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ORIGINAL ARTICLE

Effect of variations from the recommended powder/liquid ratio on some properties of resin-modified cements

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Abstract

Objectives. The powder and liquid contents of cements are mixed in accordance with the recommended mixing ratio, but discrepancies occur despite the use of proportioning scoops. Little is known about powder/liquid ratio variations on certain properties of resin-modified cements. ***Methods***. Two resin-modified glass ionomer cements (RMGIC) were mixed using various powder/liquid ratios: (a) Recommended ratio: Fuji Plus: powder/liquid 2:1; ProTecCEM p/l 2.25:1. (b) Maximal variation arising using proportioning aids (17% liquid surplus): Fuji Plus 1.66:1, ProTecCEM 1.875:1. Limits of mixing and specimen construction: either (c) both groups with more liquid (1.5:1) or (d) more powder (3:1). Flexural strength was determined using a 3-point bending test (after 24 h) and wear using a 3-body abrasion device. The extent of cure reaction was characterized using differential thermal analysis (DTA). ***Results***. While higher powder content did not significantly affect the flexural strength of Fuji Plus and ProTecCEM, it considerably reduced wear of Fuji Plus. Increasing liquid content reduced flexural strength. A substantial increase in wear for Fuji Plus 1.5:1, and ProTecCEM 1.875:1 and 1.5:1 mixtures was observed. DTA demonstrated that a higher liquid content resulted in incomplete setting reactions, which could be detected even after 24 h of cure. ***Conclusions***. If RMGICs are mixed with powder/liquid variations, given the inaccuracy of proportioning aids the properties of RMGIC will change slightly and may be disregarded. If set with higher powder/liquid variations, a surplus of powder has less influence on the properties than a surplus of liquid.

Key Words: *Differential thermal analysis, flexural modulus, flexural strength, powder liquid ratio, resin modified glass ionomer, wear*

Introduction

Luting cements must withstand masticatory stress for many years in an oral environment. Maintenance of integrity while transferring stress from a crown to dentin or enamel is essential. Stress deforms the cement body. It ranges from elastic to permanent plastic deformation and depends on the cement's mechanical properties. Such stressed cement can fail by micro-cracks and subsequent bacterial ingress or by gross failure with crown dislodgement [1,2]. Dentists are therefore challenged with "ideal cement" selection for their clinical cases.

Although there are ample strength tests, it has not been identified which *in vitro* tests can be considered to have clinical validity [3,4]. Some guidelines suggest that investigations of flexural strength or flexural modulus are steps toward a correlation of mechanical properties and clinical performance [1]. Prosser et al. [5] have reported that the most appropriate measurement of glass ionomer cements (GICs) is obtained with a flexural strength test. In the discussion on the clinical validity of cement strength tests in the literature, Fleming et al. [6] emphasize that, in the past, cement properties were determined by test results achieved under ideal mixing conditions of the cement components, e.g. powder/liquid. In a clinical test in which 40 dental nurses mixed zinc phosphate cement, a wide range of compressive strength values was found. They stated that 25% of the cement mixes resulted in strength values below 40 MPa, and less than 60% achieved the required specification standard compressive strength value of 70 MPa. Although an accurate mixture of cement is essential, disparities occur, and are described for zinc oxide phosphate or glass ionomer cements [6]. These mixing variations occur as a result of the traditional hand-mixing of cements, a habit that is still extensively in use [6,7]. Compared

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to encapsulated systems, hand mixing is cheaper and does not require mixing devices, but there are some disadvantages: (i) The powder is relatively inaccurately proportioned by the user himself with the aid of scoops. (ii) The dispensed powder volume depends on the scoop filling method and on the liquid volume on the bottle inclination during dispensing respective to the construction of the bottle outlet [6]. This problem has been evident in dental practices over a long period of time [7]. However, few investigations have been published concerning the effect of not-recommended mixing ratios on mechanical properties or possible increased wear [8].

In addition, with mechanical properties such as represented by flexural strength tests, the clinical behavior of luting cements is reflected in marginal wear. It has been clinically observed that cements lose substance after several years of oral service [2]. This wear depends on the cement type [1,9], the curing procedure [9], and the timing of finishing and polishing [10].

Two types of wear can occur: two-body or three-body abrasion [11]. Two-body wear is the result of tooth-to-tooth contact. Three-body wear is characterized by an abrasive medium such as food or toothpaste, and is the dominant wear factor in the marginal area. DeGee [12] described a method that investigates three-body abrasion. This test cannot directly represent the clinical situation, but ranks dental luting cements according to their three-body abrasion behavior.

The aim of this study was to investigate the influence of incongruent mixing ratios on the flexural strength, flexural modulus, and three-body wear of resin-modified glass ionomers (RMGICs). Although this cement type is popular, only a few investigations [8] have addressed the question as to whether these cements have a wide therapeutic range or not. The hypothesis stated is that variations from the ideal mixing ratio increase the wear and decrease the flexural modulus of RMGIC and that even low-grade variations caused by the inaccuracy of proportioning aids have an influence.

Material and methods

The RMGICs Fuji Plus (GC, Tokyo, J) and ProTecCEM (Ivoclar-Vivadent, Schaan, FL) were mixed using different powder/liquid ratios (for composition, see Table I). With a chemical balance (Sartorius, Göttingen, G), powder and liquid were weighted with a tolerance limit of ± 0.1 mg. The recommended powder/liquid ratio was 2:1 for Fuji Plus and 2.25:1 for ProTecCEM. These ratios should be achieved using proportioning scoops included in the cements kits. However, measurements with the chemical balance demonstrated that even careful use of the proportioning scoops and bottles resulted in variations. The lowest and the highest measured weight of powder proportioned using the aids showed a variation of about 17% (25.234 – 30.498 mg). Clinical observation revealed a tendency to add more liquid than powder to the cement mix [6]. Therefore, groups were assigned for this investigation with the recommended powder/liquid ratio, including a surplus of liquid for Fuji Plus of 1.66:1, ProTecCEM 1.875:1 (17%, respectively, low-grade variations) and with a powder/liquid ratio of 1.5:1 (both cements). In order to investigate the limits of possible powder/liquid ratios, two high-grade variations were selected: (i) liquid surplus 1.5:1, (ii) powder surplus 3:1.

All cement mixes were carried out on a glass slab (20 s) and the samples then stored in an (dark) incubator at 37°C for 3 min, and 24 h, respectively.

Table I. Mixing ratios used in the investigation and cement composition

	Fuji Plus	ProTecCEM
Powder/liquid recommended	2:1	2.25:1
More liquid (17%)	1.66:1	1.875:1
More liquid	1.5:1	1.5:1
More powder	3:1	3:1
Fuji Plus		
Liquid	2-Hydroxyethyl-methacrylate Polyacrylic acid Tri-ethylen-glycol-dimethacrylate	
Powder	Aluminum silicate glass	
ProTecCEM		
Liquid	Hydroxyethyl-methacrylate Distilled water Di-methacrylate Methacrylate-modfied polyacrylic acid	
Powder	Ba-Al-Fluorosilicate glass Ytterbium-tri-fluoride SiO_2	

Differential thermal analysis (DTA)

The samples were subjected after 3 min and 24 h of curing time, respectively, to a dynamic temperature program using the thermal analytical unit DSC Plus (Rheometric Scientific, Surrey, UK) [13,14]. All investigations were carried out in open aluminum crucibles with a constant air flow of 5 ml/min. The DTA furnace was common to sample and reference. Samples and reference were cooled down to −100°C using liquid nitrogen and then heated at a rate of 10 K/min up to 300°C, at which temperature thermal degradation of the resins began. The DTA instrument signal was derived from the temperature difference between the sample and reference. If heat was released inside the sample (exothermic reaction), this was indicated by a peak plotted "upwards" (positive direction on the y-axis). A second heating was carried out to compare complete and non-complete curing reactions. As a further control, the powder and the liquid of both cements were investigated separately.

Flexural strength, flexural modulus

Rectangular beams of 25 mm length, 2 mm height, and 2 mm width were constructed in silicone molds using the above-mentioned blend ratios of powder and liquid. Eight specimens were constructed per group. Cement mixing and application into the molds took place at room temperature. The cement surplus was removed, the specimens were covered with a polyethylene foil, and the mold was closed with a glass slab. Care was taken that the beams did not show entrapped air or surface flaws. The specimens were allowed to set in a dark incubator at a temperature of 37°C. Owing to the slow setting reaction of the cements, it was not possible to use beams for the flexural test after 3 min of curing. There was a large sticky mass surrounding the beams or they did not withstand the force necessary for removing them from the mold. Thus, investigation of flexural strength could be carried out only for beams after 24 h.

After 24 h of storage, all beams were loaded to failure using a 3-point bending test. The support distance was 20 mm. The load was applied using a Zwick universal testing machine 1446 (Zwick, Ulm, G). Cross-head speed was 1 mm/min. Flexural strength [σ_s] of the beams was calculated using the formula [15]:

$$\sigma_s = \frac{3\,F\,l}{2\,b\,h^2} \qquad F = force,\ l = length,\ h = height,\ b = width$$

The flexural modulus E_f was calculated as follows:

$$E_f = \frac{F\,L^3}{4\,b\,h^3\sigma_s} \qquad F = force,\ L = support\ length,\ b = width,\ h = height,\ \sigma_s = flexural\ strength$$

Three-body abrasion test

A sample wheel with 12 individual chambers for specimens was chosen in accordance with the method described by DeGee [12]. Three samples of the (four) blend ratios were randomly fixed on the sample wheel. Only samples after 24 h storage were chosen. A test after 3 min could not be carried out, because it was impossible to construct and apply specimens for 12 chambers during 3 min.

The specimens were fixed on the sample wheel with adhesive. 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) which moved under a pressure of 15 N counterclockwise [12]. The abrasion medium was a mixture of millet seed shells (30 g) and rice (120 g), which were ground in a rotating blade grinder for 60 s (Moulinette, Moulinex, Paris, F). Rice and seed shells were mixed with 275 ml distilled water and allowed to swell for 1 h.

The smaller antagonist caused a 6-mm-wide trace in the specimens on the sample wheel. The unworn area served as the control. Compared to the control, the wear track was measured after 50,000 and 100,000 cycles using a profilometer testing device (Perthometer S6P, Perthen-Feinprüf, Göttingen, G) [12].

Statistics

Mean and standard deviation were calculated and one-way ANOVA was used to investigate statistical differences. The level of significance was set at $\alpha = 0.05$.

Results

DTA ProTecCEM

The DTA curves of mixtures with the recommended ratio did not show peaks either after 3 min or 24 h of cure (Figure 1). Mixtures with a liquid ratio of 1.5:1 and 1.875:1 had 3 exothermal peaks after 3 min of cure. The peaks shifted in the investigation 24 h later to higher temperatures and to lower heat flow levels (Figure 2). The 1.5:1 ratio showed the highest exothermal peaks. These were present after 3 min setting time as well after 24 h storage in the incubator, while the 1.875:1 ratio had 3 peaks only after 3 min. After 24 h of cure, only one peak at 185°C was clearly detectable. With a powder surplus (3:1 p/l), the DTA curve had a peak at 185°C which was reduced but detectable after 24 h.

DTA Fuji Plus

The recommended 2:1 blend ratio did not show exothermal peaks after 3 min (Figure 3) nor after 24 h of cure. However, all divergent blend ratios had 2

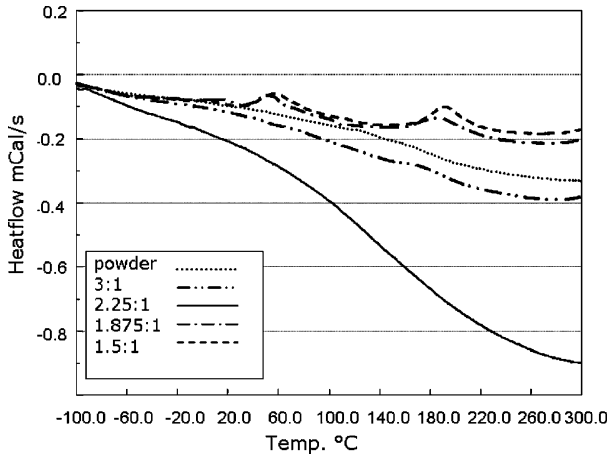

Figure 1. DTA curves of ProTecCEM with various mixing ratios. The DTA instrument signal was derived from the temperature difference between the sample and reference. If heat was released inside the sample (exothermic reaction), it was indicated by a peak plotted "upwards" (positive direction on the y-axis).

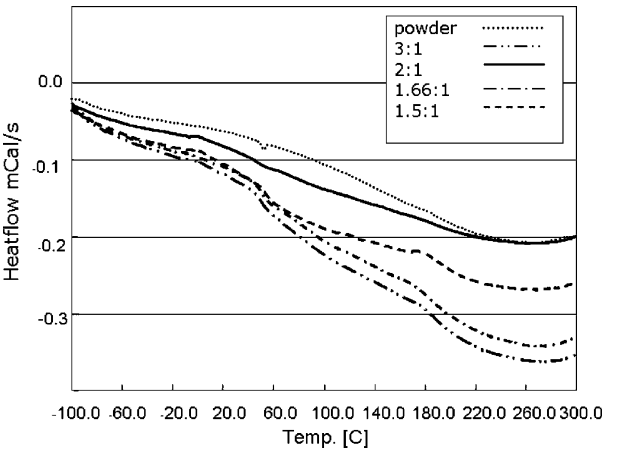

Figure 3. DTA curves of Fuji Plus with various mixing ratios. The DTA instrument signal was derived from the temperature difference between the sample and reference. If heat was released inside the sample (exothermic reaction), it was indicated by a peak plotted "upwards" (positive direction on the y-axis).

peaks after 3 min of cure. Their peak levels lowered for specimens after 24 h of storage in the incubator (Figure 4). The blend ratio with the highest liquid content had the highest exothermal peaks. In contrast to ProTecCEM, the peaks of the blends reached lower levels with higher liquid ratio. However, Fuji Plus mixed with more powder content showed higher peaks than ProTecCEM.

Flexural strength, flexural modulus

Compared to the recommended powder/liquid ratio, higher powder content increased while higher liquid content reduced the flexural strength and modulus of both investigated cements (Table II). However, the mean differences were not statistically different at a level of $\alpha = 0.05$. Owing to the incomplete setting of ProTecCEM with a p/l ratio of 1.5:1, specimens could not be constructed. The beams could not be removed from the mold with subsequent damage. Statistically significant mean differences were only found in the comparison between the blend ratios with highest (1.5:1, 1.875:1) and lowest (3:1) liquid content ($p < 0.05$).

Three-body wear ProTecCEM

The wear rate of the samples depended on the powder/liquid content after 50,000 cycles in the abrasion device (Table III). The higher the liquid content the more increased the wear. The means differed statistically ($p = 0.001$) with the exception of the means between the ratios of 1.875:1 and 1.5:1 and 3:1 and 2.25:1 of powder/liquid, respectively. The highest wear resistance was found for ProTec-

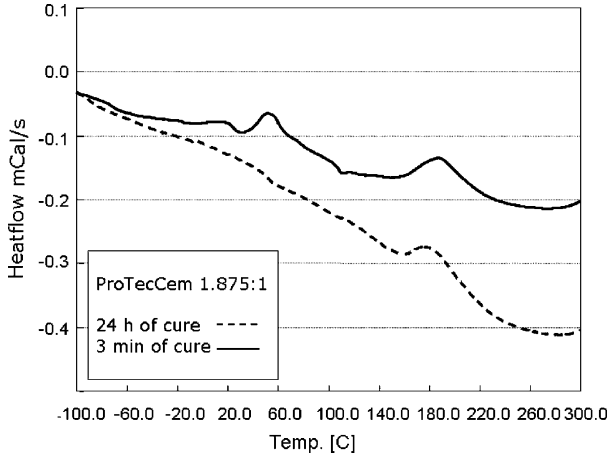

Figure 2. DTA curves of ProTecCEM with 1.875:1 mixing ratio after 3 min and after 24 h of cure. The DTA instrument signal was derived from the temperature difference between the sample and reference. If heat was released inside the sample (exothermic reaction), it was indicated by a peak plotted "upwards" (positive direction on the y-axis)

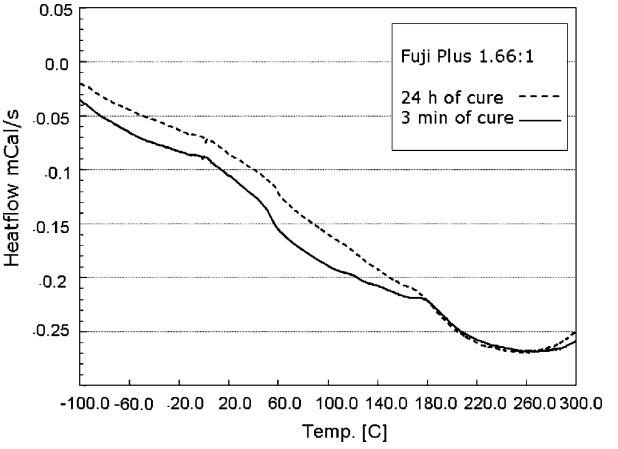

Figure 4. DTA curves Fuji Plus with 1.66:1 mixing ratio after 3 min and after 24 h or cure. The DTA instrument signal was derived from the temperature difference between the sample and reference. If heat was released inside the sample (exothermic reaction), it was indicated by a peak plotted "upwards" (positive direction on the y-axis).

Table II. Flexural strength and flexural modulus of resin-modified glass ionomers with different mixing ratios (mean and standard deviation are depicted)

Fuji Plus powder/ liquid	Flexural strength [N/mm^2]	Flexural modulus [N/mm^2]	ProTecCEM powder/ liquid	Flexural strength [N/mm^2]	Flexural modulus [N/mm^2]
3:1	53.8±4.9	2506±96	**3:1**	55.2±7.8	1789±94
2:1	52.9±7.8	2925±148	**2.25:1**	48.0±8.8	2399±201
1.66:1	47.3±5.3	2570±154	**1.875:1**	42.9±3.5	3048±229
1.5:1	41.7±8.5	3487±270	**1.5:1**	–	

CEM with the highest powder content. With increasing cycles (100,000), the wear increased significantly ($p=0.001$). However, the ranking of the blend ratios did not change with the cycles. The lowest wear resistance was demonstrated by the recommended blend ratio.

Three-body wear Fuji Plus

The wear resistance of Fuji Plus mixed with a higher liquid content of 1.66:1 p/l was lower than the recommended 2:1 blend ratio (Table III). The mean values did not differ significantly. A higher liquid content of 1.5:1 increased the wear ($p=0.14$), while higher powder content reduced wear considerably ($p=0.001$). The ranking between the blend ratio groups did not change after 100,000, but a notable increase in wear was observed ($p=0.001$). The highest wear resistance showed the blend with the highest powder content.

Discussion

Only a few investigations have been carried out on the physical properties of glass ionomers or resin-modified glass ionomers when the powder/liquid ratio has been changed [7,8]. Generally, it is expected that the cement properties decrease depending on the reduction of the powder volume. Mitsuhashi and co-workers found that the fracture toughness of glass ionomer was significantly reduced by lower powder content, while the fracture toughness of resin-modified glass ionomers was unaffected [8].

A similar result was shown for the flexural strength and modulus in this investigation of resin-modified glass ionomers. Lower powder content reduced the flexural modulus or strength. However, the changes did not differ statistically. In this respect, both tested cements seem to have a wide therapeutic range. The importance in clinical use is due to the fact that mixing the recommended ratio can entice inexperienced users to add more liquid than necessary. An attribute of these cements is the initial deceptive lack of liquid. However, with increasing mixing intensity the cement adapts the fluid consistency more and more, which allows crown setting. A further mixing error can occur using the proportioning aids. Our pre-investigations showed that, despite their careful use, the variations reached values of 17%. However, both cements mixed with 17% more liquid (1.66:1 Fuji Plus: 1.875:1 ProTecCEM) had no significant influence on flexural strength or modulus. The hypothesis can therefore be rejected that powder/liquid variations given by the inaccuracy of proportioning aids change the properties of RMGIC.

However, greater variations may influence the cement properties. Higher powder content increased the strength values. During bending, more filler particles are in contact with and support each other. The mechanism is well known for high-filled composites [16].

Even though the flexural strength and modulus seem not to be significantly affected by higher liquid content, the DTA curves demonstrated that the setting reactions were not entirely complete (Figure 1). Exothermal peaks were shown for mixtures with higher liquid content. These peaks became larger with increasing liquid content, but they were reduced after 24 h (Figure 2). It can be expected that with time the mechanical properties of these resin-modified glass ionomers may approximate those of the correct mixtures. However, the DTA curves measured after 3 min of cure indicate the possible risk of micro-cracks and subsequent bacterial ingress or gross failure with crown dislodgement due to incomplete setting reactions during the first hours in the oral cavity. Nothing is known as to whether long-term clinical failures of resin-GIC cemented restora-

Table III. Three-body abrasion of resin-modified glass ionomers with different mixing ratios (mean and standard deviation are depicted)

Fuji Plus powder/ liquid	Wear [γm] 5*10^{5}cycle	Track 10^6 cycle	ProTecCEM powder liquid	Wear- [γm] 5*10^{5}cycle	Track 10^6 cycle
3:1	92±4	135±10	**3:1**	98±6	158±16
2:1	128±7	195±11	**2.25:1**	105±8	156±12
1.66:1	114±8	169±9	**1.875:1**	124±10	184±23
1.5:1	144±9	215±4	**1.5:1**	128±12	201±19

tions are initiated by initial loss of cement integrity, or whether an incomplete setting reaction influences the biological properties of cements in the long term. It has been demonstrated that unconverted monomers can be released from resins into an adjacent aqueous phase and therefore enter the human body [17]. Both cements contain hydroxy-ethylene-methacrylate (HEMA), Fuji Plus includes tri-ethylenglycol-dimethacrylate (TEGDMA) and ProTec-CEM di-methacrylates. Although this study did not investigate the content of residual monomers of cement mixtures after 3 min and 24 h of cure, respectively, the peaks of DTA curves indicate that reactable groups are present. It is reported that HEMA or TEGDMA, in particular, can cause the oral gingiva to produce an adverse reaction or induce allergic reactions [18,19].

A 3-point bending test of dental cement beams is a common way to obtain design data [15] and can provide information about material properties. In isotropic material, the results of the 3-point bending test follow the simple beam theory [20], which is not true for anisotropic materials such as fiber-reinforced composites. Resin-reinforced glass ionomers display an isotropic behavior. Some guidelines suggest that investigations of flexural strength or flexural modulus are a step toward correlation of mechanical properties with clinical performance [1,4]. However, it has to be taken into account that the simple 3-point bending test cannot reflect the complex clinical situation; for example, an all-ceramic crown in the oral cavity cemented with a RMGIC. Instead, this test can estimate and compare the load bearing capacity of different materials under flexure and differing mixing ratios [15]. Furthermore, it can offer information about the parameters that should be chosen for an *in vitro* test of different cemented crowns in an artificial masticator.

Wear of dental materials occurs as three-body abrasion or attrition [11]. Three-body abrasion takes place if toothpaste or a food bolus is moved between two surfaces [21]. Attrition occurs between two occlusal surfaces when, for example, a tooth cusp is moved under load in an occlusal fossa. The cement layer at the crown margin can be loaded directly by a tooth. Most frequently, three-body abrasion takes place. Therefore, the three-body abrasion test was chosen for this investigation. Limitations of this method have been described by DeGee [12].

As expected, wear increases with liquid content. This follows the theory that fillers (powder) protect the cement matrix from abrasion [22]. Similarly to filling composites, higher powder content reduces wear. A further reason could be that the mixtures with higher liquid had no finished setting reaction even after 24 h, which is indicated by the DTA curves with exothermal peaks. Irie and coworkers presumed that the complete setting reaction of resin-GIC lasted about 24 h. They recommend a delayed polishing procedure to avoid marginal gaps [10].

Blends with a higher liquid level seem to need considerably more setting time. The three-body abrasion test was performed after 24 h and the specimens were worn because of the retarded and therefore incomplete setting reaction. The problem with this study lies in the fact that the results cannot be directly transferred to the clinical situation. There are no long-term clinical reports available dealing with cementation-caused failures and the question arises whether unrecommended ratios could be of clinical importance. The results indicate that mechanical properties such as flexural strength or modulus are not particularly affected by adverse mixing ratios. However, the three-body abrasion test showed statistically significant increased wear rates, which may have had a clinical effect. Therefore, a further investigation is suggested using a chewing simulator, where crowns have to be cemented with correct and adverse mixing ratios. After simulation of 5 years' oral service, the marginal integrity and a final load to fracture test may demonstrate possible clinical effects of incorrectly mixed cement ratios.

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