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Original

The effects of erroneous mixing of zinc carboxylate cements

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Abstract: The mechanical properties of luting agents are determined by the mixing ratio of powder and liquid. The purpose of this *in vitro* study was to evaluate the therapeutic range of zinc carboxylate cements by investigating the mechanical properties of such cements when formulated using erroneous powder/liquid ratios. Using the recommended powder/liquid ratio as a standard, four different mixing ratios (25% and 50% more or less powder) were used to prepare each carboxylate cement (*Aqualox*, *Carboco*, *Durelon*). A veneering composite (*Sinfony*) was used as control. Vickers hardness and three-body abrasion were evaluated. In each carboxylate cement, both a 25% and 50% increase in liquid content resulted in significantly lower Vickers hardness, whereas a higher powder content altered the saturation concentration but did not result in increased hardness; this effect was particularly obvious in *Carboco*. *Durelon* showed a linear relationship between wear and powder/liquid ratio, whereas *Carboco* and *Aqualox* displayed a more exponential increase in wear in mixings with an increased liquid content. The mechanical properties of carboxylate cements were altered in mixtures with an increased liquid content, but not in those with a high powder content. Mixing ratio errors greater than 25% may significantly affect the clinical performance of carboxylate cements. (J Oral Sci 52, 89-93, 2010)

Keywords: cement; luting; powder liquid ratio; mixing error; hardness; wear resistance.

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Introduction

Zinc carboxylate cements are primarily used for luting permanent alloy restorations (1,2). These cements are preferred over zinc phosphate or glass-ionomer cements because they avoid pulpal irritation (2,3). When first mixed, zinc carboxylate cements are slightly more acidic than zinc phosphate cements. However, since acid is weakly dissociated (4), there is minimal penetration of the pulp tissue by high-molecular-weight polymer molecules. Despite this advantageous clinical characteristic, the propagation of zinc polycarboxylate cement is low, possibly because of its poor mechanical properties (5). There are no clinical data on the performance of zinc polycarboxylate cements; indeed, even *in vitro* studies of their mechanical properties are few in number (1,5,6). Therefore, this *in vitro* study aimed to increase knowledge of zinc carboxylate cements by investigating the mechanical properties of various mixing ratios of three representative zinc polycarboxylate cements. It was hypothesized that variations in the powder/liquid ratio, such as those caused by mixing errors, would further degrade the mechanical properties of zinc polycarboxylate cements.

Materials and Methods

Three carboxylate cements were used in this investigation: *Durelon* (3M Espe, Seefeld, Germany), *Aqualox* (Voco, Cuxhaven, Germany), and *Carboco* (Voco, Cuxhaven, Germany). A light-curing veneering composite (*Sinfony*, 3M Espe, Seefeld, Germany) was used as an internal standard for control of the wear assay. In addition to the recommended powder/liquid ratios, which are shown in Table 1, four additional mixing ratios, requiring 25% or 50% more or less powder, were investigated for each cement. Vickers hardness and three-body abrasion were then evaluated.

Vickers hardness

Vickers hardness (VH) was measured using the Zwick device B3212001 (Zwick, Ulm, Germany). A load of 0.5 kg was applied to coplanar specimens for 60 s by using a pyramid-shaped die. The depth of the impression represented the hardness of the sample. Vickers hardness was proportional to the quotient of the applied force and the impression surface. This surface was part of the pyramid with a square base. Pyramid and impression were considered to have identical surface angles. Therefore, Vickers hardness was calculated using the following formula:

$$VH = \frac{0.102 \times F \times \sin 136°/2}{d^2}$$

F = force (9.81 m/s² × 0.5 kg)
d = diagonal of pyramid base

Three-body abrasion test

Twelve specimens were mounted onto a tooth-wheel shaped sample holder and stored in distilled water for 24 h (7). The equipped wheel (diameter = 50 mm, thickness = 10 mm; 130 rpm) was ground with a smaller diamond wheel (d = 16 mm, t = 6 mm; 60 rpm) that moved in a counter-clockwise direction under a pressure of 15 N. The abrasion medium consisted of millet seed shells (30 g) and rice (120 g), which were ground in a rotating blade grinder for 60 s (Moulinette, Moulinex, Paris, France). Rice and seed shells were mixed with 275 ml distilled water and soaked for 1 h.

The smaller antagonist created a 6-mm wide trace in the middle of the specimens on the sample wheel. The unworn margin area served as reference. Wear track depth was measured after 15,000 cycles using a profilometric surface contact testing device (Perthometer S6P, Perthen-Feinprüf, Göttingen, Germany). A higher number of cycles caused degeneration of the cement surfaces, which made measurements impossible.

Statistical analysis

Statistical analysis was performed using SPSS 13.0 for Windows (SPSS Inc., Chicago, USA). Means plus standard deviation or 95% confidence interval (CI) (Figs. 1 and 2) were calculated. One-way ANOVA was used to investigate statistical differences, and a P value of less than 0.05 was considered to indicate statistical significance.

Results

Vickers hardness

In each carboxylate cement, Vickers hardness was significantly lower when liquid content was 25% and 50% higher; however, hardness was significantly higher for *Aqualox* and *Durelon* when using 20% more powder. In each cement tested, a further increase in powder content appeared to alter the saturation concentration but did not increase hardness. This effect was particularly obvious with *Carboco* (Fig. 1 and Table 1).

Three-body abrasion

Durelon showed a more linear relationship between wear and powder/liquid ratio; however, the presence of 25% more or less liquid did not significantly change the wear rate. *Aqualox* showed an exponential increase in wear with increased liquid content. A decrease in liquid of more than 25% reduced the wear rate significantly; however, the changes were not statistically significant when more liquid was added. *Carboco* had the highest wear rates at the standard mixing ratio, but this difference was not statistically insignificant.

Each cement and mixing ratio investigated had

Table 1 Means plus standard deviation for Vickers hardness (VH) and three-body abrasion (wear) of polycarboxylate cements mixed in various powder/liquid ratios

Powder content	Aqualox	VH	Wear [μm]	Carboco	VH	Wear [μm]	Durelon	VH	Wear [μm]
-50%	2.7:1	7.7 ± 1.0	220.7 ± 50	1.5:1	10.9 ± 2.5	184.1 ± 36	1:1	12.5 ± 2.6	112.8 ± 23
-25%	4.1:1	15.2 ± 1.8	134.8 ± 29	2.25:1	18.1 ± 2.8	162.7 ± 28	1.5:1	15.9 ± 2.5	99.7 ± 17
Standard	5.5:1	26.8 ± 3.7	88.5 ± 24	3:1	24.1 ± 6.2	139.1 ± 40	2:1	19.6 ± 2.0	76.8 ± 11
+25%	6.9:1	37.9 ± 4.6	78.7 ± 10	3.75:1	27.1 ± 5.6	154.1 ± 26	2.5:1	26.2 ± 2.1	66.6 ± 14
+50%	8.3:1	41.4 ± 3.3	62.6 ± 15	4.5:1	29.8 ± 6.2	143.4 ± 33	3:1	29.2 ± 2.7	57.4 ± 11

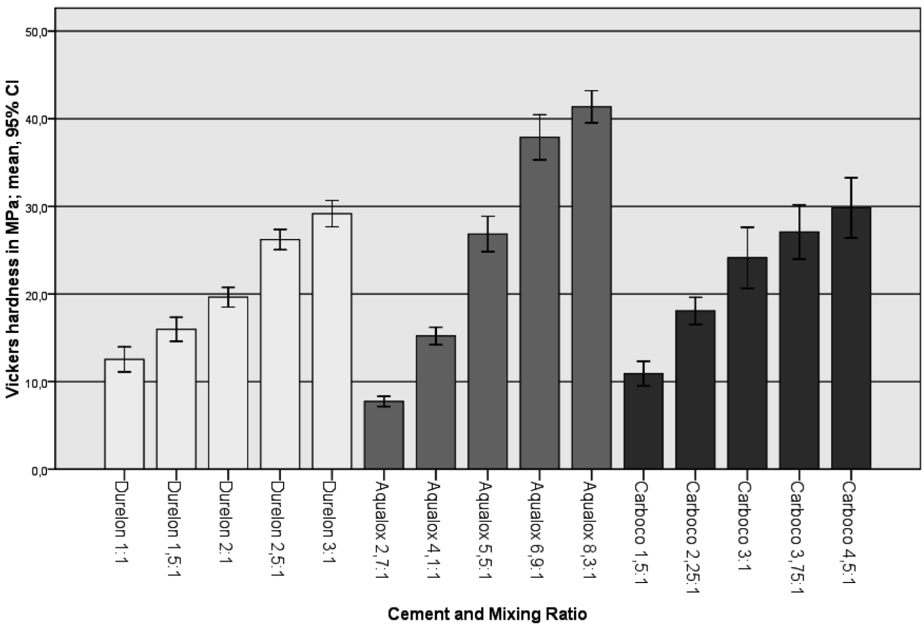

Fig. 1 Vickers hardness of polycarboxylate cements mixed in various powder/liquid ratios.

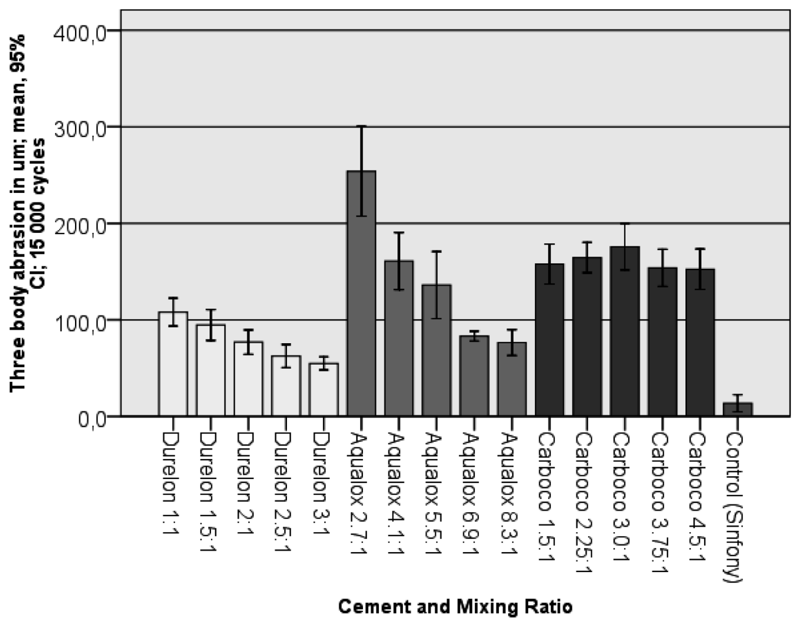

Fig. 2 Three-body abrasion of polycarboxylate cements mixed in various powder/liquid ratios.

considerably higher wear rates than did the veneering composite *Sinfony* (control) (Fig. 2 and Table 1).

Discussion

Clinical trials of conventionally luted fixed dentures have described the frequencies of secondary caries defects, endodontic treatment, and loss of retention (8-11). However, no study has investigated whether these complications were caused by cement mixing errors. Because a clinical trial using various cement mixing ratios cannot be conducted for ethical reasons, *in vitro* assays are necessary to determine how mixing errors affect the properties and clinical characteristics of cement. There have been studies of the effect of variability and errors in mixing on the mechanical properties of glass-ionomer, zinc phosphate, and resin cements (12-15); however, there have been no such studies of carboxylate cements. The most common relevant measurements are those related to flexural strength, fracture strength, hardness, and wear, as these properties largely determine clinical performance (16). Flexural and fracture strength were not measured in this study because of the brittleness of carboxylate cement. We instead measured Vickers hardness and three-body abrasion, as these are important parameters for the marginal area of fixed dentures. However, it might be useful for future studies to elucidate the effects of various (erroneous) cement mixing ratios on additional parameters, including compressive strength, acid erosion, and cement layer thickness.

In addition to the standard ratio of powder and liquid, we used four other mixing ratios – plus and minus 25% and 50% powder – for each cement. It was shown that even the use of proportioning aids resulted in ratio errors of up to 20%. The aim of this study was to investigate the effects of major (50%) and moderate (25%) mixing errors, so as to determine the therapeutic range of carboxylate cements. Although all three investigated cements were carboxylate cements, they had different hardness characteristics when the powder-liquid ratio was changed. We found that the greater the difference from the standard ratio, the greater the increase or decrease in hardness. Therefore, narrow cement powder/liquid ratios – ideally at a ratio of 1:1 – are clearly preferable. When powder content was increased, all the carboxylate cements tended to shift toward saturation, and hardness and wear improved less or not at all. However, clinical observation has shown that the tendency to use too much liquid is more common. Unfortunately, excess liquid content results in significantly decreased hardness.

Variation in mixing ratios considerably changed the three-body abrasion results of the cements. *Durelon* showed a linear relationship between powder content and wear, and the highest mean value was double the lowest. For *Carboco*, variation in mixing ratios had an insignificant effect on wear properties, which was unexpected. For *Aqualox*, however, the large difference in the standard powder/liquid ratio (5.5:1) may explain the considerable increase in wear rate that occurred with rising liquid content: there was an almost four-fold difference between the lowest and the highest wear values.

It should be borne in mind that the main objective of this laboratory investigation was to evaluate the mechanical properties of dental cements mixed in erroneous ratios; thus, we cannot exclude the possibility that cements with a powder content that is 50% higher than recommended may cause difficulties with the cementation of dental restorations.

From a clinical perspective, carboxylate cements, which have narrowly restricted powder/liquid ratios, are more likely to be mixed incorrectly than are zinc phosphate or glass-ionomer cements. Our results show that, in carboxylate cements, excessive liquid content is worse than too much powder, and that hardness is more seriously affected by mixing errors than is three-body abrasion. Thus, we conclude that carboxylate cements should always be mixed with the careful use of proportioning aids.

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