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ORIGINAL ARTICLE

Different scanning electron microscopic evaluation methods of cement interface homogeneity of adhesively luted glass fiber posts

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Abstract

Objective. To compare two methods used to examine the cement interface homogeneity of adhesively luted glass fiber posts (GFPs). ***Material and methods.*** GFPs were divided into four groups (n = 5 in each) and inserted into artificial root canals under standardized conditions: Group I = RelyX Unicem, application with application aid; Group II = RelyX Unicem; Group III = Panavia F 2.0; and Group IV = Variolink II. Posts in Groups II–IV were cemented without using an appliance. All specimens were sectioned at three levels (cervical, middle and apical) perpendicularly to the post's long axis and examined and photographed (n = 60) using scanning electron microscopy (SEM). Cement interface inhomogeneities were (A) measured by means of SEM software and (B) estimated using a graphics program with SEM images being divided into 72 equal circle segments to calculate a percentage value of inhomogeneities of the 360° circumference. ***Results.*** Median values of inhomogeneities (A/B; %) within the cement interface for the cervical, middle and apical levels of analysis, respectively were 1.4/2.1, 2.2/4.2 and 1.9/2.1 for Group I; 21.0/20.1, 24.8/23.6 and 27.0/24.3 for Group II; 1.5/1.7, 5.5/6.3 and 19.4/20.8 for Group III; and 18.1/16.7, 16.1/15.3 and 27.2/25.7 for Group IV. The two methods correlated very well (0.994), with a value of one indicating a 100% correlation. ***Conclusion.*** Both evaluation methods were found to be equally appropriate for quantifying the cement interface homogeneity of SEM cross-sections of adhesively luted GFPs.

Key Words: *Cement interface, evaluation method, fiber post, homogeneity, scanning electron microscopy*

Introduction

During recent years, the use of tooth-colored posts (mainly glass fiber-reinforced composite posts; GFPs) to rebuild the dentin core instead of metal posts has become daily routine in dental practice [1,2]. With GFPs it is possible to eliminate some of the disadvantages of cemented metal posts, such as discolorations of cervical root dentin and shading alterations of all-ceramic crowns and bridges [1]. Also, increased stress formation within the root, a common effect of using zinc phosphate cements for placing metal posts, is avoided when modern adhesive luting systems are used in combination with GFPs [3]. All modern systems for adhesive post placement are technique-sensitive [4–6] and involve special treatments of both the post surface and root dentin to achieve durable adhesion [7,8]. An inhomogeneous dispersion of the luting material within the cement interface may weaken the adhesion of endodontic posts and affect the durability of post-and-core restorations [9]. The homogeneity of the cement interface increases when special needle-like application aids are used [10,11].

In dentistry, four microscopic techniques [12] are commonly used to visualize resin–dentin interfaces: scanning electron microscopy (SEM) [13,14]; confocal laser scanning microscopy (CLSM) [15]; transmission electron microscopy (TEM) [16,17]; and atomic force microscopy (AFM) [18]. In addition, a new light optical microscopic evaluation method (LOM) was described by Watzke et al. [10]. The standard evaluation approach for cement interface imperfections is to use SEM for cross- or longitudinal sections of the cemented posts [13,19]. To analyze the homogeneity of the cement interface of cemented endodontic posts, two different methods have been

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used. The first evaluation method of SEM cross-sections measures the dimension of each cement interface imperfection and the post's diameter to calculate the percentage rate of inhomogeneities within the cement interface of cemented posts (measure method = A). The second evaluation method divides the micrographs of the cross-sections into equal circle segments to calculate a percentage value of inhomogeneities within the cement interface (estimation method = B) [20]. When different evaluation methods are used to analyze cement interface homogeneity, the comparability of data is restricted. Therefore, our objective was to comparatively evaluate the two methods used to examine the cement interface homogeneity of adhesively luted GFPs. The null hypothesis tested was that both evaluation methods of SEM cross-sections would not differ in terms of being appropriate for analyzing cement interface homogeneity.

Material and methods

Five GFPs (RelyX Fiber Post; 3M ESPE, Seefeld, Germany) in each of four groups were inserted into artificial root canals under standardized conditions. For the simulation of artificial root canals, transparent Polymethylmethacrylat (PMMA) blocks were used. The preparation of the post spaces (length = 13 mm) into the PMMA blocks was performed using the corresponding cavity drill of the GFP (size 2 RelyX Fiber Post universal cavity drill; 3M ESPE). The following experimental cementation approaches of the GFP were examined: Group I = RelyX™ Unicem and cement application with a needle-like application aid (elongation tip; 3M ESPE); Group II = RelyX Unicem; Group III = Panavia F 2.0 (Kuraray Dental, Düsseldorf, Germany); and Group IV = Variolink II (Ivoclar Vivadent, Schaan, Liechtenstein). The conventional post cementation in Groups II–IV involved placement of the adhesive luting material into the root canal, which was accessed by means of a dental probe, and consequent rotary insertion of the post loaded with luting material into the root canal [10].

Group I

GFPs and the artificial root canal were pretreated as per the manufacturer's recommendations. GFPs were degreased with propanol (70%) and dried with air. A silane coupling agent (ESPE Sil; 3M ESPE) was applied to the post surface and dried for 5 min. The artificial root canal was pretreated by rinsing with NaOCl and H_2O and dried with paper points.

The cementation procedure started by clicking the flexible root canal-shaped application aid (elongation tip) on the Unicem Aplicap. After activating the Aplicap for 2–4 s, the resin cement was machine-mixed for 15 s with the Capmix machine. Then the application aid was inserted to the bottom of the root canal and the self-adhesive resin cement (RelyX Unicem) was applied by slowly pulling the application aid out of the canal (5–10 s). Thus, the tip of the application aid was always embedded within the cementation material during the cement application. The post, loaded with the rest of the resin, was inserted without pressure into the artificial root canal. The self-adhesive resin cement was light-activated for 2 s. Excess material was removed using cotton pellets. The GFP was kept in position with the polymerization unit. The cement was finally polymerized for 20 s.

Group II

In Group II the same pretreatment of the GFP and the artificial root canal and the same cementation procedure as described for Group I were used, with the exception of use of the application aid. The cement was solely applied at the root canal access using the Unicem Aplicap.

Group III

In Group III the pretreatment of the GFP and the artificial root canal were identical to that described in Group I. The cementation process started with applying the primer (Panavia F 2.0 ED primer; Kuraray) using a microbrush. The primer was dried gently with air and excessive primer was removed with paper points. Then the mixed resin cement (Panavia F 2.0; Kuraray) was applied into the root canal by means of a microbrush. The GFP was also loaded with resin cement and inserted without pressure into the root canal. The excess resin material was removed using cotton pellets. Then the GFP was kept in position by means of the polymerization unit and the cement was finally polymerized for 20 s.

Group IV

In Group IV the pretreatment of the GFP was similar to that described for Groups I–III. The pretreatment of the artificial root canal involved some additional steps. After rinsing the canal with NaOCl and drying with paper points, root canal walls were etched with H_3PO_4 (37%) for 10–15 s. Afterwards, rinsing and drying was performed as described above.

The cementation process began with application of the primer (Syntac; Ivoclar Vivadent) with a microbrush. The primer was dried gently with air for 15 s and excessive primer was removed with paper points. Then the adhesive (Syntac; Ivoclar Vivadent) was applied and dried in the same way as the primer. The mixed dual-curing composite cement (Variolink II; Ivoclar Vivadent; base and catalyst 1:1 for 10 s) was applied into the root canal using a microbrush. The GFP was also loaded with mixed dual-curing composite cement and inserted without pressure into the root canal. The excess cement was removed using cotton pellets. Then the GFP was kept in position by means of the polymerization unit. The dual-curing composite cement was polymerized for 60 s.

Evaluation methods and statistics

After the post cementation procedure, all cemented GFPs were sectioned in a standardized manner perpendicular to the longitudinal axis of the post at three levels (cervical, middle and apical) and photographed and examined (n = 60) in a scanning electron microscope (Amray 1810) (Figure 1). First (A), the length of imperfections within the cement interface (U_1 in

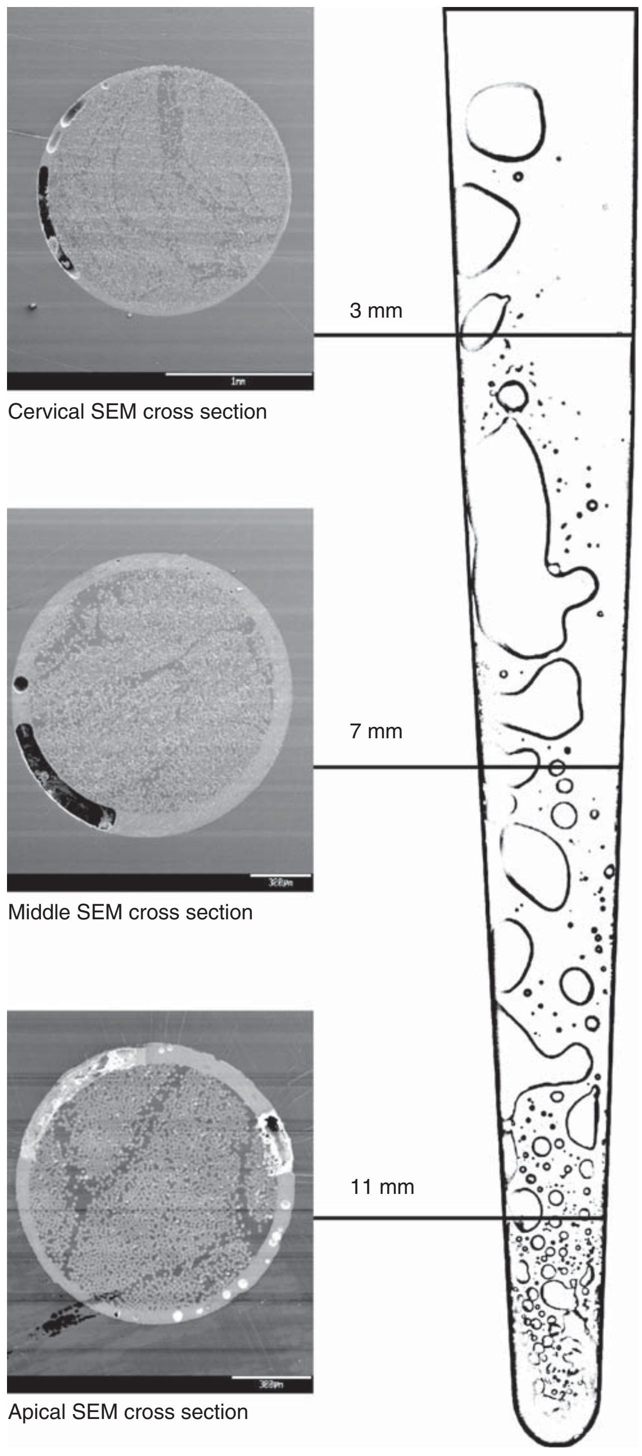
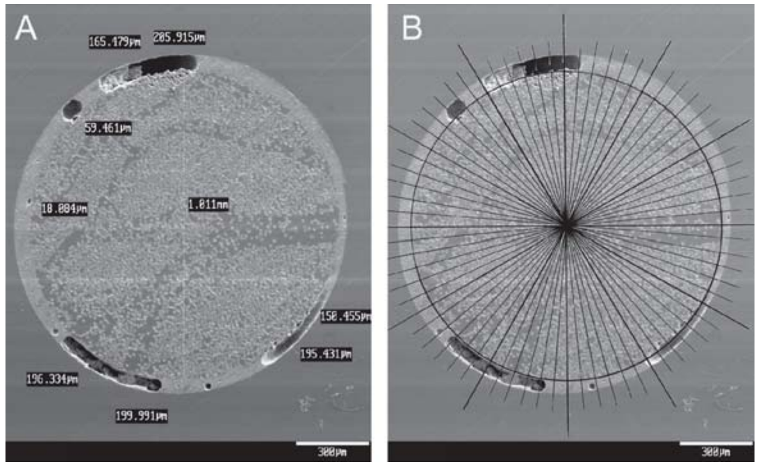


Figure 1. Example of graduation of analysis levels of GFPs and associated SEM cross-sections.



$$Y_A[\%] = \frac{U_1[\text{mm}] \times 100}{\pi \times d\,[\text{mm}]} \qquad Y_B[\%] = \frac{(\text{number of segments with imperfections})}{72}$$

$$Y_A[\%] = \frac{1.19115 \times 100}{\pi \times 1.011} \qquad Y_B[\%] = \frac{25}{72}$$

$$Y_A[\%] \approx 37.5 \qquad Y_B[\%] \approx 34.7$$

Figure 2. Details of (A) measure method and (B) estimation method for SEM cross-section evaluation of the cement interface homogeneity of adhesively luted GFPs. Examples are shown for the middle cross-sections of Groups I–IV.

millimeters) and the post diameter (d in millimeters) for all the SEM images were measured by means of an SEM-analysis program (measure method = A) to calculate the percentage rate (Y_A) of inhomogeneities within the cement interface of the adhesively luted GFPs according to the following formula:

$$Y_A = (U_1 \times 100)/(\pi \times d)$$

Secondly (B), all SEM images were divided into 72 equal segments of a circle using a computer graphics tool (Adobe Photoshop 5.5; Adobe Systems Incorporated, San Jose, CA) to estimate the imperfections within the cement interface and to calculate a percentage value of inhomogeneities of the 360° circumference (estimation method = B) (Figure 2). The number of segments depends on the size of the inhomogeneities. The circular arc length of one segment is similar to the length of small-sized inhomogeneities of a partition in 72 segments. Therefore, it is possible to count small-sized inhomogeneities in one segment.

The statistical analysis was performed by using the Pearson correlation and Kruskal–Wallis tests, and the Mann–Whitney test as a post-hoc test (SPSS 12.0G for Windows; SPSS Inc, Chicago, IL). The level of significance was set at $\alpha = 0.05$.

Results

The median values of inhomogeneities within the cement interface of adhesively luted GFPs for the two evaluation methods of cervical, middle and apical SEM cross-sections are displayed in Table I. The Pearson correlation coefficient for the correlation between evaluation methods (A) and (B) was 0.994, where a value of one indicates a perfect correlation of 100%.

The data for the apical SEM cross-sections showed significantly fewer inhomogeneities within the cement interface for Group I compared to the three other groups (Kruskal–Wallis: $P = 0.015$). The percentage rate of inhomogeneities of the apical cross-sections of Group I differed significantly from those for Groups II ($P = 0.008$), III ($P = 0.032$) and IV ($P = 0.008$) (Figure 3). There were no statistically significant differences between the four groups regarding cement interface homogeneity for the cervical (Kruskal–Wallis: $P = 0.301$) and middle (Kruskal–Wallis: $P = 0.096$) SEM cross-sections.

Discussion

The aim of this comparative *in vitro* study was to investigate two different evaluation methods, the measure (A) and the estimation (B) method, for analyzing the cement interface homogeneity of SEM cross-sections of adhesively luted GFPs. The Pearson correlation coefficient for the correlation between the two methods was 0.994, where a value of one indicates a perfect correlation of 100%. Therefore, the null hypothesis was accepted.

Data for both evaluation methods also showed that the use of a flexible root canal-shaped application aid was superior compared to the conventional cement

Table I. Inhomogeneities [median (min/max) (%)] within the cement interface of SEM cross-sections for the measure method (A) and the estimation method (B).

	Application aid cementation		Conventional cement application					
	Unicem		Unicem		Panavia		Variolink II	
Level of analysis	A	B	A	B	A	B	A	B
Cervical	1.4 (0.0/5.4)	2.1 (0.0/5.6)	21.0 (0.0/47.2)	20.1 (0.0/43.1)	1.5 (0.4/21.1)	1.7 (0.4/19.4)	18.1 (0.0/20.9)	16.7 (0.0/20.1)
Middle	2.2 (1.4/5.3)	4.2 (2.1/4.9)	24.8 (11.1/37.5)	23.6 (9.7/34.7)	5.5 (0.0/66.4)	6.3 (0.0/57.6)	16.1 (1.2/42.2)	15.3 (1.4/41.7)
Apical	1.9 (0.3/6.5)	2.1 (0.7/7.6)	27.0 (14.7/29.7)	24.3 (14.6/29.5)	19.4 (2.4/44.0)	20.8 (2.8/41.7)	27.2 (19.3/41.2)	25.7 (19.4/41.7)

application technique in terms of cement interface homogeneity for the apical level of analysis. When an application aid was used, inhomogeneities within the cement interface for the apical level of analysis decreased to ≈2%, compared to ≈25% for the conventional application procedure. These findings were in accordance with those reported previously [10]. It was shown by means of a LOM analysis method that the homogeneity of the cement interface of adhesively luted GFPs depends on the cement application method and that the use of an application aid increases the homogeneity of the complete cement interface. In the present study there was no statistically significant difference in cement interface homogeneity for the cervical and middle levels of analysis. An explanation for this could be that, under a scanning electron microscope, only a small area of the real interface can be evaluated and this area may not always be representative of the whole specimen [12]. In the literature, it has been discussed whether application procedures influence the cement interface homogeneity of adhesively luted endodontic posts [21] when SEM cross-sections are evaluated [7,22]. If the number of cross-sections for SEM evaluation increases, a less restricted conclusion concerning cement interface homogeneity for the whole specimen depending on the cement application procedure is more likely [23].

The main advantage of measure method (A) is that SEM cross-section images of adhesively luted GFPs can be analyzed directly concerning cement interface homogeneity without the use of a computer graphics tool which was previously required. The main disadvantage is the time-consuming measurement of the contact area of each cement imperfection with the post surface and the post diameter. In contrast, the estimation of the contact area of the cement imperfection with the post surface using method (B) is really fast, but the preparatory editing of the SEM cross-section pictures by means of a computer graphics tool is time-consuming. In summary, the cement interface evaluation seems faster and more practicable when using the estimation method.

One general limitation of this *in vitro* evaluation is the use of PMMA blocks to simulate artificial root canals instead of natural teeth, because there are differences in wettability between the surfaces of artificial acrylic root canals and natural root canals. When human teeth are used, the contact area between root dentin and luting cement can be examined in more detail, e.g. evaluation of the number and location of resin tags [7,13,14].

In addition to the homogeneity of the cement interface of adhesively luted endodontic posts, the durability of post-and-core restorations is influenced by several factors, which were analyzed extensively *in vitro* [24,25] and less extensively *in vivo* [26–28]. Such influencing factors include for example the

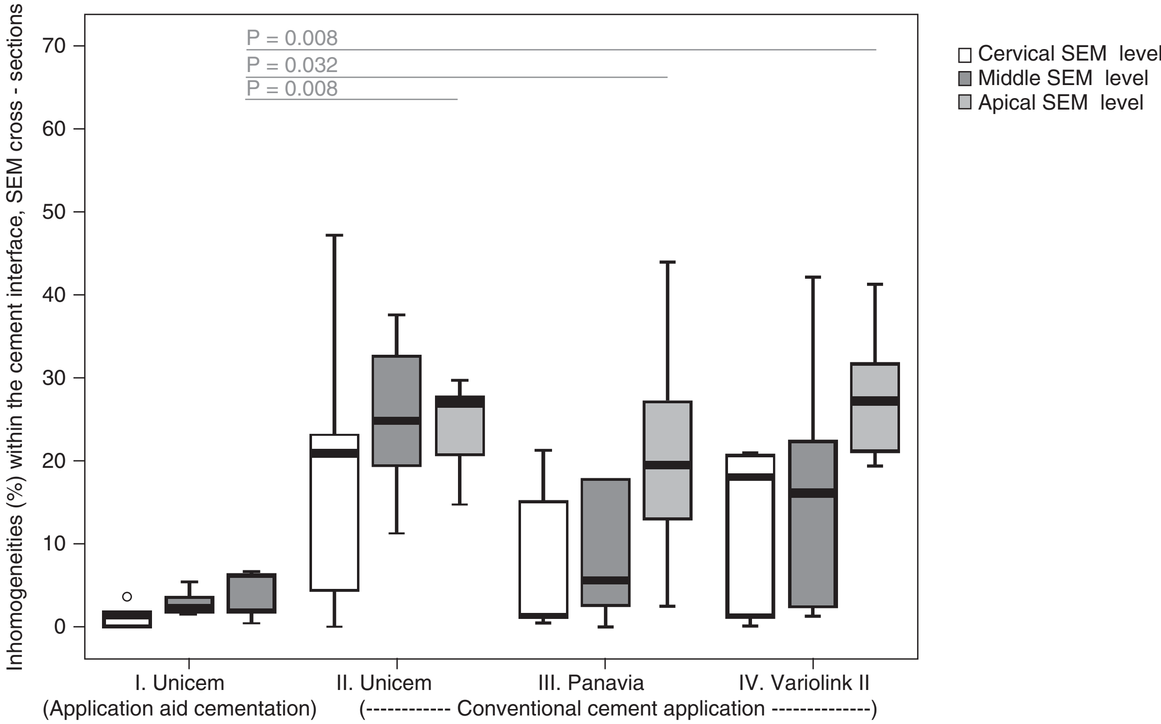


Figure 3. Box-plots showing inhomogeneities (%) within the cement interface of SEM cross-sections. Examples are shown for the data obtained using the measure method. Lines in the upper part of the Figure represent significant differences between tested groups ($P < 0.05$).

ferrule design preparation [29], the residual part of sound dentin [30,31], the length of the post [32] and the bone level of the restored tooth [33]. The clinical steps before post placement also exert an influence, as the technique-sensitive application [4–6] of the bonding system can have an effect on the durability of post-and-core restorations [34]. In summary, the restoration of a tooth by means of a post and core is a complex treatment. Therefore, further developments of adhesive luting materials for post cementation should provide an easy, homogenous and technique-insensitive application, in combination with a reduced number of clinical steps and a universal and strong bond to all types of surface qualities.

Within the limitations of this *in vitro* comparison of two SEM cross-section evaluation methods, the measure and estimation methods were shown to be appropriate for quantifying the cement interface homogeneity of adhesively luted GFPs.

Declaration of interest: The authors report no conflicts of interest. The authors alone are responsible for the content and writing of the paper.

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