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Determining the influence of fibre post light transmission on polymerization depth and viscoelastic behaviour of dual-cured resin cement

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Abstract

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Aim To evaluate the effect of quartz-fibre posts on the depth of polymerization of a dual-cure resin cement using Raman spectroscopy and to determine the physical properties of the polymerized cement using a dynamic mechanical analyzer (DMA).

Methodology Twenty-five fibre and 25 CrNi posts were used to evaluate depth of polymerization. Posts were cemented with dual-cure resin into root canals formed from silicone moulds, without using bonding or etching agents. After polymerization, resin layers on each sample were removed using a curette and cut into three equal parts (apical, middle and coronal). All resin specimens for every third were gathered and crushed. Resin powder samples were analysed using Dynamic Mechanical Analysis and Raman spectroscopy for each third.

Results The numerical data revealed that the thermal transitions of the materials took place at higher temperatures from the apical to the coronal sections in both groups. C=C double bond intensity was lower in fibre post-resin cement samples when compared to their intensity in metal post-resin cement samples.

Conclusion Dual-cured resin cements had more rigid properties and better polymerization for fibre posts when compared with metal posts. Polymerization quantity was affected by position in the canal.

Keywords: dual-cure resin cement, dynamic mechanical analyzer, fibre-post, light transmitting, Raman spectroscopy.

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Introduction

Several different post-core systems are used for the restoration of root filled teeth before placement of the final restoration. In the last two decades, fibre-reinforced posts have gained in popularity because of their favourable flexibility and modulus of elasticity when compared with metal posts (Schwartz & Robbins 2004). Laboratory studies have revealed that fibre posts are associated with more favourable fractures on teeth compared to metal posts (Akkayan & Gulmez 2002, Newman *et al.* 2003, Fokkinga *et al.* 2004). Besides this mechanical superiority, fibre posts, including quartz or glass fibre, have aesthetic advantages.

To provide lower microleakage and higher retention, adhesive cementation is recommended for fibre posts (Peroz *et al.* 2005). However, during cementation, bonding to the apical region of canals can be compromised because of lack of light exposure (Pegoraro *et al.* 2007). Therefore, dual-cure resin cements, which are both chemical and light activated, are used as a luting agent for light-transmitting fibre posts. However, light activation is still a prerequisite for the polymerization of dual-cure resin cements, and

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inadequate polymerization can occur when light transmission is not sufficient (Kumbuloglu *et al.* 2004, Faria e Silva *et al.* 2007, Acquaviva *et al.* 2009). Giachetti *et al.* (2009) reported that light-curing resin cements had similar polymerization depths to dual-curing resin cements only when translucent fibre posts were used. Sigemori *et al.* (2005) concluded that the chemical activation modes of resin cements were inadequate for optimal hardness values. In several studies, it has been reported that using translucent glass–fibre posts increases the depth of polymerization in root canals (Lui 1994, Yoldas & Alacam 2005, Faria e Silva *et al.* 2007).

The mechanical properties of polymerized resin cement are essential for the success of post-core systems. As the polymers are viscoelastic materials, dental resin composites demonstrate viscoelastic behaviour under cyclic loading in the oral cavity (Sideridou *et al.* 2008). Dynamic mechanical analyzers (DMA) are widely used for polymer characterization and can simulate chewing forces (Mesquita *et al.* 2006, Sideridou *et al.* 2009). During testing with a DMA, an oscillating strain is applied and the resulting oscillating stress is measured; conversely, an oscillating stress is applied and the resulting oscillating strain is measured (Craver & Carraher 2000).

Unpolymerized resin cement contains C=C double bonds, and during polymerization, these bonds are broken. Raman spectroscopy provides quantitative information about C=C double bonds.

The aim of this study was to evaluate the effect of currently used quartz-fibre posts on the depth of polymerization of dual-cure resin cement using Raman spectroscopy and to determine the physical properties of the polymerized cement using a DMA. The null hypothesis was that when fibre posts are compared with metal posts for their light transmission behaviours on dual-cured resin cement, more C=C double bonds will be relieved, and therefore, the polymerization degree will increase if fibre posts are used.

Materials and methods

Double-tapered translucent quartz-fibre posts (Light-Post, Bisco Inc., Schaumburg, IL, USA) and cast chrome-nickel posts were used. To ensure the same diameter and length in both types of posts, metal posts were produced from the fibre posts. After the impression was taken from a fibre post, the post space was filled with acrylic resin (Palavit G, Heraeus Kulzer, Hanau, Germany), which was then cast with Cr-Ni alloy. Their diameters were 1.8 mm in the coronal and 1 mm in the apical region, and their lengths were 20 mm.

To easily remove the posts that were surrounded by dual-cured resin cement, simulated root canals were prepared from polyvinylsiloxane impression material. To establish a convenient and accurate space for the post and resin inside the moulds, an impression coping was produced. For this purpose, a fibre post was thickened by 0.5 mm with a plastic strip that was placed 2 mm shorter than the top and 3 mm shorter than the bottom of the post (Fig. 1a). After taking the impression of this thickened fibre post, the space created was filled with acrylic resin and left to set. Then, it was cast with Cr-Ni alloy, and

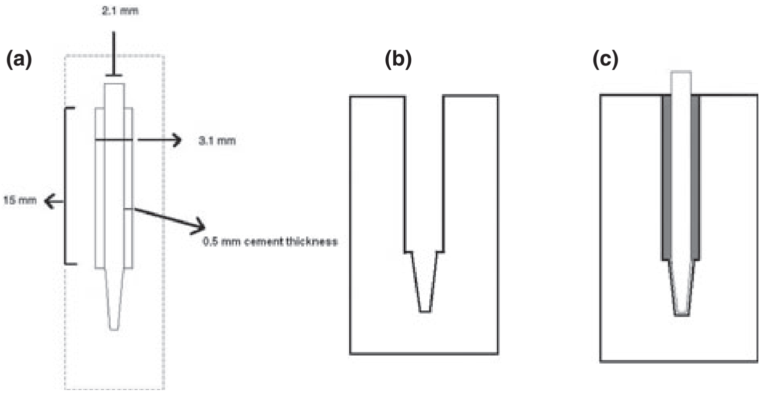

Figure 1 Schematic illustration of mould and sample preparation: (a) fibre post surrounded by 0.5 film layer used for impression post preparation; (b) artificial root canal system formation in polyvinylsiloxane mould by impression post; (c) post, cemented using dual-cure resin cement.

the impression coping was produced. This impression coping was used to produce fifty simulated root canals (Fig. 1b).

These moulds were divided into two groups (Group F and Group M, 25 moulds each). Quartz-fibre posts were used in Group F moulds, whilst Cr-Ni metal posts were used in Group M.

Dual-cured resin cement Rely X ARC (3M ESPE, St. Paul, MN, USA) shade A3 was used for both groups. The base and catalyst pastes were mixed following the manufacturer's instructions and inserted in the simulated root canal using a lentulo spiral (Size 40, FGK Dentaire, La Chaux-de-Fonds, Switzerland). The posts were placed immediately in the root canals with finger pressure. To ensure that the post was centred laterally and the cement was distributed evenly around it, the 3-mm part of the apical portion was fixed into the narrower hole at the bottom of the mould (Fig. 1c).

Excess cement was removed by a brush tip. A halogen light (Hilux, Benlioglu Dental, Ankara, Turkey) of 685 ± 10 mW cm^{-2} wavelength was applied perpendicularly over the post for 40 s to polymerize the resin cement. Before the experiments, the excitation density of the light source was measured by a radiometer (Hilux Curing Lightmeter, Benlioglu Dental, Ankara, Turkey) to ensure that the light source had the correct wavelength.

The samples were stored at 37 ± 0.2 °C for 24 h to simulate clinical conditions, and then the post samples with surrounding resin cement were removed from their moulds. The 15-mm part of the posts (the part covered with the resin cement) was cut into three equal parts (5 mm) as apical, medium and coronal thirds by means of a diamond disc on a handpiece.

The resin cement on each third of every sample was collected separately using a spatula and crushed. DMA and Raman spectroscopy analysis were performed for the resin in the three different canal thirds, and the values were compared.

Dynamic mechanical behaviours of polymers were analysed using a TA Instruments Q800 Dynamic Mechanic Analyzer (New Castle, DE, USA). The temperature dependence of polymers was measured at a constant frequency ($\omega = 1$ Hz) using a standard temperature sweep (3 °C per min) from −50 to 100 °C.

The Raman spectra of polymers were obtained using a Jobin-Yvon LabRam HR800 Raman spectrometer (Longjumeau Cedex, France) equipped with a charge coupled device (CCD) detector using the 632.8-nm line of a He-Ne laser. The powdered samples were pressed onto microscope slides.

Results

DMA

The derivative storage modulus-Temperature (T) behaviour of samples, which contained metal posts and fibre posts and were separated into three parts (apical, medium and coronal), are shown in Fig. 2a,b. The inverted peaks (also marked in Fig. 2) show the temperatures at which the rigidity properties of the polymers change; in other words, they represent the thermal transition temperatures of the polymers.

When derivative storage modulus-T behaviours of the apical thirds of group M and F samples are considered, under 20 °C, it was seen that the thermal transition temperatures at −39.64, −24.05, −19.60, −11.26 °C for group M-apical samples were shifted to

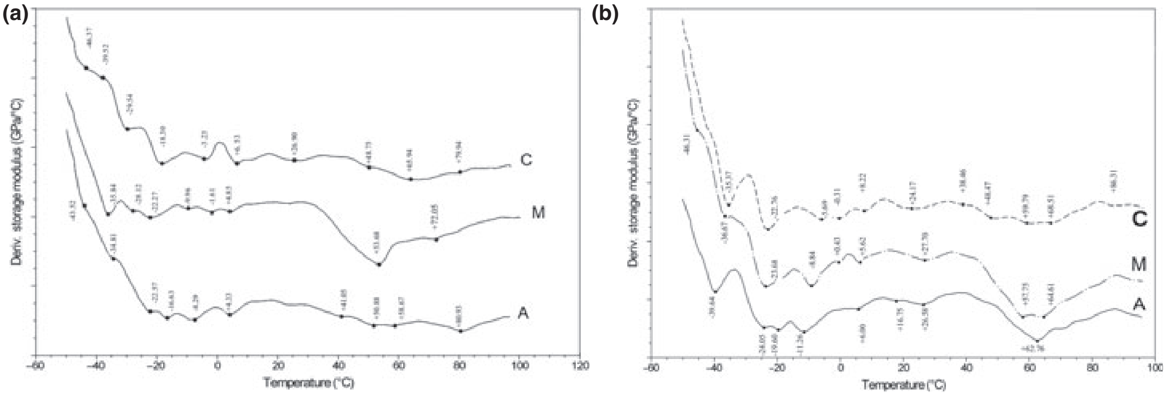

Figure 2 (a) Derivative-storage modulus –T behaviour of group F; (b) derivative-storage modulus –T behaviour of group M.

higher temperatures −34.81, −22.57, −16.83, −8.29, +4.33 °C for group F-apical samples. A similar behaviour for temperatures over 20 °C was also demonstrated as thermal transition temperatures at +26.58 and +62.76 °C for group M-apical samples that shifted to +41.05, +50.88, +58.67, +80.93 °C for group F-apical samples.

When the thermal transitions of the medium thirds of group F and M samples were compared, it was seen that the thermal transition temperatures of group M-medium samples at −36.67 and −23.68 °C were higher for group F-medium samples. Although not regular, changes in transition temperatures were detected between −20 °C and +20 °C. At the >20 °C temperatures, transitions of group F-medium samples were again higher at +62.66, +72.05, +79.56 °C compared to the thermal transitions of group M-medium samples at +57.75 and +64.61 °C.

When the thermal transition temperatures of the coronal thirds of groups F and M samples were examined, it was seen that the transitions for group M were recorded at −35.37 and −22.76 °C but for group F at −29.54 and −18.30 °C, respectively. These transition temperatures for group F can be considered very high. Between temperatures −20 and +20, there were again slight shifts into higher temperatures. For temperatures >20, the thermal transitions for group M-coronal samples were recorded at +48.47, +59.78 and +68.51 °C; for group F-coronal samples, they were recorded at +48.75, +65.94 and +79.94 °C, respectively.

The numerical data also suggested that the thermal transition temperatures of the materials increased from the apical to the coronal region.

Raman

When the Raman spectrums of group F-coronal and group M-coronal structures were examined (Fig. 3b, d), the low frequency bands at 1624, 1636 and 1640 cm^{-1} could be detected. At the middle samples, Raman bands are seen at 1625, 1621, 1631, 1641 and 1647 cm^{-1}. In the apical portion of both post-types, Raman bands that have similar wave numbers to the coronal and medium parts can be observed (Fig. 3a,c).

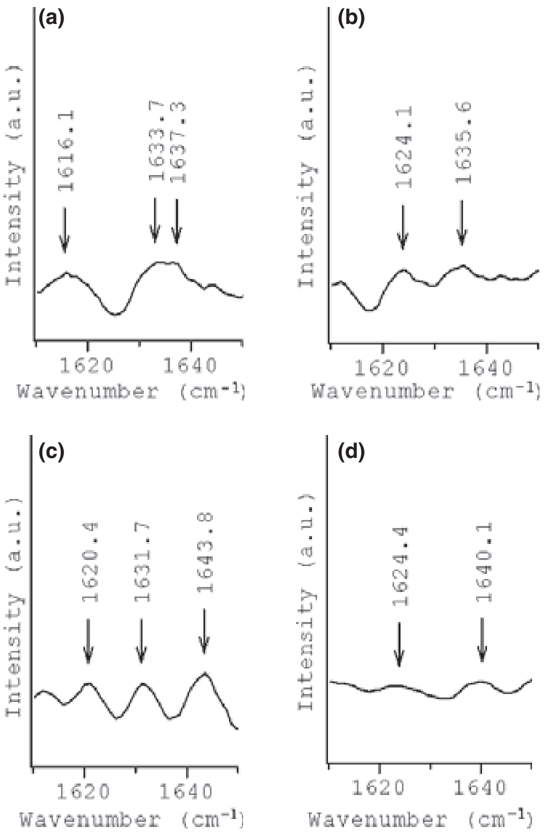

Figure 3 Raman spectrums obtained from (a) apical part of group F; (b) coronal part of group F; (c) apical part of Group M; (d) coronal part of Group M.

Discussion

In this study, the effect of light translucency of quartz-fibre-reinforced posts over polymerization depth was investigated by means of DMA and Raman spectroscopy.

In the literature, static tests are widely used to determine the mechanical properties of dental composites. However, this can be insufficient to compare materials that have viscoelastic behaviour. Dynamic tests are required when comparing these groups of materials, because they can simulate periodical chewing forces that occur *in vivo*.

In contrast to other studies (Mesquita *et al.* 2006, Wu & Nie 2007, Sideridou *et al.* 2008, Mesquita & Geis-Gerstorfer 2008, Sideridou *et al.* 2009) in which the storage modulus-T behaviours were investigated, in this work the derivative storage modulus-T behaviours were studied because the resin cement used has a heterogeneous structure. Every single polymer structure inside the resin cement shows thermal transition at different temperatures. Therefore, every polymer has different elasticity modulus values, and one cannot determine the temperatures at which thermal transition occurs just by investigating the storage modulus-T behaviour. When the derivative is taken (hence the derivative storage modulus-T graph), every inverted peak shows the thermal transition temperatures of a different component in the heterogeneous structure. Thermal transitions show the existence of cross-linked structures. However, which of these structures has a thermal transition at what temperature is still unknown. Further comprehensive studies are required to define these structures.

If the derivative storage modulus-T graphs of polymerized resin samples between −50 and 100 °C are considered, it can be said that the low thermal transition temperatures correspond to soft structures, whilst the higher transition temperatures correspond to more rigid structures. Thermal transitions do not display a stable behaviour around 20 °C. Therefore, 20 °C was set as the reference point, and the transitions below and above this point were evaluated separately. The reason for exposing polymerized resin cement to a temperature between −50 and 100 °C was to investigate the rigidity behaviour of all the components in the heterogeneous structure.

When comparing group M and group F's apical, medium and coronal derivative storage modulus-T behaviour, the shift in thermal transition temperatures to higher degrees and the formation of variable peaks for group F point to an increase in three-dimensional cross-linked structures. Therefore, it can be said that the translucency of the fibre posts used in this study enhanced resin polymerization. Also, it was seen that the thermal transition temperatures for both groups increased from apical to coronal. This demonstrates a difference between canal regions in cross-linked structure density, even within the fibre posts.

Raman spectroscopy values also support this result. Compared to infrared spectroscopy, Raman spectroscopy provides more valuable information about the stretching vibrations of C=C bonds. Resin cement contains C=C functional groups, both in aliphatic and in aromatic characters. Aliphatic C=C structures enter reaction during polymerization, but aromatic structures do not. Therefore, with increased polymerization, a decrease in aliphatic C=C magnitude is expected in Raman studies.

In previous studies that used Raman spectroscopy (Kumbuloglu *et al.* 2004, Arrais *et al.* 2007, Faria e Silva *et al.* 2007), aliphatic C=C stretching for resin cement was found to be at 1638 cm^{-1} and it was shown that the peak magnitude at this wave number decreased as polymerization increased. However, the band related with C=C stretching vibration in aromatic structures was recorded at 1608 cm^{-1} and did not change with polymerization. Other studies reported that aliphatic C=C stretching was recorded at 1640 cm^{-1} and aromatic C=C stretching at 1610 cm^{-1} for resin cement Raman spectroscopy (Peutzfeldt *et al.* 2000, Asmussen & Peutzfeldt 2001, Asmussen & Peutzfeldt 2003, Kumbuloglu *et al.* 2004). Geurtsen & Leyhausen (2001) reported that C=C stretching vibration was 1583 cm^{-1} in aromatic structures and at 1637 cm^{-1} in aliphatic structures.

In this study, a decrease in the peak magnitude of group F samples was detected when the C=C stretching vibrations were compared with group M samples. The peaks in the apical portion of group M were especially high. This phenomenon, which indicated that there was still partial aliphatic C=C stretching in the structure, was partially seen in the fibre structure. As a result, a smaller number of C=C double bonds were broken and joined in a cross-linked chain in the apical portion of group M. Between 1620 and 1645 cm^{-1} wave numbers, numerous bands are seen because of the different characters of aliphatic C=C structures (TEGDMA and Bis-GMA).

Insufficient polymerization can cause biological and mechanical problems. Especially in the apical part, a leakage of monomers from unpolymerized resin cement through root fillings may lead to inflammatory reactions, cytotoxicity and necrosis in periodontal tissues (Stansbury & Dickens 2001, Janke *et al.* 2003, Volk *et al.* 2007).

Conclusion

Using translucent fibre posts rather than metal posts resulted in a more stable resin cement in the deeper parts of the root canal. With fibre posts, more C=C double bonds were broken to form a cross-linked structure, resulting in better viscoelastic behaviour. However, the coronal region was still the most rigid when compared with middle and apical regions. This showed that, apart from post material selection, polymerization increased from the apical to the coronal region. From the clinical point of view, fibre posts can help create a more stable and sound post-resin unit and thus contribute to the durability of restored teeth.

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