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Wear Resistance of Luting Cements and the Influence of Marginal Gap Width on Substance Loss

Rita Trumpaite-Vanagiene

SUMMARY

Purpose: Scientific data on wear from toothbrushing of luting cements for fixed partial dentures (FPDs) are limited and inconsistent, therefore the study aimed: (1) to investigate the effect of marginal gap width on cement wear and (2) to compare the resistance to wear from toothbrushing of five luting materials in an *in vitro* model. **Materials and Methods:** one zinc phosphate cement (ZPC), glass-ionomer cement (GIC), resin-modified glass ionomer-cement (RGIC), compomer cement (CC) and resin cement (RC) were tested in a toothbrushing device for 2 hours (18,000 double strokes) using a standard tooth paste and a crown-cement-tooth model simulating the cervical region of a crowned tooth. Two gap widths, i.e. 100 and 250 μm were used. Tested surfaces were replicated and wear measured in a scanning electron microscope at three locations: at the metal/cement interface, the middle of the cement film and the cement/dentin interface. Wear of cements were compared by one way analysis of variance (ANOVA) followed by Bonferroni multiple comparison tests. Two-sample *t* test was used for assessing the effect of marginal gap width on each product separately. **Results and Conclusion:** a tendency of reduced wear of water-based cements at the wider gaps was observed. Statistically significant differences in wear resistance were found between the cements at all three measurement places ($P < 0.001$), i.e. ZPC and GIC were found to have the least, CC and RC the highest and RGIC an intermediate resistance to wear.

Key words: dental materials, wear resistance, luting cements, marginal gap.

INTRODUCTION

Dental caries at the cervical junction between a restoration and a tooth has been reported as the major reason for the failure of crowns and fixed partial dentures (FPDs) [1-7]. Another reported less frequent biological reason for failure of FPDs, also related to the cervical region, is periodontal complications [2, 4-7]. The transition between a full prosthesis and a tooth as a potential plaque-retaining area is, therefore, considered being of significant clinical importance for the long-term success of fixed dental prostheses.

A narrow film of luting cement, together with a restoration margin, is responsible for an uninterrupted, smooth transition between the prosthesis and the tooth. Dental luting cements, inevitably exposed to the oral environment along the crown margin, are susceptible to dissolution and wear. Gaps or niches created by wear may become a potential plaque trap and, thus, may predispose to caries and periodontal disease. The cement, therefore, should have an adequate resistance to wear in order to maintain the marginal integrity.

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Various types of luting materials are available for the permanent cementation of crowns and fixed partial dentures. These include zinc phosphate, glass-ionomer, resin-modified glass ionomer, compomer and resin luting cements. Previous studies of cement wear have been limited to resin cements [8-11] and have focused on occlusal wear applicable to luting materials sealing intra-coronal prosthetic restorations (inlays). The wear of cement films exposed at the cervical area, however, is determined by other wear mechanisms with toothbrushing being the predominant one. The research into wear from toothbrushing of luting cements exposed along the crown margin is scarce.

The width of the cement film along the margin is determined by the fit (the size of marginal discrepancy or gap) of the cemented cast restoration and may vary considerably. Fransson et al [12] reported a mean marginal gap for metal-ceramic crowns of 100 μm and a large variation with several measurements above 200 μm. The marginal discrepancies for crowns and FPDs from 5 to 430 μm with a mean of 160 μm have been measured in another in vivo study [13].

The rate of occlusal wear of luting cements has been empirically related to the marginal gap size, i.e. to an area of the exposed cement [8, 14, 15]. A reported relationship between cement wear and gap size expressed by a correlation coefficient varied considerably (in the range $r = 0.3$ to $r > 0.9$). Additionally, no clear pattern of cement disintegration in relation to the gap size could be elucidated [16]. Thus, there is a need to further investigate the influence of the size of the exposed cement area on substance loss. The present study aimed:

1) to investigate the effect of marginal gap width on the wear of luting cements in an in vitro simulated crown-cement-tooth model;

2) to compare the resistance to wear from toothbrushing of five different types of luting cements.

Table 1. *Tested luting cements.*

Luting cement type	Cement code	Product name	Manufacturer	Batch no.
Zinc phosphate	**ZPC**	DeTrey® Zinc Crown & Bridge Fixodont® Plus	Densply De Trey GmbH	9802001013
Glass ionomer	**GIC**	GC Fuji I	GC Corporation	050871
Resin-modified glass ionomer	**RGIC**	GC Fuji PLUS	GC Corporation	171271
Compomer	**CC**	Dyract® Cem	Densply De Trey GmbH	9804001534
Adhesive resin cement	**RC**	Panavia®21	Kuraray Co.,Ltd.	41225

MATERIALS AND METHODS

The materials tested in the present study were selected to represent the wide range of commercially available types of luting cements with the intention of assessing a systematic pattern of wear of different cement types. Specifications of luting cements tested under in vitro experimental conditions are listed in Table 1.

Specimen Design

A specimen simulating the cervical region of a crown-cement-tooth construction was designed by cementing restorative material to a tooth structure. A standard palladium-silver ceramic alloy (Will-Ceram® W-1, Williams®, batch no. $$785740L, Ivoclar North America, New York, US) used as the restorative material was cemented to bovine dentin serving as the tooth structure.

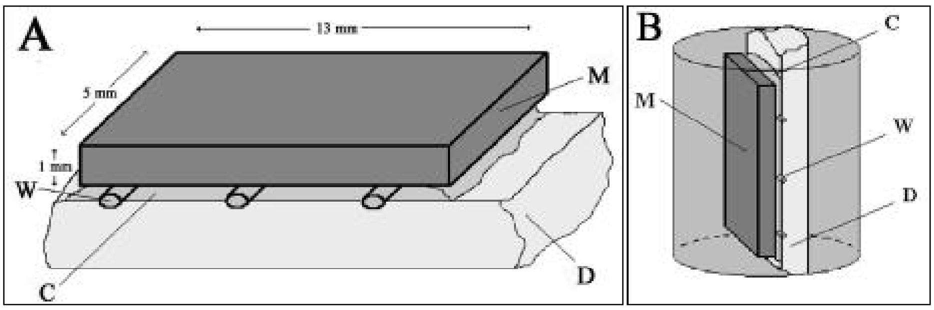
Plates of the alloy (13 × 5 × 1 mm) were planed and polished by wet grinding on 500-grit silicon-carbide abrasive paper (on a rotating wheel) (Waterproof Silicon Carbide Paper, Struers, Denmark). The flatness of the surface was controlled using a profile projector (Model 6C-2, Nikon, Nippon-Kogaku, Japan).

Fifty sound bovine front teeth were extracted. Pulp tissue and remaining periodontal ligament were immediately removed. Until use the teeth were refrigerated in 0.9 % sodium chloride solution. Blocks with two parallel walls (~ 15 × 5 × 3 mm) were cut out from the teeth using a rotating diamond saw under water cooling in a laboratory cutting machine (Accutom, Struers, Denmark) and flattened by wet grinding on silicon-carbide abrasive paper (500-grit). Care was taken to ensure that the entire surface facing the luting cement consisted of dentin.

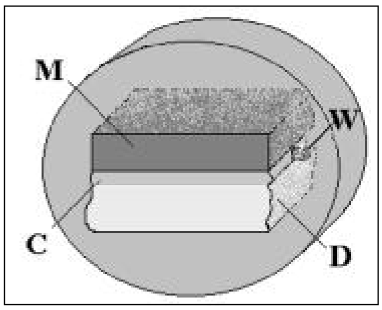
The cements were handled and proportioned according to manufacturers' instructions in a climate-conditioned room (23 ± 1)° C and relative humidity of (50 ± 5) %. In order to achieve the recommended powder/liquid ratio powder and liquid components were weighed for each cement mix. The cements were mixed manually.

The two gap distances selected for the study were 100 µm and 250 µm. The cement was placed between the restorative material and dentin plates separated from each other by stainless steel wires of the predetermined diameter (100 and 250 µm) serving as space holders to control the gap width between the metal plate and the dentin (Picture 1A). While setting the load of 2 kilograms was applied on the top of the metal plate. After setting the dentin, cement and restorative material assembly was placed in distilled water at 37°C for two hours. Thereafter it was embedded in a cold curing epoxy resin (Epofix, Struers, Denmark) (Picture 1B). The mounted block was then cut into three sections with a diamond saw under running water and three operational specimens were obtained (Picture 2).

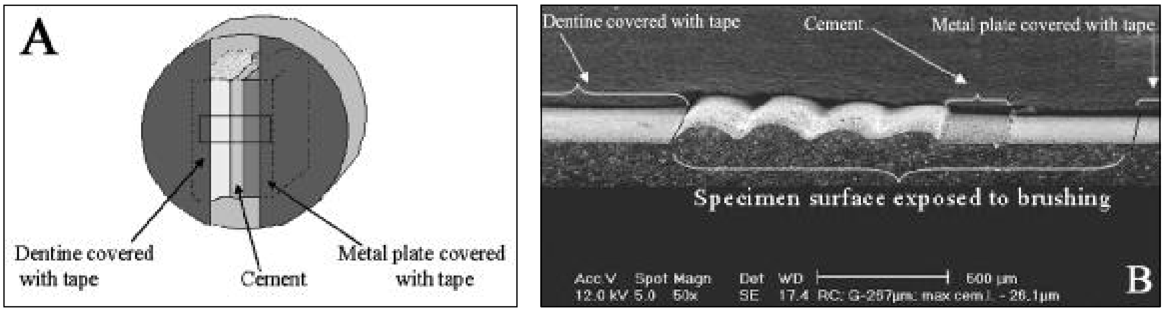
Test surfaces of each specimen were planed gradually by wet grinding on 500, 1000 and 4000-grit carborundum paper in order to create a flat and smooth surface. The surface quality was evaluated using an optical microscope. The marginal gap width was controlled using the profile projector at 50 × magnification at three points, i.e. at each end and in the middle of the



Picture 1. *Preparation of specimen: A - Stage 1, B - Stage 2 (M – metal plate, C – luting cement, D – dentin, W – spacer (wire of certain diameter).*



Picture 2. *Schematic representation of specimen (M – metal plate, C – luting cement, D – dentin, W – spacer (wire of certain diameter).*



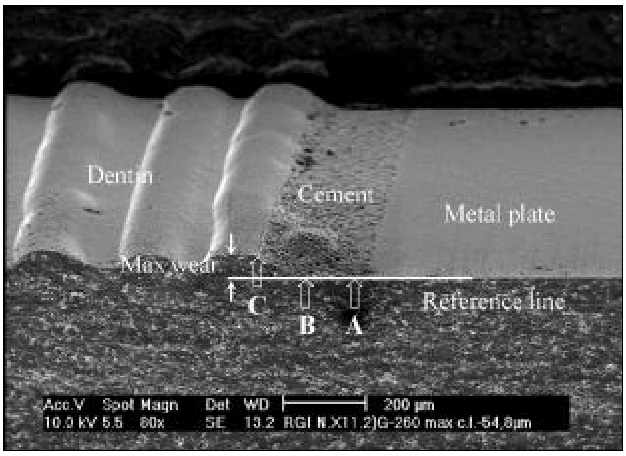
Picture 3. *A. Schematic representation of specimen covered with an adhesive tape.*
B. SEM micrograph of one section of a negative replica illustrating an exposed test track.

specimen. Only the specimens with gap distances within the range of 100 ± 25 µm and 250 ± 25 µm were accepted. The specimens were stored in distilled water at 37°C for two weeks prior to the wear testing. Sixty operational specimens (12 specimens for each cement, 6 for each gap distance) were obtained. In order to obtain a reference surface and a distinct outline of the abraded surface structure, part of both the metal plate and the dentine were covered with a resin adhesive tape (about 50 µm thick), exposing a 2 to 3-mm wide test track (Picture 3).

Wear Test

A toothbrushing wear test simulating the three-body type of wear (expected in the mouth) was selected for the present study. The specimens were tested in an automatic tooth-brushing machine (analogous to that used in the British Standards technique for the abrasivity studies of toothpastes) [17] using flat profile toothbrushes with medium bristle stiffness (Jordan*Classic, Jordan, Oslo, Norway). Bristle diameters were of ~ 200 µm, ends of bristles were rounded. The toothbrushes were changed after brushing of three specimens, i.e. in each experimental group, consisting of six specimens of the same cement with a certain gap width, two specimens were brushed with the toothbrushes used for the first time, two with the brushes used for the second, and two with the toothbrushes used for the third time.

Each specimen was fixed in a hole in the bottom of a trough. The top surface of the specimen was levelled with the bottom surface of the trough. The brushing "surface" of the toothbrush head was adjusted parallel to the test surface to ensure full contact over the total length of the head. The toothbrush rested on the test surface with a load of 2 N. The direction of the reciprocating brushing motions (150 cycles/min) was parallel to the dentin-cement-restorative interface. In order to obtain observable wear, two hours of continuous brushing corresponding to 18000 double strokes were employed in the study. This represents about three and a half years of brushing twice daily according to our calculations based on the estimated average toothbrushing time (60 s) [18], the rate of brushing (4,6 strokes/s) [19], the length of toothbrush head (30 mm) and the average length of a brushing area (calculated on the average mesiodistal diameters of permanent teeth) [20]. The specimens were brushed with a slurry of the standard reference paste (BS 5136 : 1981) [17]. The slurry was prepared by mixing 40 ml distilled water with 25 g reference paste and was renewed for each specimen.



Picture 4. *An example of a negative replica. The thick arrows indicate the three measurement points (A, B and C).*

Wear Measurements

The specimens were examined by means of a replica technique [21] in a scanning electron microscope (SEM) (Philips XL 30, Philips Electron Optics, Eindhoven, The Netherlands). After brushing the covering tapes were removed and the test surface was replicated with an elastomeric impression material (Permadyne® Garant 2:1, ESPE, Seefeld, Germany). The impressions were viewed directly. The reliability and accuracy of the replicating technique were verified by comparing a scanning electron microscopic image of the impression of a standard ruled test block [22] (ISO 4823:1992(E)) with the actual structure of the block. Reproducibility (reliability of the method) was considered to be acceptable, since the accurate duplication of the position, size and shape of the fine lines (20 ± 4 µm, 50 ± 8 µm and 75 ± 8 µm) was obtained.

In order to cover possible variations in the toothbrushing effect due to differences in the quality of luting material and/or dentin, wear was measured at three locations along the cement film in each specimen. Each negative replica was thus cut at three different places transversely to the direction of the luting cement interface, and three different slices of each replica were then obtained for wear measurements (Picture 3B). Each section was mounted and coated with 20 nm of a gold palladium alloy (SCD 050 Sputter Coater, Balzers AG, Balzers, Liechtenstein) for SEM inspection and measurements. The wear (cement loss in µm) was defined as the vertical difference between the abraded cement surface and the non-affected metal plate used as a reference line.

A pilot study showed that wear of luting cements was not constant within the marginal gap film. In order to obtain a more comprehensive picture, wear was measured at three points across the cement film, specifically at the metal/cement interface (measurement point A), in the middle of the cement film (measurement point B) and at the cement/dentine interface (measurement point C) (Picture 4). All measurements were conducted by the author at up to 300x magnification and 10° tilt with an accuracy of ± 1 μm.

SEM images were photographed in order to illustrate the typical surface topography for each of the luting cements studied.

Statistical Analysis

A two-sample *t* test was used in order to assess the effect of marginal gap width on cement wear for each product separately. The aggregated data on wear of both the 100 and 250 μm gaps were used for the comparison of the five luting cements studied by applying a one way analysis of variance (ANOVA) followed by Bonferroni multiple comparison tests. The level of significance was set at $P < 0.05$.

Table 2. *Means and standard deviations, SD, of wear (μm) of luting cements at two different gap widths and three different measurement points (A, B, C)* [a].

Luting cement	Gap width (μm)	A		B		C	
		Mean	SD	Mean	SD	Mean	SD
ZPC							
	100	18	11	15 *[b]	9	37	11
	250	18	8	5	4	50	26
GIC							
	100	12	7	20 *	13	27 *	16
	250	9	6	9	8	48	30
RGIC							
	100	7 *	4	8 *	6	31	16
	250	4	4	3	4	29	19
CC							
	100	2	2	1 *	2	21	15
	250	3	5	0	1	26	11
RC							
	100	3 *	2	2	2	22	12
	250	7	7	1	2	19	11

[a] Measurement points: A – wear at the metal/cement interface, B – wear in the middle of the cement film, C – wear at the cement/dentin interface.

[b] Groups marked by asterisks are significantly different (P < 0.05).

RESULTS

Effect of Marginal Gap Width (Table 2)

RGIC demonstrated significantly more loss of substance at the measurement point A (metal/cement interface) at the 100 μm gap than at the gap of 250 μm ($P = 0.026$). By contrast, RC showed significantly less wear at the 100 μm gap than at the gap of 250 μm ($P = 0.04$).

An increased substance loss at the narrow gap was seen for all luting materials at the measurement point B (the middle of the cement film). The differences in wear between the two gap widths for ZPC, GIC, RGIC and CC were statistically significant ($P < 0.01$).

The observations at the measurement point C (wear at the cement/dentin interface) indicated a greater cement loss for ZPC, GIC and CC at 250 μm compared with the 100 μm gap. However, the only statistically significant difference was found for GIC ($P = 0.013$).

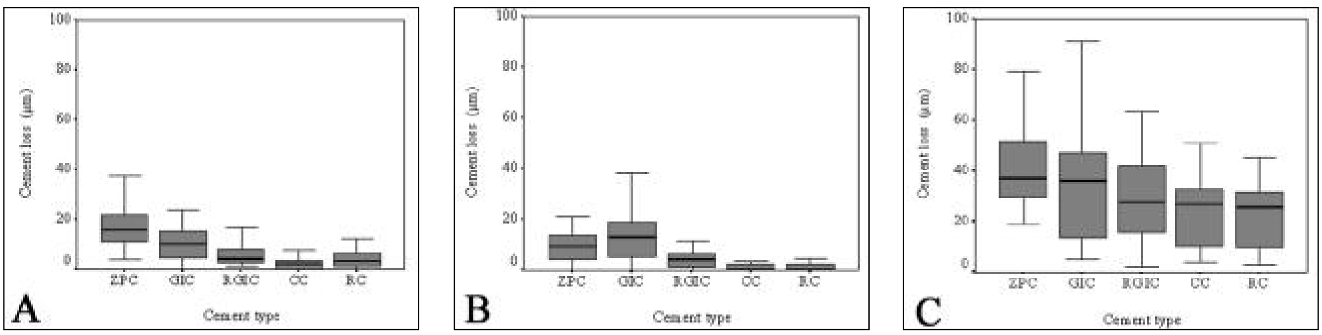
Effect of Luting Material

Pooled results from two gap widths for five luting cements are displayed in box-and-whisker plots for each measurement point separately (Picture 5 A,B and C).

The mean wear values at the metal/cement interface (measurement point A) ranged from 3 to 18 μm (Picture 5A). The largest substance loss was observed for ZPC while CC demonstrated the least wear. The analysis of variance (ANOVA) followed by multiple pairwise comparisons revealed that ZPC demonstrated significantly more loss of substance than all others, i.e. GIC, RGIC, CC and RC ($P < 0.001$). GIC showed more wear than RGIC, CC and RC ($P = 0.014$, $P < 0.001$, $P = 0.003$ respectively).

The averages for wear in the middle of the cement film (measurement point B) ranged from 1 to 14 μm (Picture 5B) . GIC was found to have the least, and CC - the highest resistance to wear. The analysis of variance (ANOVA) revealed statistically significant differences in the wear of the luting materials studied ($P < 0.001$). The wear of GIC was significantly greater than that of RGIC, CC and RC ($P < 0.001$). ZPC demonstrated more loss of substance than CC and RC ($P < 0.001$).

The largest loss of substance for all luting materials was observed at the cement/dentin interface (mea-



Picture 5. *The wear of luting cements at three different measurement points. The boxes indicate the lower and upper quartiles, the points at the end of the "whiskers" are the smallest and largest values, the central lines mark the medians.*

surement point C) (Picture 5C). The means for wear ranged from 23 to 43 μm. ZPC was found to have the least resistance to wear (mean 43 μm). RC and CC presented the highest resistance to wear exhibiting mean wear values of 22 and 24 μm respectively. Statistical analysis revealed that ZPC demonstrated significantly more substance loss than RGIC, CC and RC ($P = 0.022$, $P < 0.001$, $P < 0.001$ respectively) and GIC showed more wear than CC and RC ($P = 0.022$, $P = 0.009$).

Evaluation of Surface Topography

Representative scanning electron photographs illustrating the roughness of surfaces after brushing of the five luting cements tested are presented in Pictures 6-10. By a subjective evaluation of replicas the cements could be ranked in order of increasing surface roughness as follows: RC, CC < GIC, RGIC < ZPC.

DISCUSSION

Wear is a complex phenomenon resulting throughout the interaction of many contributing parameters, such as the properties of the opposing materials, the contact load and duration, the velocity of the movement, and the surrounding and interfacial media (saliva, food, toothpaste) [23]. All these factors vary greatly in the oral cavity. Therefore, an *in vitro* experimental design with the possibility of standardising some parameters in order to study the separate effects of other parameters was considered as optional in answering the specific research questions.

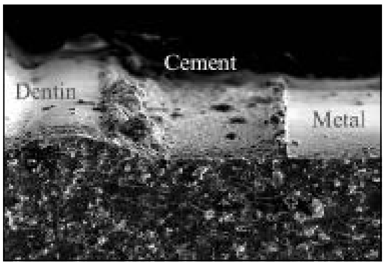
In most of the previous *in vitro* studies the wear behaviour of dental luting cements was determined with different wear testing devices simulating occlusal wear. The present study focused on cervical wear that is not influenced by occlusal forces. Although dissolution in saliva and other wear modalities might contribute to substance loss, cervical wear, especially at the facial and buccal surfaces, is predominantly caused by toothbrushing.

The choice of bovine teeth - advantageous for both availability and size - was based on the findings of *in vitro* studies justifying the use of bovine dentin as a substitute for human dentin in laboratory investigations [24 – 26].

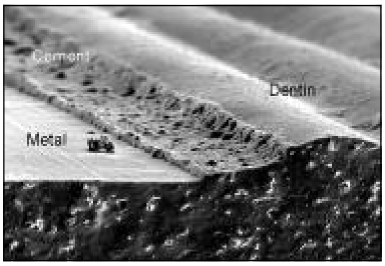
Several previous studies defined wear as an average gap depth, not specifying an exact measurement point within the gap. The present study, however, clearly indicated the need to consider wear at various places within the cement film, as the results from three measurement points demonstrated substantial differences (Picture 11).

For all the luting materials studied the greatest wear was measured at the cement/dentin interface, i.e. cement wear was strongly influenced by the less wear resistant dentin. Although it makes the interpretation of the results more complicated, several different measurement points provide a more comprehensive picture. Wear in the middle of the cement gap is likely to be the most representative for the material, since wear at the two other measurement points could be influenced by the amount of wear in the neighbouring materials, especially dentin.

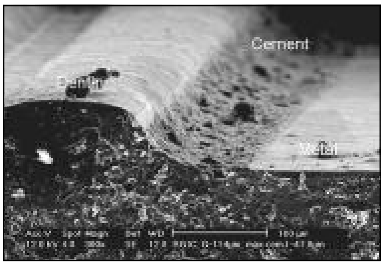
The results regarding the effect of marginal gap width on wear in luting cements were inconsistent between the three measurement points for all studied materials. A tendency towards larger substance loss, particularly for water-based cements, at narrower gaps was observed in the middle of the cement film. These unexpected findings, even being statistically significant, should be interpreted with caution as, due to the different distances from the dentin to measurement points B (50 and 125 μm), they might by caused by the wear of dentin (Picture 12). Specifically, the middle point (measurement point B) in the 100 μm gap was closer to and probably more affected by the dentin than the



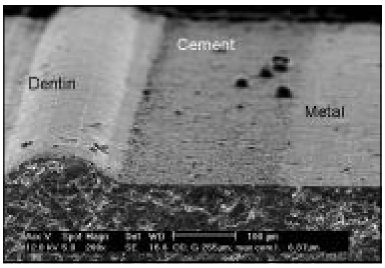
Picture 6. *Surface topography of ZPC. The negative image of the test surface after 2 hours of continuous brushing.*



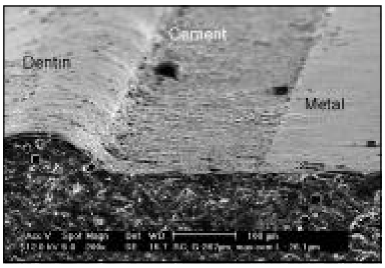
Picture 7. *Surface topography of GIC. The negative image of the test surface after 2 hours of continuous brushing.*



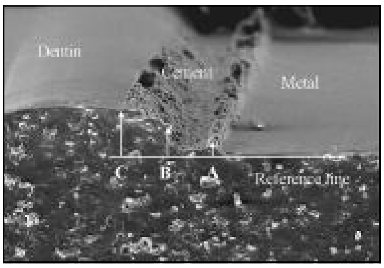
Picture 8. *Surface topography of RGIC. The negative image of the test surface after 2 hours of continuous brushing.*



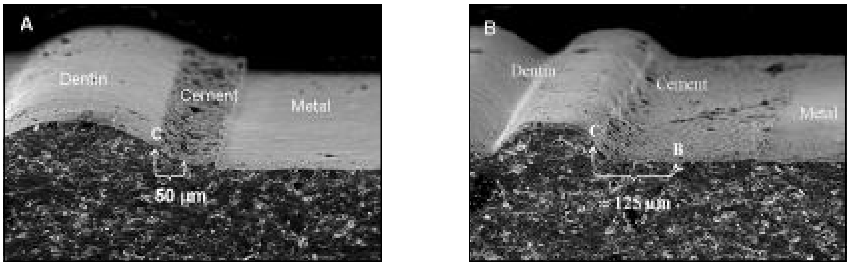
Picture 9. *Surface topography of CC. The negative image of the test surface after 2 hours of continuous brushing.*



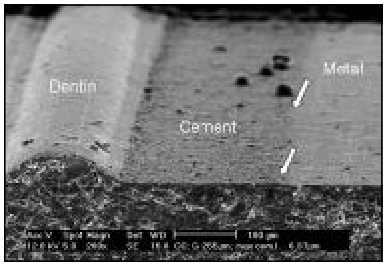
Picture 10. *Surface topography of RC. The negative image of the test surface after 2 hours of continuous brushing.*



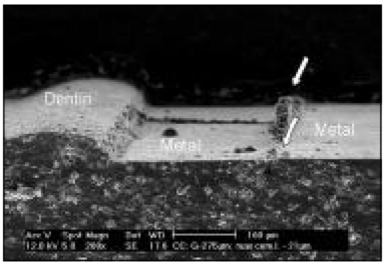
Picture 11. *Wear in the luting cement at three different measurement points (A, B, C) (negative image of the test surface).*



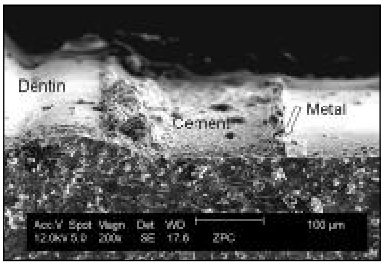
Picture 12. *The location of the middle measurement point (B) with respect to dentin in specimens with a gap of 100 µm (a) and 250 µm (b).*



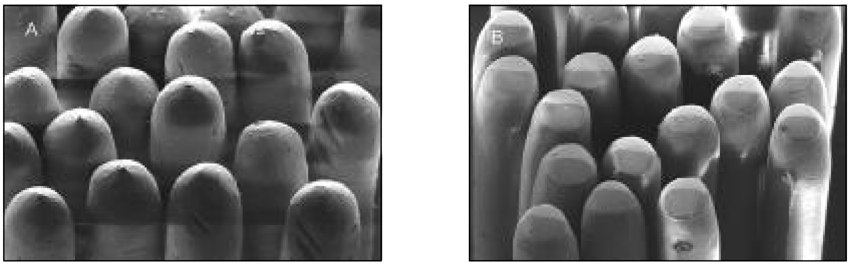
Picture 13. *Cement adaptation to metal (measurement point A). Negative image of the test surface illustrating a smooth and uninterrupted transition from the cement to the metal.*



Picture 14. *Cement adaptation to metal (measurement point A). Negative image of the test surface illustrating the defect of cement at the metal/ cement interface.*



Picture 15. *Cement adaptation to metal. Negative image of the test surface illustrating the defect of cement at the metal/ cement interface (measurement point A).*



Picture 16. *Endings of the bristles of a new toothbrush (a) and of a toothbrush submitted to 54 000 double strokes (b).*

middle point of the 250 µm gap.

The diversity in wear at the two different gaps obtained between three measurement points might indicate that the marginal gap width has no effect on cement wear or that some of unidentified factors or possible interactions between various factors have had a substantial influence and masked the effect of marginal gap width.

Previous studies, however, presented the importance of gap width on wear in luting cements, i.e. the vertical wear of studied materials increased with the marginal gap width [14, 15]. The strong relationship (correlation coefficient > 0.9) between substance loss due to wear and horizontal gap width reported by Kawai and co-workers [8] could be a confounding finding since the wear calculated as the vertical surface area of lost material (µm^2) was not adjusted to the gap width.

The second aim of the study was to compare the wear resistance of five different luting materials. Under the conditions of this experiment ZPC and GIC showed the least, CC and RC exhibited the greatest resistance to wear by toothbrushing, while RGIC was intermediate in wear resistance as compared to the other four materials.

The wear process is very complex, and the resistance of a certain material to wear may reflect many interplaying factors of the process and of the properties of the material [10]. Filler content and the degree of conversion in resinous types of luting cements were found to be among these factors [10], as well as the type of filler and polymer, the method of silanization and the filler size distribution [11]. These factors might be responsible for the differences in wear of RGIC, CC and RC in the present study. The lower resistance to wear of ZPC and GIC might be attributed to the difference in physical properties and structure of water-based compared to polymer-based luting materials [27]. Water-based cements are inherently brittle and, because of the powder-liquid formulation, also prone to porosity what might lead to an increased wear rate.

The results of the present study are in accordance with the findings by Shinkai and Suzuki [14] where wear resistance of microfilled resin cement was found to be superior to that of glass ionomer. Guzman et al. [15], however, found resin-modified glass-ionomer more wear resistant than the four resin cements studied.

The wide range of recorded wear values (especially at the cement/dentin interface) in the present study demonstrates a relatively high intra-variability (Picture 5). Varying quality in the bovine dentin employed could be one of the possible explanations for this high variability. Variations in bovine dentin structure depending on the location (occlusal or buccal) and the depth of the dentin (the location between the enamel/dentin junction and the pulp), similar to that of human dentin, has been reported [24, 25]. In fact, a highly statistically significant inverse correlation between dentin microhardness and tubular density has been reported [28]. In addition, as the age of the animals from which teeth were extracted was not specified, a varying extent of calcification might also have influenced the physical properties of the dentin. Although there is a lack of data indicating that the structure and physical properties of the dentin reflect its resistance to wear, it seems reasonable to assume that dentin quality is likely to influence this property.

One possible explanation for variation in wear at the metal/cement interface was linked to the difference in cement adaptation to metal. In contrast to a smooth and uninterrupted transition giving low loss value (Picture 13), cement defects (slits or small grooves that might increase effect of brushing) at the metal interface might result in considerably higher wear values (Picture 14,15).

Another possible explanation for the high intra-variability could be the use of toothbrushes (one toothbrush for three specimens). Although the toothbrushes showed no visible changes after use, SEM examination revealed flattening of the bristle tips (Picture 16). It could be postulated that the flattened tips may be less abrasive and result in less severe wear.

Possible manipulatory variations incorporated during the manual mixing of the cements or the manual fixation of the specimens into the brushing devise should be considered. The horizontality of a fixed specimen could be of crucial importance as even a minor inclination might create protruded areas subsequently more affected by toothbrushing.

Laboratory studies are important for understanding the performance of materials and the underlying mechanisms of wear. Moreover, all new materials have to be tested and approved in a laboratory before marketing. Many methods with a varying degree of sophistication have been designed to predict wear *in vivo*. Most tests focus on occlusal wear. Luting agents used to seal complete crowns are exposed to the oral environment only in the cervical region where different wear mechanisms are present. The present experimental design may, thus, be suitable for *in vitro* wear testing of any type of dental material exposed at the cervical area.

Extrapolation of an *in vitro* data into prediction of clinical behaviour is not always possible. However, the ranking of the cements tested under identical experimental conditions may be useful as a guideline for the selection of luting material in a particular clinical situation.

CONCLUSIONS

Within the confines of this study, ZPC and GIC were found to have the least, while CC and RC had the highest resistance to wear. RGIC was intermediate in wear resistance as compared with the other cements tested. The effect of gap width on substance loss could not be defined with certainty, as no consistent systematic pattern (of wear in the two different gap widths) was observed. Across the cement film the wear of all luting cements was the most marked at the cement/dentine interface, indicating a strong influence from the wear of the neighbouring dentin.

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