0.63 ± 0.2	−1.14 ± 0.1	−1.21 ± 0.1	−1.37 ± 0.2
CFA	5.61 ± 0.3	7.57 ± 0.3	7.76 ± 0.2	8.32 ± 0.2	−7.27 ± 0.5	−7.37 ± 0.6	−6.64 ± 0.8	−6.43 ± 0.8
CFAG	2.22 ± 0.6	3.23 ± 0.8	3.22 ± 0.7	3.57 ± 0.7	−5.10 ± 0.2	−5.14 ± 0.1	−4.83 ± 0.2	−4.81 ± 0.2
GIC	1.32 ± 0.0	5.05 ± 2.1	2.92 ± 0.1	3.32 ± 0.2	−2.17 ± 0.3	−3.36 ± 0.4	−3.19 ± 0.4	−3.87 ± 0.5

by the hydration of the tricalcium silicate that increased the ettringite deposition and resulted in an increase in initial strength (Knöfel & Wang 1993). Strength development seemed to show a maximum within the first few days. A qualitative explanation of this behaviour is based on a combined process of expansive cracking and

self-healing, the balance of which appears to determine the course of expansion and strength development (Bentur *et al.* 1975). The extent of ettringite-related expansion depends on the conditions of curing. It increases with increasing amounts of water taken up from the environment while ettringite is formed, indicating the participation of swelling phenomena in the expansion process (Odler & Colán-Subauste 1999). Curing in water enhances early ettringite formation and increase in strength. The fluoro-aluminate cement contains sufficient tricalcium silicate to achieve high early strengths (Costa *et al.* 2000). Utilization of calcium fluoro-aluminate leads to slow ettringite formation, which could explain the low compressive strength as compared with the sulpho-aluminate cement.

Attempts at improving the material strength were made by using a superplacticizing admixture and by addition of inert filler. The superplasticizing admixture causes particle dispersion and allows workability at low water/cement ratios. Maximum particle packing is attained by dispersion of the cement grains at low water/cement ratios with the use of high levels of a dispersing agent. There is a practical limit to the reduction of water/cement ratio as the mixture would become unworkable. The flocculated nature of the cement particles does not permit fluid suspensions unless they are dilute (Malhotra *et al.* 1978). In addition, the cement was mixed with an inert material of different particle size in an attempt to improve its flexural strength. In a particle admixture macroscopic porosity remains as a result of air entrainment and defective particle packing. To attain the ideal structure of strong cement paste, maximum particle packing with minimal total porosity and elimination of macroscopic voids is necessary. The use of cement on its own to produce any kind of material is difficult because of development of microcracks. The addition of an aggregate is thus mandatory. The strength of a composite material is influenced by the ratio of cement to mixing water, the ratio of cement to aggregate, the grading, surface texture, shape, strength, and stiffness of aggregate particles, and the maximum size of the aggregate (Gilkey 1961). The Theory of Particle Admixtures (Dewar 1999) utilizes parameters calculated by physical tests carried out on the raw materials to calculate the percentages of materials required to produce optimal particle packing for maximum strength.

Glass–ionomer cements exhibited hygroscopic expansion on immersion in water. This expansion reached its peak after 7 days immersion. Both prototype cements showed higher water absorption than glass–ionomer cement. Addition of granite to the cements reduced the water exchange within the material. Both calcium sulpho-aluminate cement and its composite lost less water when cured at 100% humidity than glass–ionomer cement. The results obtained for the prototype dental cements are comparable with those of proprietary brand glass–ionomer cement (McCabe *et al.* 1990, Pereira *et al.* 2002, Piwowarczyk *et al.* 2002).

Conclusions

The CSA and CFA cements had adequate setting times and compressive strength values when compared with proprietary brand glass–ionomer cement. CSA was superior to CFA and had more promise as a prospective dental material. The improved properties of the modified cement could allow for a reduction in the chair-side time and amount of visits per treatment when the cement is used for apexification procedures and pulpotomies, and enable the material to withstand packing pressures of restorative materials if used as a pulp capping agent.

Acknowledgements

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