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**“DENTAL MATERIALS AND THEIR CLINICAL
APPLICATIONS”**

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**A study into the application of fiber posts and composite core materials for
restoring endodontically treated teeth**

*[Valoración de diferentes composites y técnicas para la reconstrucción de los dientes
endodonciados utilizando postes de fibra]*

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Chapter 1

1.1. General Introduction

Dental practitioners are often faced with the task of restoring endodontically treated teeth always considered a controversial topic. Recently a new approach, providing the use of restorative materials structurally compatible with tooth hard tissues combined with the advantages of the adhesive technique, has been introduced.

Since their invention at the beginning of the 90s, fiber posts acquired growing importance becoming more and more popular in the restoration of non-vital teeth (Duret *et al*, 1990).

There is scientific evidence showing differences between pulpless and healthy teeth. They contain up to 10% less collagen bounded moisture, but this factor does not result in a significant change of physical properties, like modulus of elasticity, hardness or fracture strength (Helfer *et al*, 1972) (Sedgley and Messer, 1992) (Papa *et al*, 1994). It is assumed that the cavity access preparation for the endodontic treatment is the first factor involved in the reduction of tooth strength, due to the loss of the roof of the pulp chamber and the marginal ridges (Ross, 1980) (Trope *et al*, 1985) (Howe and McKandry, 1990) (Morgano *et al*, 1993). Caries, traumas or removal of previous restorations contribute to reduce the residual tooth structure and the fracture resistance (Fredriksson *et al*, 1998) (Ferrari and Scotti, 2002) (Fernandes *et al*, 2001). Thus, prosthetic crown coverage is traditionally recommended.

Clinical and *in vitro* studies clearly have been shown that metal cast post and cores have no longer to be considered the first choice treatment in terms of incidence of root fracture, aesthetic appearance and clinical performance (Shillingburg *et al*, 1997) (Heydecke *et al*, 2002) (Akkayan *et al*, 2002). Controversially, fiber posts were developed with the purpose of overcoming some of the limitations of metallic posts.

The increase in popularity of the use of fiber posts and the systematic appearance in the market of new composites with improved properties (wear resistance, filler content, viscosity etc.), is the purpose for an evaluation of the different restorative materials commercially available.

This thesis contains a study about several different aspects related to the use of fiber posts, the selection of the composite core material and the core build-up procedure when restoring structurally compromised endodontically treated teeth.

Different criteria are usually employed based on experimental and basic evaluation: in the thesis, a qualitative analysis of the morphologic characteristics of different types of composites used to build-up the core around translucent fiber post has been described (Schwartz and Robbins, 2004). In particular, the integrity of the abutment and the quality of the post/core interface was evaluated through scanning electron microscopy. As a matter of fact, the quality of a restoration is dependent on the properties of the composite and its handling: the study was conducted with the aim of identifying the most appropriate core material to achieve the best adaptation to the post surface.

The build-up procedure for replacing lost coronal dentin is a key factor as well as the selection of the proper material. Many different techniques have been described for the direct restoration of endodontically treated teeth in combination with resin composites: a comparison between the incremental technique and the use of pre-shaped shells to build-up the core around fiber posts with different composite materials was performed in the thesis. The study was conducted through microscopic analysis to reveal if the quality of the restoration, despite of the selected resinous material, is also technique sensitive.

A clinical prospective trial was performed to confirm the findings of the *in vitro* studies, to assess if flowable resin composites used for build-up the core could be able to provide a valid support to all-ceramics crowns during medium-term of clinical service. Also, the failure mode of the restorations was taken into consideration. Silane coupling agents are commonly used to improve the interfacial strength between fiber posts and composite core build-ups; however, it still remains a relatively weak bond.

This circumstance is more likely to occur in the absence of a chemical compatibility between the methacrylate-based composites and some types of fiber posts (mainly due to the nature of their resinous matrix). The idea of improving the interfacial strength of composites to fiber posts is desirable, especially when compared to the results normally achieved on tooth substrates. In this project three different *in vitro* studies were performed with the purpose of applying to fiber post restorations the concept of creating a chemical and micromechanical interlocking, commonly described for dentin and ceramic substrates. Different chemicals are used in laboratory and industrial fields showing their effectiveness on epoxy resin substrates. Based on the same principle, superficial pre-treatments of epoxy matrix fiber posts have been performed before silanization to assess if a substantial increment in bond strength may

be achieved by micromechanical interlocking. The aim was to identify an easy, effective and clinically feasible method for enhancing bond strength. The effectiveness of etching fiber posts in terms of superior bond and improved quality of the restoration would be the rationale for performing the technique clinically.

A further object in this project was therefore to evaluate the potentials of different silane coupling agents to improve the strength of the bond between etched fiber posts and core materials. Microtensile bond strength test and scanning electron microscopic analysis were performed in the studies conducted for this purpose.

1.2. Introducción general

Los odontólogos se enfrentan a menudo con el problema de la restauración de los dientes endodonciados, siempre considerado un tema de discusión.

Recientemente ha sido introducido un nuevo enfoque, basado en la selección de materiales estructuralmente más compatibles con la dentina y de técnicas operatorias de tipo adhesivo.

Desde que fueron propuestos al principio de los años noventa, los postes de fibra adquirieron una importancia creciente en la reconstrucción de los dientes endodonciados (Duret *et al*, 1990). Existe una evidencia científica de que la actuación de los dientes endodonciados es diferente a la de los dientes vitales. El contenido de humedad relacionada a las fibras de collagen es hasta un 10% inferior, aunque este factor no determine un cambio significativo en términos físicos, como el modulo elástico, la dureza o la resistencia a la fractura (Helfer *et al*, 1972) (Sedgley and Messer, 1992) (Papa *et al*, 1994).

La preparación del acceso para la realización del tratamiento endodontico es la primera causa de reducción de la resistencia del diente, debida a la eliminación del techo de la cámara pulpar y de las crestas marginales (Ross, 1980) (Trope *et al*, 1985) (Howe and McKandry, 1990) (Morgano *et al*, 1993).

Las caries, los traumas y la remoción de restauraciones precedentes contribuyen a la reducción de la estructura dentaria y de la resistencia a la fractura (Fredriksson *et al*, 1998) (Ferrari and Scotti, 2002) (Fernandes *et al*, 2001). La cobertura con una corona protésica es recomendada comúnmente.

Estudios clínicos y de laboratorio recientes demuestran claramente como la utilización de postes y muñones metálicos no debería ser siempre el tratamiento de elección para la restauración de los dientes endodonciados debido a la elevada incidencia de fracasos radiculares, a la falta de propiedades estéticas y al comportamiento clínico (Shillingburg *et al*, 1997) (Heydecke *et al*, 2002) (Akkayan *et al*, 2002). Los postes de fibra de vidrio fueron introducidos con el propósito de solucionar las limitaciones que presentan los postes metálicos.

La reciente incorporación y creciente popularidad del uso de los postes de fibra y la difusión de nuevos composites con mejoradas propiedades mecánicas (resistencia al desgaste, cantidad de relleno, viscosidad etc.) es la presupuesto para proponer y valorar distintas técnicas de adhesión, tipos de postes de fibra y composites con el fin de mejorar la interfase adhesiva y la longevidad de la unión.

El objetivo principal que se plantea en esta tesis doctoral es estudiar y determinar cual es la mejor técnica para la reconstrucción de los muñones con postes de fibra de vidrio, seleccionando el mejor acondicionamiento de la superficie del poste, material compuesto de unión y agente adhesivo.

Diferentes criterios básicos y científicos de evaluación han sido comúnmente utilizados para el estudio de la restauración de los dientes endodonciados (Schwartz y Robbins 2004). Se realizó una evaluación de las características morfológicas de distintas resinas compuestas para la restauración de los muñones con postes de fibra mediante microscopia electrónica de barrido.

En particular, se analizaron la calidad de la adhesión entre los postes de fibra y el composite y la integridad de las reconstrucciones. Las propiedades de la resina compuesta y su modo de aplicación influyen en la calidad de la restauración: el trabajo de investigación se realizó con el objetivo de identificar el composite que mejor se adapte a la superficie del poste.

La técnica utilizada para la reconstrucción de la estructura dentaria residual es un factor importante así como la selección de una apropiada resina compuesta. Se describieron distintas técnicas de restauración directa de los dientes endodonciados: se realizó una comparación entre la técnica incremental y el uso de matrices preformadas para la reconstrucción de muñones con postes de fibra y resinas compuestas. La microscopia electrónica de barrido fue utilizada en la tesis para caracterizar la calidad de la interfase poniendo de manifiesto que la calidad de la

restauración a pesar del material seleccionado, es también dependiente de la técnica operativa.

A continuación, se realizó un estudio clínico para obtener una confirmación *in Vivo* de las conclusiones encontradas en los estudios de laboratorio. Se puso de manifiesto que las resinas compuestas de tipo fluido pueden ser utilizadas para las reconstrucciones con postes de fibra de los dientes desvitalizados y pueden soportar reconstrucciones protésicas en cerámica sola durante 2-3 años de estudio.

Es necesario también tener en cuenta el tipo de fracaso de las restauraciones. Los agentes de acoplamiento silano se aplican comúnmente para mejorar la fuerza de adhesión a la interfase entre el composite y el poste de fibra, sin embargo la adhesión sigue siendo débil.

Esta circunstancia probablemente es la consecuencia de la falta de compatibilidad química entre los grupos methacrílicos de las resinas compuestas y los postes de fibra (principalmente debido a la naturaleza de sus matrices resinosas). La idea de mejorar la fuerza de adhesión a la interfase poste de fibra/composite es deseable, especialmente si comparada a los resultados obtenidos en esmalte o dentina. En la tesis, se realizaron tres distintas investigaciones *in Vitro* con el objetivo de aplicar el concepto de adhesión química y micromecánica, utilizado para el sustrato dentinal o la cerámica a las restauraciones con postes de fibra y resina compuestas.

Se conoce la eficacia sobre la resina epóxica de diferentes agentes químicos utilizados para métodos de laboratorio o de grabado industrial. Basado en el mismo principio, se desarrollaron distintos tratamientos superficiales de los postes de fibra con matriz de resina epóxica para evaluar si una adhesión de tipo micromecánico pueda garantizar un aumento substancial de la fuerza de adhesión.

El objetivo principal fue el de individuar un método simple y eficaz para mejorar la fuerza de adhesión y justificar su aplicación en la clínica.

Lo último objetivo que este proyecto se planteó es la evaluación de la aplicación de distintos agentes de acoplamiento silano para promover la adhesión entre los postes de fibra grabados y las resinas compuestas. Se utilizaron la técnica de microtensión para medir la fuerza de adhesión en las interfaces de interés y la microscopía electrónica de barrido para caracterizar la interfase.

1.3. Characteristics of endodontically treated teeth

Several studies stated the existence of substantial differences between endodontically treated and vital teeth.

Healthy dentin contains about 50 vol % mineral, 20 vol % water and 30 vol % organic matrix and back in 1997 Marshall described it as a "...biological composite of a collagen matrix filled with submicron to nanometerized calcium deficient, carbonate-rich apatite crystallites dispersed between micron-sized hyper-mineralised collagen-poor hollow cylinders.." (Marshall *et al*, 1997).

Dentin after root canal treatment has been considered more brittle due to the loss of water content (Helfer *et al*, 1972) or the absence of collagen cross-linking (Rivera and Yamauchi, 1993). A reduction of the percentage of collagen has been shown to occur overtime after endodontic treatment both in root and coronal dentin (Mason, 2001). A recent transmission electron microscopy study revealed a decrease in the distribution of collagen fibrils at the radicular level in teeth that have been endodontically treated for 5 years (Ferrari *et al*, 2004).

Type I collagen fibers are one of the main constituent of healthy dentin: Nakabayashi and Pashley (1998) demonstrated the preventive role in microcrack propagation exerted by these fibers, stating that fractures are more likely to occur after root canal treatment as consequence of their removal.

Back in 1992, Sedgley and Messer tested the biomechanical properties of dentin from endodontically treated teeth with an average of 10 years after treatment, revealing that apart from a decrease in hardness, the properties did not differ much from vital dentin (Sedgley and Messer, 1992). A reduction in stiffness of intertubular matrix may be the possible explanation (Kinney *et al*, 1996).

In any case, degradation of the physical properties (Young's modulus, hardness or fracture strength) of dentin can not be directly associated with the endodontic treatment, since age and disease may also influence its composition.

The lack of a protective feed-back mechanism due to the removal of the pulp may contribute to increase the risk of fracture after root canal treatment, even if there is no clear evidence about this topic (Randow and Glanz, 1986) (Fennis *et al*, 2002).

The majority of the studies consider the loss of structural integrity, due to access cavity preparation, rather than changes in dentin composition, the main factor

responsible of the higher occurrence of fracture in endodontically treated teeth (Reeh, 1989).

Access preparation and the consequent loss of the central tooth structure, may induce a great deformation during function increasing cuspal deflection and risk of fracture than in healthy teeth (Fuzzi, 1996).

The cleaning and shaping procedures may determine additional morphological changes at the radicular level, weakening the tooth. Thanks to the introduction of Ni-Ti instruments, a more conservative shaping of the root canal has been adopted with respect to the conventional stainless steel instruments (Gambill *et al*, 1996) (Glosson *et al*, 1995). Moreover, endodontic therapy is often the ultimate treatment after the removal of pre-existing restorations in teeth in which a substantial part of dental tissue have been already compromised.

1.4. Restorative techniques and principles for post placement

For many years it was considered as necessary placing post and cores in every tooth that received an endodontic therapy. Post restorations were considered a way for reinforcing endodontically treated teeth in an attempt to strengthen the root. However, as a significant amount of sound dentin has to be scarified, especially for cast post placement, it was realized that the primary purpose of a post is connecting the root with the build-up material placed on the coronal portion when an extensive loss of coronal structure does not ensure the retention of a core (Goodacre and Spolnik, 1994).

Cast post and core restorations have been advocated as the gold standard for decades when restoring endodontically treated teeth. During the last few years, there has been a major shift away from metal custom-cast post and cores toward prefabricated metal posts and more recently, to fiber-reinforced composite post used with bonded resin-based composite build-ups. Even if custom-cast posts still offer some advantages in some clinical situations (misaligned teeth or multiple teeth restorations) and the high success rate reported in some recent studies (Walton, 2003), they have fallen from favour being a time-consuming technique, frequently involving high laboratory and materials costs. The incidence of root fracture that will be discussed later, the risk of strip perforation of the middle root during the preparation of the dowel space (Schwartz and Robbins, 2004) and the lack of aesthetic, especially in the presence of high translucent all-ceramic crowns has widely reduced the fabrication of cast post and cores

and have led to the development of more “aesthetic” posts. Moreover, temporary restorations are not effective to prevent contamination of the root canal system (Demarchi and Sato, 2002) (Sorensen and Martinoff, 1984). “Coronal leakage” is considered one of the main causes of endodontic failure, due to the contamination of the root canal system by saliva (Saunders and Saunders, 1994). Magura (1991) demonstrated that in case of coronal exposure of gutta-percha, bacteria and its by-products infect the root canal after few days. In order to prevent contamination, once the root canal treatment is completed, an immediate restoration of the tooth is recommended (Heling *et al*, 2002). Bonded restorations should be used to prevent coronal leakage (Howdle *et al*, 2002): post spaces in particular, should be restored immediately after endodontic treatment, due to the difficulty of maintaining a temporary seal (Fox and Gutteridge, 1997) (Demarchi and Sato, 2002). Thus, direct restorations have been advocated to solve these limitations (Demirel *et al*, 2005).

One of the basic principles concerning with the restoration of endodontically treated teeth is the preservation of tooth structure at radicular level during shaping and dowel space preparation to prevent weakening (Hunter *et al*, 1989). Resistance to fracture of non-vital teeth is dependent on the quality and quantity of remaining radicular dentin (Guzy and Nicholls, 1979). Clinical and laboratory studies clearly identified what are the main factors that affect fracture resistance of post-core restored teeth (Stockton, 1999) (Morgano and Milot, 1999). Premolars are more susceptible of root fracture, especially when residual sound structure is inferior to 1-2 mm (Pilo *et al*, 1998, 2000). Post length and diameter have been reported as significant factors in increasing this risk (Guzy and Nicholls, 1979) (Standlee *et al*, 1980).

Coronal tooth structure should be preserved when possible to provide resistance and retention forms for to the restoration (Cheung and Chan, 2003) (Al-Wahadni and Gutteridge, 2002). Posts restorations are requested to resist to vertical dislodging forces and to withstand to lateral and rotational forces. Resistance form is influenced by the residual tooth structure and the presence of antirotation features, the “ferrule effect” in particular, has been recognized as an important long-term success parameter when using a post: Stankiewicz and Wilson (2002) reported that 1 mm ferrule doubles tooth resistance to fracture.

The use of post-retained cores is often unnecessary in the presence of a sufficient dentin bulk (Heydecke and Peters, 2002). Since the purpose of a post is to help to retain the core, its complete elimination is an area that still requires further investigations. If

any doubt exists as to whether a post is necessary, placing it in a pulpless tooth is, anyway, a prudent alternative. Anterior teeth with a minimal loss of tooth structure can be restored conservatively (Sorensen and Martinoff, 1984), but if the tooth is planned to receive a crown, a post is often required: single-rooted teeth are loaded non-axially and in most cases the remaining tooth structure is not able to provide adequate resistance and retention for a crown without a post (Peters *et al*, 1983).

Molars should receive a cuspal coverage after endodontic treatment but sometimes cast post is not necessary if the pulp chamber may provide an adequate retention for a core build-up (Kane and Burgess, 1991). Premolars require post more often than molars: functional demands and the amount of remaining tooth structure are, once again, key factors for treatment planning. These aspects were recently confirmed by a 5 years follow-up prospective clinical study, in which the survival rate of cast vs. direct post and core restoration was evaluated revealing that the amount of remaining dentin height after preparation influenced the longevity of the restoration (Creugers *et al*, 2005).

1.5. The use of fiber post in dentistry: composition and mechanical properties

Fiber posts were first introduced by Duret at the beginning of the 90s (Duret *et al*, 1990 a, 1990b). As described by its inventors, a fiber post is made of continuous, unidirectional fibers embedded in a resin matrix and is fabricated through a semi-automated industrial process called pultrusion (Grandini, 2004). The diameter and density of the fibers (that represent the main constituent of the post) as well as the adhesion between them and the matrix, strictly influence the quality of the post and its mechanical properties.

In particular, fibers have to be pre-treated with a silane coupling agent to facilitate a chemical bond with the resinous matrix and to assemble them easily. The addition of silane during the pultrusion process has been recognized as key factor for improving the stability of the system. The role of silane as adhesion promoter in post/core restoration will be discussed in Chapter 5.

The resinous matrix, usually epoxy, is injected into the pre-tensioned fiber bundle to completely fill the spaces between fibers. As alternative, fibers are simply immersed in a resin bath. Differences in manufacturing are strictly related to the quality,

mechanical and clinical behaviour of posts (Grandini *et al*, 2005). Many *in vitro* studies were conducted to evaluate fiber posts mechanical properties with the purpose of predict their clinical performance.

Fiber posts main advantage is the variability of their modulus of elasticity depending on loading direction: in particular, when considering a transversal loading, the modulus of elasticity has a value close to sound dentin (Ferrari and Scotti, 2002). This property reduces stress transmission to root canal walls and thus the risk of vertical fractures (Asmussen *et al*, 1999). Rigid metal posts are able to resist lateral forces without distortion resulting in a significant stress transfer to the less rigid dentin, potentially causing root fracture (Bateman *et al*, 2003).

The combination of fiber posts with resinous luting cements has been recommended for their strengthening effect in roots with thin walls (Katebzadeh *et al*, 1998) (Mannocci *et al*, 1999), leading to the formation of a more homogenous structure able to mechanically interact with residual dentin and uniformly distribute stress along the entire root.

These aspects were recently confirmed by finite element analysis (FEA) evaluations of three-dimensional models in which the use of high rigidity materials, like metal post, resulted in a inhomogeneous stress distribution, increasing the risk of root fracture (De Santis *et al*, 2000). High density of fibers, absence of internal defects and strong adhesion between fibers and matrix contribute to improve posts quality and performances. A wide variety of posts is available and includes parallel-sided, smooth, tapered and serrated forms. Many different types of carbon, silica, quartz and glass fiber posts are today present in the market; carbon fibers were first used for manufacturing posts, representing the first true alternative to metal cast posts and cores. The increased demand for newer products influenced research on posts with the purpose of saving tooth structure modifying their shape and improving aesthetics.

The introduction of translucent fibers (glass or quartz) was first proposed as an alternative to dark fibers to improve aesthetics (Vichi *et al*, 2000) (Ferrari *et al*, 2001) and are today better accepted than carbon post, especially when restoring anterior teeth to provide support to all-ceramic crowns. Moreover, they permit to light passing through the post facilitating luting cement polymerization process. The mechanical behaviour of the new generation of posts does not seem to differ from traditional carbon post (Mannocci *et al*, 1999) (Grandini *et al*, 2005).

1.6. Studies comparing post systems

Post and core systems should provide sufficient retention to the final restoration, showing acceptable fracture resistance and protecting the remaining tooth. The literature on post and core restorations is abundant and numerous articles have been published investigating the above properties. Failure mode is an important factor related to the resistance of post restorations. Clinical failures may occur with all post systems, however in some situations they result in teeth that are no more restorable. Load-to-failure of the post/tooth complex has been usually applied for comparison purposes in most of the *in vitro* studies. The application of continuous or, more properly cyclic loading is thought to be representative of the forces that occur *in vivo* (Drummond and Bapna, 2003). It's well known the importance of clinical trials for producing reliable information, but the time required for completing them, together with economical and ethical aspects, tend to limitate their applicability. Thus, laboratory tests still remain useful to give first-handling information of new products. According to some recent systematic reviews, the results achieved are controversial, since the materials and the experimental design used tend to vary widely (Schwartz and Robbins, 2004) (Heydecke and Peters, 2002). However, some general conclusions can be found: cast post and cores are able to withstand higher loads, but tooth fractures are often classified as catastrophic (vertical root fracture) leading to extraction (Akkayan and Gulmetz, 2002). More favourable failure modes, involving only the core or horizontal fracture of the root have been reported when using fiber post/composite core restorations (Akkayan and Gulmetz, 2002).

In a recent literature review, Fokkinga and colleagues (2004) collected the data regarding fracture resistance of different post systems reported *in vitro* with clinical fracture mode: significantly more favourable failures were reported for FRC posts as more advisable than high fracture resistance. Most of the *in vivo* studies on root fracture of endodontically treated teeth are retrospective in nature (Heydecke and Peters, 2002). Longevity studies are difficult to compare due to differences in study design and recall periods. Nevertheless, most of them reported high percentage of survival rate for metal posts (Martinez-Insua *et al*, 1998) (Cormier *et al*, 2001). Recent studies evaluated the clinical behaviour of fiber post restorations (Ferrari *et al*, 2000) (Glazer, 2000) (Malferrari *et al*, 2003): after a follow-up between 1 and 6 yr, a low percentage of failures were reported (from 1.6 to 7.7 %). Failure can occur as “debonding” of the post,

especially when removing the temporary restoration, but it can easily be solved by repeating the adhesive procedures. Similar results were reported by Mannocci and colleagues (2005) in a 5-years prospective study in which restorations with fiber posts and composite were found to be more effective in preventing root fracture than secondary caries. Recently, Naumann (2005) reported a similar rate of failure after 2 years of service both with parallel-sided and tapered glass fiber posts and the majority of failures were restorable. The results seem to be promising, for this reason clinical acceptance of fiber posts is now higher than it was before; however, there is common evidence that longer follow-up periods are needed.

1.7. The final restoration

Many of the teeth being endodontically treated require crown coverage as a part of the tooth treatment. Post and core build-up are necessary in some of these teeth to provide support to the final restoration. Post placement and root canal treatment are traditionally considered etiological factors for root fracture thus, fixed prosthodontics procedures continue to be accomplished in significant quantities (Fuss *et al*, 2001). A relation between crown placement and survival rate of non-vital teeth with a severe loss of tooth structure was assessed (Newmann *et al*, 2003).

Some recent prospective and retrospective studies evaluated the clinical performance of fiber posts/resin restorations in endodontically treated teeth either with all-ceramic or metal-ceramic crowns with favourable results (Fredriksson *et al*, 1998) (Ferrari *et al*, 2000) (Ferrari *et al*, 2002).

However, the real need of crown coverage, as indicated in the literature (Sorensen and Martinoff, 1984) is considered still conjectural and some *in vivo* clinical studies evaluated the performance of fiber post/ resin composite restorations of non-vital teeth without crown covering (Grandini, 2004) (Creugers *et al*, 2005). It appears that the technique of direct teeth restoration is both promising and feasible, especially when transitional solutions are required. Longer clinical trials should be performed to validate the use of fiber posts and direct restorations thus, the traditional prosthetic approach to the rehabilitation of endodontically treated teeth is still advisable (Christensen, 2004).

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Chapter 2

2.1. Core build-up material selection

When a tooth is to be crowned, there must be an adequate remaining structure for creating retention and resistance forms for the final treatment. A core build-up is a restoration placed in a compromised tooth to replace the coronal portion to the extent necessary to support a crown or bridge abutment (Combe *et al*, 1999). The goal dentists are pursuing is to provide the foundation for a restoration that will endure the masticatory stress that occurs in the oral cavity for prolonged periods. Core build-up is required to provide satisfactory strength and resistance to fracture before and after crown preparation (Burke *et al*, 2000).

Several dental materials have been proposed for core build-up procedures. The ease of use of direct materials is certainly dominating their selection: despite of their mechanical properties, many other factors have to be taken into account, like working time, handling or sealing ability.

Amalgam, glass-ionomer cements (GICs) and resin composites have found application in core build-up procedures, even if most of them were not specifically developed for this purpose (Saygili and Mahmali, 2002). Two are the most critical issues regarding the choice of an ideal material: retention and strength. Core build-up must be resistant enough short-term to oppose dislodgment forces during the preparation and impression making and long-term, once the final restoration is completed.

According to the documented evidence of long-term performance of amalgam as direct core build-up material, clinicians still have confidence on its application, especially when the build-up is required to remain in function for some time as provisional restoration (Combe *et al*, 1999) (Lang *et al*, 2003). On the other hand, poor aesthetics and the lack of adhesion to tooth structure (especially when restoring badly broken-down teeth) have limited the use of amalgam in recent years (Charlton *et al*, 1992) (Eakle and Staninec, 1992).

New formulations of GICs have resulted in a substantial acceptance as alternative core materials (Wasson, 1993). Although their favourable characteristics of adhering to enamel and dentin releasing fluoride and the good clinical success, some

studies reported poor clinical properties, considering GICs not particularly suitable for build-up procedures (Gateau *et al*, 1999) (Lang *et al*, 2003).

Improvements in composite technology and the development of new dentinal adhesive systems, made resin composites the election material in the restoration of non-vital teeth with or without post placement. Aesthetics, satisfactory mechanical properties, high adhesion to dental structure and easy handling are some of the advantages of these materials (Ferrari and Scotti, 2002). Fewer complications caused by total or partial loss of the core build-up are to be expected in case of adhesively retained composite restorations (Stober and Rammelsberg, 2005). On the other hand, some intrinsic limits of resinous materials like poor dimensional stability, polymerization shrinkage, compatibility and high thermal expansion coefficient may affect their clinical behaviour (O'Keefe and Powers, 2001). There are many composite build-up materials available, most of them are either self- or dual-cured. In the presence of thick restorations, chemically curing capability is considered an advantage. In general, dual-/self-cured composites have lower bond strength to dentin than light-cured products (Sanares *et al*, 2000) (Schultz *et al*, 2000) (O'Keefe *et al*, 1998).

There has been much discussion in the literature relating to adhesive/core incompatibility: many dual- and self-curing core materials seem not to adhere properly to light- or dual-cured adhesives (O'Keefe and Powers, 2001). Recently, less incompatibility has been reported but, in general light-cured materials still achieve a more predictable bond (CRA Newsletter, 2003). Both packable and flowable composite build-up materials have been recently recommended for reducing early failure rate of non-vital teeth (Stober and Rammelsberg, 2005). Low-viscosity resins are currently promoted for superior handling properties. Thanks to their injectability, these resins are suitable for conservative tooth restorations. The physical properties of flowable composites have been recently evaluated with controversial results: high polymerization shrinkage and lower mechanical properties are reported by some Authors to limit their clinical application (Bayne *et al*, 1998) (Labella *et al*, 1999). On the contrary, a recent fracture toughness study concluded that flowables may be appropriate for conservative restorations (Bonilla *et al*, 2003). Low-viscosity composites exhibit more wear than highly filled resins, thus they are not recommended for contact supporting restorations (Clelland *et al*, 2005); nevertheless, thanks to their adaptability, they have been proven to improve the marginal seal of the restorations (Leevailoj *et al*, 2001): fewer voids

were observed when flowable liners were applied (Chuang *et al*, 2003) (Opdam *et al*, 2003).

Core material selection must include the understanding of materials' properties, but as concluded in many *in vitro* studies no one material may be considered ideal and capable of truly replacing lost tooth structure (Saygili and Mahmali, 2002). As a consequence, clinicians should have a clear knowledge of the individual patient's oral conditions to understand the advantages and limitations of different alternative materials, enhancing the best clinical outcome.

In the following SEM study the morphological properties of abutments built-up with different composite resins around fiber posts was evaluated, as representing one aspect in the selection of materials for clinical applications.

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2.2. Scanning electron microscopic evaluation of fiber post-resin core units built up with different resin composite materials.

Francesca Monticelli, Cecilia Goracci, Simone Grandini, Franklin Garcia-Godoy, Marco Ferrari. *American Journal of Dentistry* 2005; 18: 61-65.

Introduction

Many abutment teeth planned to receive fixed prosthetic treatment require a post-and-core build up because of their extensive destruction due to caries, trauma, or the afterwards of a previous restoration. When a significant horizontal loss of dental tissue occurred at the coronal level and a small ferrule can be created in the residual tooth structure, a post-and-core build up is needed with the purpose of achieving the most reliable retention for the prosthetic crown (Sorensen and Engleman, 1990) (Libman and Nicholls, 1995) (Shillingburg *et al*, 1997). Metal cast post and core has been regarded as the typical indication in this clinical situation (Shillingburg *et al*, 1997).

Many alternatives to cast posts and cores have been developed over the recent years to create a durable interaction with the residual tooth structure and to achieve dentin-like properties, in terms of hardness and modulus of elasticity. Among them, the insertion of a prefabricated post followed by the chair-side fabrication of a composite core offers the advantage of simplifying the operative procedure, by eliminating a laboratory phase (Mentik *et al*, 1993). Due to their greater similarity in elastic properties with root dentin (Ausiello *et al*, 2001) (Shaafi *et al*, 2004), post-and-core units made of a fiber post and a resinous material, have proved less prone to cause root fractures as compared with traditional cast post and cores (Sirimai *et al*, 1999) (Reagan *et al*, 1999). Moreover, if a root fracture occurs in the presence of a fiber post, it is usually less catastrophic, being mainly located in the coronal third of the root (Monticelli *et al*, 2003). The use of tooth-coloured like fiber posts, together with the wide choice of resin composites materials available for core build-up is also an aesthetic solution, allowing to satisfactorily reproducing the natural shade of the tooth (Trope and Ray, 1992).

It is still unclear what type of resin-based composite could be the best choice to build-up an abutment onto fiber posts (Ferrari and Scotti, 2002). Different materials are today available in the market and can be use for this purpose, such as self-curing (Drummond *et al*, 1999) (Ferrari *et al*, 2000a) (Akkayan and Gulmez, 2002), light-

curing, microhybrids (Ferrari *et al*, 2000) (Fredriksson *et al* 1998) (Ferrari *et al*, 2000a) (Ferrari *et al*, 2000b) (Ferrari *et al*, 2001), packables (Leinfelder *et al*, 1999) (Ferracane, 2000) (Ferrari and Scotti, 2002) and flowable resin-based composite (Ferrari and Scotti, 2002) (Ferrari *et al*, 2001) (Freedman, 2001) (Bayne *et al*, 1998), even if most of them are not specifically conceived for this purpose. These composites differ for many characteristics, such as strength and elasticity that can influence the longevity of the final restoration (Freedman, 2001) (Bayne *et al*, 1998). A recent prospective clinical trial showed that flowable composites used as core materials in combination with fiber posts performed successfully and were able to provide a good retention for a prosthetic crown over a follow-up period of 2-3 years (Monticelli *et al*, 2003).

In order to achieve a clinical success over time, an intimate union between post and core material is needed. The integrity of the adhesive interface between post surface and composite resin abutment was previously evaluated using Scanning Electron Microscopy (Ferrari and Scotti, 2002). Recently this method was applied to evaluate abutment integrity and composite resin adaptation to the post surface when the cores were built up free-hand or using pre-shaped plastic shells (Monticelli *et al*, 2004).

The present study evaluated the presence of: 1) voids/bubbles within the abutments and 2) defects at the interface with the post in post-core units prepared *in vitro* with different resin composites used as core material.

The null hypothesis that the use of different resin-based composites does not have a significant influence on the integrity of the abutment or on the morphology of the interface between post surface and core material was tested.

Materials and Methods

Fifty-five human maxillary incisors were collected immediately after extraction and stored in a physiologic solution during the course of the study (Monticelli *et al*, 2004). Only virgin teeth whose length was within 1 mm of a mean value of 23 mm were selected. The teeth were randomly divided into 11 groups of five teeth each.

All the teeth were instrumented at the working length with stainless-steel K-Files, Reamers and Hedstrom files to International Standard Organization size 60 under 5% sodium hypochlorite irrigation and obturated with laterally condensed guttapercha and a root canal sealer (AH plus, De Trey, Kostanz, Germany). With the use of a high-speed diamond bur under continuous water cooling, the crowns of the teeth were

removed 2 mm coronally the most incisal point of the cementum-enamel junction and perpendicularly to the long axis of the tooth. On all the teeth, a 2 mm butt shoulder finishing line was prepared. Guttapercha was removed from the root canal with a Peeso reamer, leaving 4 mm of root canal seal at the apical level. With a series of Largo drills the root canal of each specimen was enlarged and shaped specifically for the type of post to be placed (Aesthetic Post Plus, RTD, St. Egrevé, France). The post surface was silanized (Silane, Bisco, Schaumburg, IL., USA) for 60 sec and then gently air-dried, while the canal walls were etched with 37% phosphoric acid, rinsed after 15 seconds, and dried with paper points. One-Step (Bisco, Schaumburg, IL, USA) was used as adhesive bonding agent and the posts were luted with C&B, a dual-curing resin cement (Bisco, Schaumburg, IL, USA), strictly following manufacturer's instructions.

Composite core build-up and SEM analysis

Composite cores were built up to a height of about 6 mm measured from the buccal cemento-enamel junction. The total abutment height included 4 mm of core material and a dentin ferrule that measured 2 mm. These measurements were taken with a digital caliper (GAC, Central Islip, NY, USA) from the cementum-enamel-junction to the most coronal part of the resin composite restoration. No matrix was used to build-up the core. An incremental technique was performed and every 2 mm-thick layer of composite was cured for 20 seconds by the means of a Visilux 2 light-curing unit (3M ESPE, St. Paul, MN, USA, intensity 500 mW/cm²).

Samples were divided into 11 groups. In each group, a different composite was used to build-up the abutment. The materials tested were: Group 1: Z100, a microhybrid resin composite (3M ESPE, St. Paul, MN, USA); Group 2: Light-Core, a microhybrid resin composite (Kuraray, Morita); Group 3: Lumiglass, a microhybrid resin composite (RTD, St. Egrevé, France); Group 4: Gradia Direct, a microhybrid resin composite (GC Corporation, Tokyo, Japan); Group 5: Build it! (Jeneric Pentrom, Wallingsford, CT, USA), a fiber-reinforced dual-cure material, specifically developed for core build-ups; Group 6: Tetric Ceram, a microhybrid resin composite (Ivoclar-Vivadent, Schaan, Liechtenstein); Group 7: Biscore, a self-curing resin composite (Bisco, Schaumburg, IL, USA); Group 8: Unifil Flow, a flowable resin composite (GC Corporation, Tokyo, Japan); Group 9: DefiniteFlow, a flowable resin composite (Degussa Dental, Berlin, Germany); Group 10: Tetric Flow, a flowable resin composite (Ivoclar-Vivadent,

Schaan, Liechtenstein); Group 11: AeliteFlo, a flowable resin composite (Bisco, Schaumburg, IL, USA).

All the cores were prepared with a 1-2 mm butt should and a wall convergence of approximately 6°. Each specimen was then cut perpendicularly to its long axis in three sections at 1, 2 and 3 mm from the most coronal part of the abutment in order to assess the presence of voids/bubbles within the material and at the interface between the post's surface and the composite. Each section was gold sputtered using an Edwards Coater S150B (Balzer Ltd., London, UK) device, mounted on a metallic stub, and observed under a Scanning Electron Microscope (Jeol, JSM 6060 LV, Tokyo, Japan) at different magnifications. The presence of voids/bubbles within the abutments, as well as the finding of defects at the post-core interface were recorded and scored according to the following index (Monticelli *et al*, 2004):

Scores within the abutment. 0: No voids/bubbles; 1: few voids/bubbles of a small size (no more than 100-200 µm); 2: voids/bubbles of a size between 200-500 µm; 3: voids/bubbles of a size between 500 µm and 2 mm; 4: voids/bubbles larger than 2 mm.

Scores at the adhesive interface. 0: no discontinuities; 1: discontinuity no longer than 200 µm; 2: discontinuity between 200 and 500 µm; 3: discontinuity between 500 µm and 1mm; 4: discontinuity longer than 1 mm.

The sections were separately observed by two investigators. In case of a disagreement on the score assigned to a certain specimen, the higher one was chosen for the statistical analysis. Moreover, in the presence of multiple voids, the dimension of the largest one was measured for scoring purposes. The measuring bar of the Microscope was used for this measurement.

Statistical Analysis

The Kruskal-Wallis non parametric ANOVA was applied to test the significance of the differences among all of the tested groups. The Mann-Whitney U test was used for multiple comparisons. In multiple comparisons, the necessary correction for the floating error of the alpha value was performed. For all the statistical tests the level of significance was set at ($p < 0.05$) (SPSS Base 11.0).

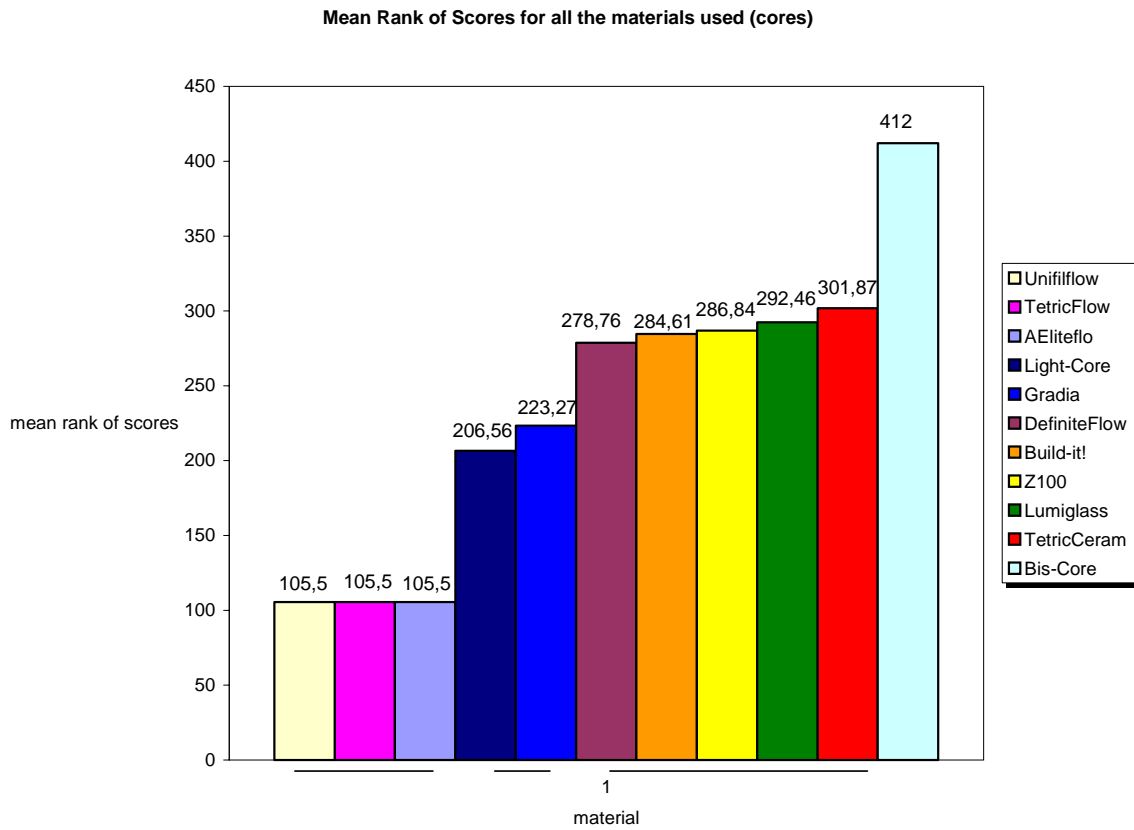
Results

The results of SEM analysis are summarized in Graph 1 (interface between post and resin material) and Graph 2 (integrity of the core). Among the 11 materials on trial, the three flowable composites (Groups 8, 10 and 11) showed the least presence of voids/bubbles within the resin abutments (Graph 2). The difference was statistically significant ($p < 0.05$). The dimensions of voids/bubbles within the cores ranged between few microns to 2 mm in diameter.

When the scores at the interface were compared among the eleven materials (Graph 1), flowables (Groups 8-11) and Z100 gave proof of a significantly better adaptation on the post than the other three materials.

In summary, Groups 8, 10 and 11 (Unifil Flow, Tetric Flow and AeliteFlo) recorded the best scores for both parameters at both the evaluated locations (Fig.1a, 2a). With the microscopic evaluation it was possible to note that only flowable sample groups did not show multiples voids/bubbles, whilst the others always showed smaller voids/bubbles than the wider that was scored. Biscore self-curing composite (Group 7) showed the lowest integrity of resin abutments and the worst continuity at the post's surface (Fig 1b, 2b).

Graph 1. Assessment of the integration between core material and fiber post. Comparison among the scores recorded for the eleven materials tested. The y-axis of the graphs indicates the mean rank assigned to each material through the statistical analysis of the ordinal data. The materials that yielded statistically similar results have their columns underlined by the same segment.



Graph 2. Evaluation of the core integrity. Comparison among the scores recorded for the 11 materials tested. The y-axis of the graphs indicates the mean rank that was assigned to each material through the statistical analysis of the ordinal data. The materials that yielded statistically similar results have their columns underlined by the same segment.

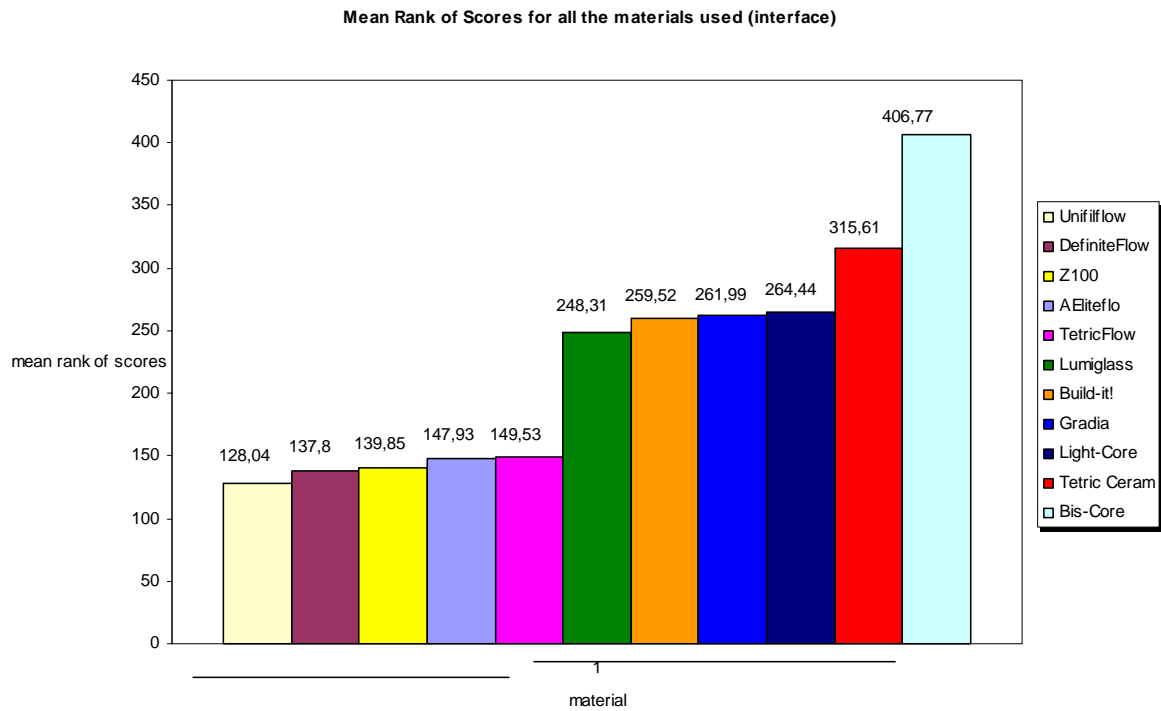


Fig 1. SEM image of Score 0 in a flowable composite material (Fig.1a): no discontinuities are detectable at the post/core adhesive interface (40x bar= 500 μ m). SEM image of Score 4 in a self-curing composite material (Fig.1b): the discontinuity along the interface extends longer than 1 mm (40x bar= 500 μ m).

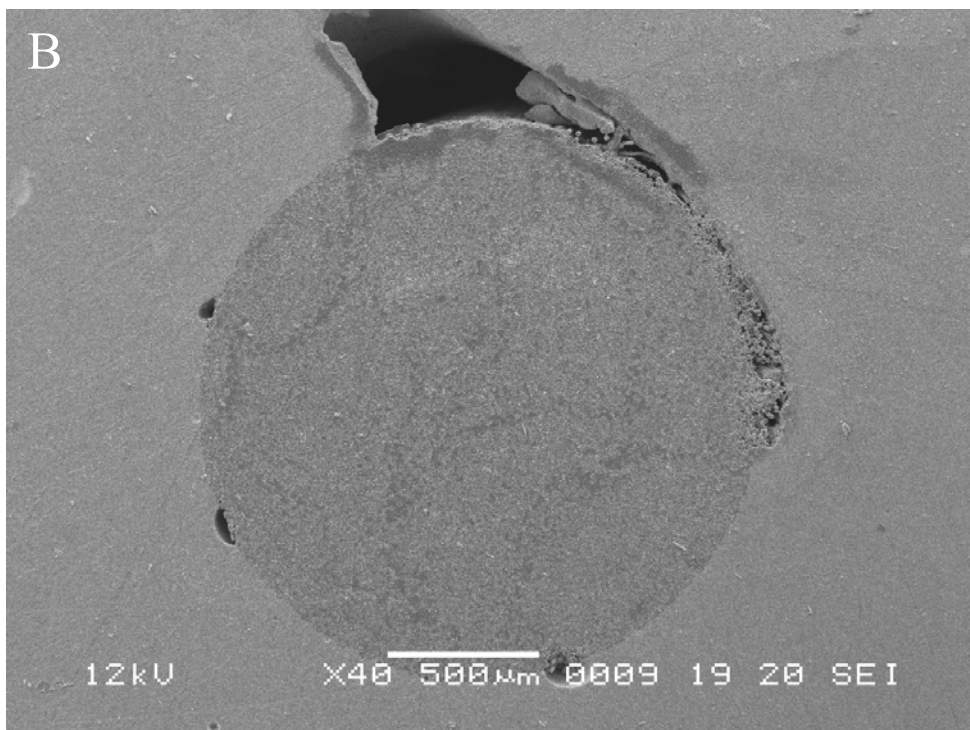
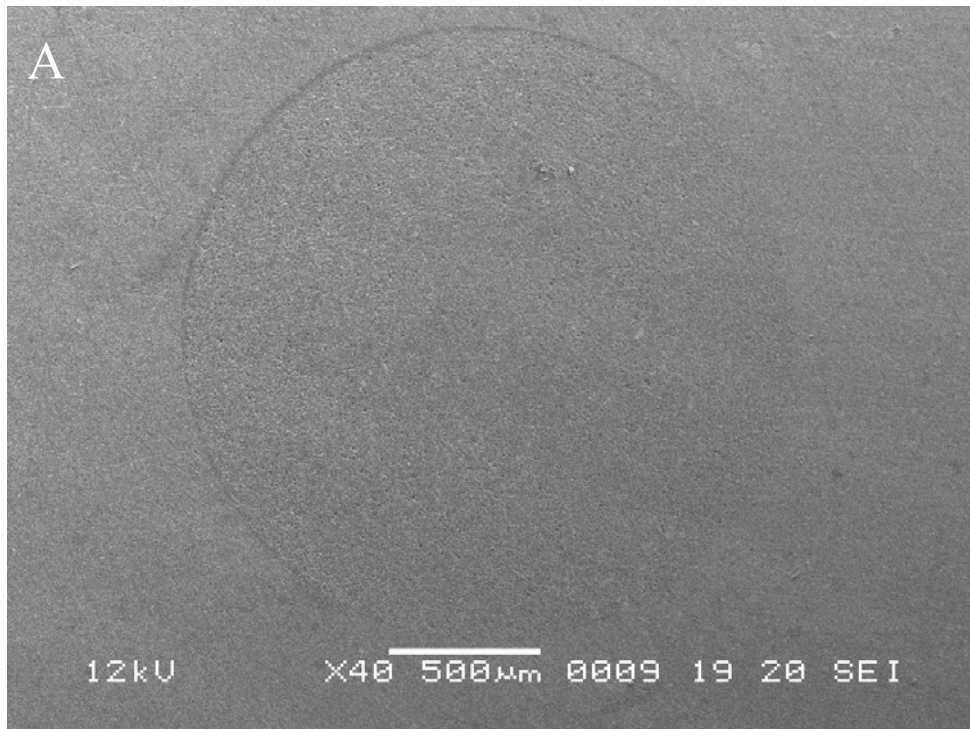
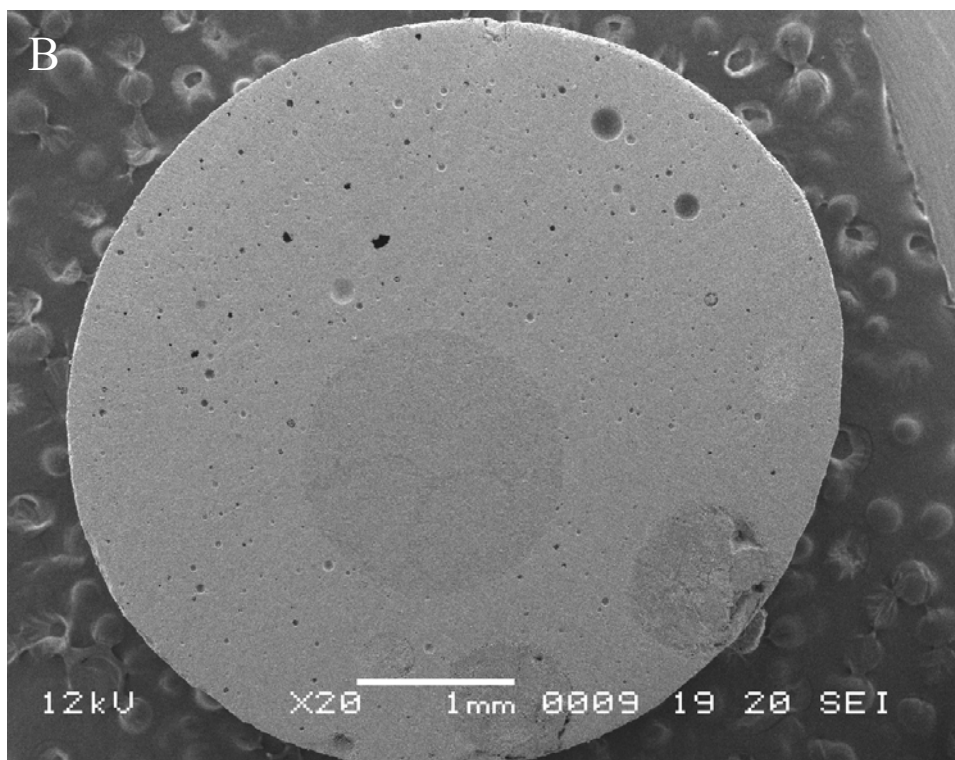
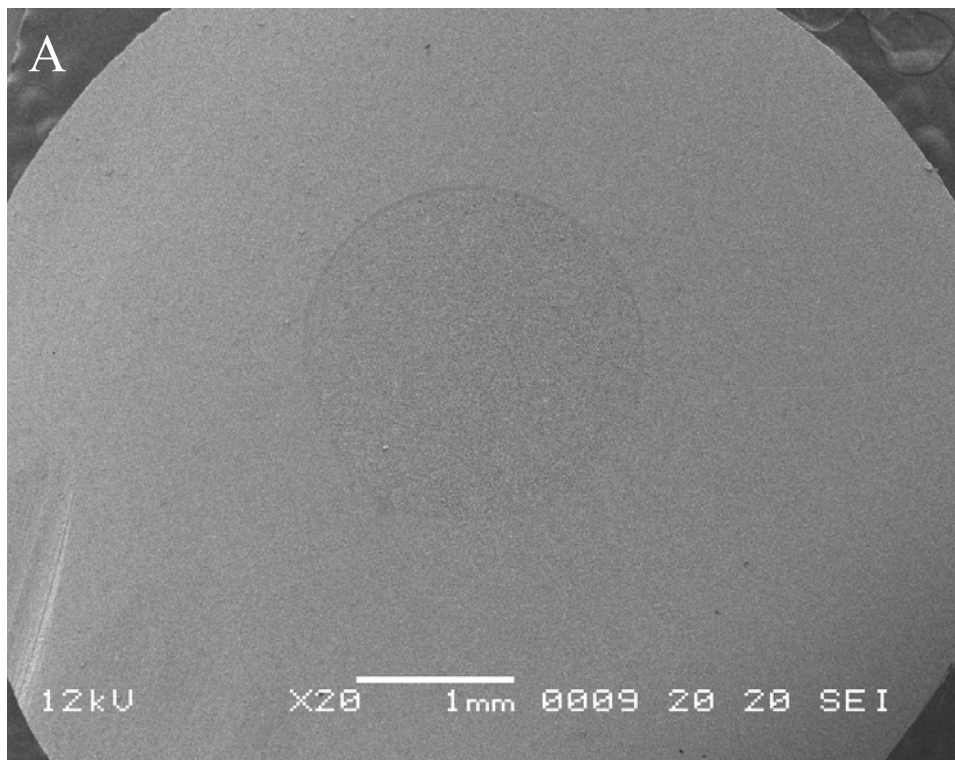


Fig 2. SEM image of Score 0 in a flowable composite material (Fig. 2 a): no defects are detectable within the abutment (20x bar= 1 mm). SEM image of Score 4 in a self-curing composite material (Fig. 2 b) where multiples voids/bubbles are evident in the composite (20x bar= 1 mm).



Discussion

Microscopic investigations usually provide a qualitative assessment of the characteristics of a material or an interface (Van Meerbeek *et al*, 2000). In the present study, a method to quantify through an ordinal scale the integrity within the resin abutment, and the adaptation of the core material on the post surface was proposed. The expression of qualitative aspects through an ordinal scale allows for the application of relatively more elaborate and powerful statistical tests, as compared with simple categorical data analysis.

The use of pre-definite scores has already been proposed for assessing the quality of endodontic and restorative procedures (Barbakow *et al*, 1999) (Van Meerbeek *et al*, 2000) (Dagostin *et al*, 2001) (Ferrari *et al*, 2001). Once an index is defined, it can be applied also in subsequent studies and, with reference to it, it is possible to compare different materials or evaluate new products.

The results of this study reveal that the use of different types of composite resins used as core materials have a significant effect on the structural properties and on the integration with the fiber post. The viscosity of the composite materials must be considered as a factor that influences the intimate adaptation and therefore the mechanical and frictional retention on the post surface.

Among the different materials for core build-up, flowable composites, because of their low viscosity, exhibited an excellent adaptation along the post's surface (Monticelli *et al*, 2004), making them potential candidates as core materials.

In the last 10 years many studies tested *in vitro* and clinically the performance of fiber posts (Fredriksson *et al*, 1998) (Ferrari *et al*, 2000a) (Ferrari *et al*, 2000b) (Akkayan *et al*, 2002) (Jordan *et al*, 1992) (Akkayan *et al*, 1998) (Asmussen *et al*, 1999) (Heydecke *et al*, 2002). None of these studies analyzed the quality of the post-core unit and the uniformity of the adhesive interface between the post and the composite at the coronal level. The integrity of the abutment and the quality of the adhesion with the post's surface can play an important role on the longevity of the final restoration of endodontically treated teeth (Ferrari *et al*, 2000a) (Monticelli *et al*, 2004) (van Noort, 2002). The presence of voids/bubbles within the resin core as well as the development of defects along the interface with the post, might negatively affect the strength of the abutment, thus increasing the risk of micro-crack propagation and fracture under

functional loading (Asmussen *et al*, 1999) (Heydecke *et al*, 2002) (Grandini *et al*, 2002) (Goracci *et al*, 2003).

In some clinical reports (Fredriksson *et al*, 1998) (Ferrari *et al*, 2000a) (Ferrari *et al*, 2000b), several small chipping of the abutment were reported. Partial detachments can alter the abutment shape and reduce the retention of the crown. However, they should not be considered as an irreversible failure, even in case of a removal of the whole core portion, since the abutment can be re-built. The procedure would be time-consuming and a replacement of a new post sometimes is needed. On the occurrence of all these clinical events, the structural integrity within the core and at the post-core interface must logically have a bearing (van Noort, 2002).

In order to avoid these clinical problems, an intimate adaptation between the composite material and the post's surface is required. Furthermore, to prevent the risk of degradation of the restoration, the resin core should completely cover the post's surface with at least 1 mm of thickness at the occlusal level. In the absence of a perfect fitting of the prosthetic crown, a fluid penetration from the coronal restoration may be expected to occur and with time to allow for hydrolytic degradation of the resin material. For a proper reconstruction of the core, it becomes crucial to cut the post to an ideal length before luting (Wolff, 1993) (Mannocci *et al*, 1999) (Grandini *et al*, 2002).

Before performing the build-up procedure, the post's surface was previously silanized: the positive effect of silane coupling agent is that it offers considerably more favourable conditions for the adhesion of the core material (Goracci *et al*, 2005). It can be speculated that a combination of low viscosity of the composite material and higher wettability of the surface achieved through silanization, may influence the good adaptation of this category of materials on the post.

Light-activated materials in general, due to their elastic properties, tend to have an easier handling and to achieve a better integration with the fiber post, thus leaving little room for bubbles/voids within the abutment and for discontinuities along the core-post interface. The reduced performances of flowable composite materials in terms of lower mechanical and physical properties when compared with traditional hybrid composites were previously evaluated (Bayne *et al*, 1998). The higher polymerization shrinkage of flowables in comparison with hybrids may indicate a superior potential interfacial stress. However their lower rigidity may play as counter-acting role (Asmussen and Peutzfeldt, 2003).

According with a recent *in vivo* study (Monticelli *et al*, 2003), when flowables were used as core materials, they are not directly exposed to mechanical forces and can provide a good support for a prosthetic crown. In addition, it is easy to work on these materials with diamond burs for crown adaptation.

On the other hand, relatively stiff self-curing and/or heavily filled light-curing hybrid resin composites would have the advantage of exhibiting more reliable mechanical properties, providing a stable support to the crown on top. However, self-curing composites which can be applied in a single bulky increment remain specifically indicated when it is possible to keep a pre-existing crown as the failure has involved only the abutment, and/or when the operator prefers to use a pre-shaped form (Ferrari and Scotti, 2002) (Monticelli *et al*, 2004).

The use of pre-shaped form was recently investigated (Monticelli *et al*, 2004). Although transparent forms supposedly allow the light to pass through, it is possible that an incomplete curing of the composite occurs in the deepest, furthest from the light source areas (Asmussen and Peutzfeldt, 2003), resulting in the development of a poor polymerized area within the material or along the interface with the post.

Apart from the material, the clinician's experience is also a critical factor that can influence the failure rate of core build-ups. Among the possible causes contributing to the development of dishomogenities in the restoration, the operator-related variables in mixing and layering the material can be mentioned (Freedamn, 2001) (Ferrari and Scotti, 2002) (Asmussen and Peutzfeldt, 2003). Flowability is regarded as an advantageous property which allows the material to be injected through a small gauge dispenser (Labella *et al*, 1999). A flowable composite, allowing an easier manipulation, should prove more forgiving to possible mistakes in handling.

On the other hand, the low viscosity of flowable composites might make them too runny to be used in combination with forms. Moreover, a procedure involving layering of the light curing composite and consecutive reapplications of the form would result in unnecessarily complicated and time-consuming steps in comparison with a free-hand incremental technique.

As regards to the materials on trial, the Aesthetic Post Plus, was chosen as being one of the most popular type of fiber posts on the market with several clinical trials already conducted on it (Fredriksson *et al*, 1998) (Ferrari *et al*, 2000a) (Ferrari *et al*, 2000b). Many types of prefabricated posts are currently available, from carbon and

quartz posts, to the wide variety of glass fiber posts (Ferrari and Scotti, 2002). The various categories of posts differ for chemical composition, physical properties, and handling. It may be interesting to carry out a similar study with the different varieties of posts available. In this study, the post surface was only silanized (Labella *et al*, 1999). It can be speculated that other post surface treatments, such as sandblasting and/or coating with an adhesive material, may enhance the integration between post and core material (Ferrari and Scotti, 2002).

For a deeper and more complete understanding of the properties of composites that can be used as core materials, microscopic data should be complemented with the results of microtensile bond strength tests and ideally with the findings of *in vivo* tests.

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Chapter 3

3.1. Selection of the clinical core-build-up procedure

The restoration of pulpless teeth is a routine procedure in everyday clinical practice. Prosthetic coverage continues to be accomplished in significant quantity. Many of the teeth being crowned require post and core build-up before prosthetic therapy for replacement of the missing coronal portion of the tooth. With the introduction of contemporary restorative materials, there was a clearly observable movement toward the use of fiber posts and direct resin-based composite build-ups.

A proper core restoration of the tooth is a prerequisite for ensuring good functional and aesthetic outcomes.

Different materials as well as restorative techniques have been proposed in the recent years to improve the therapeutic possibility. The incremental technique is considered a reliable procedure to reduce volumetric contraction of composites during polymerization (Torstenson and Oden, 1989) (Lopes *et al*, 2004) and is commonly applied to build-up the core around fiber posts. Depending on the properties of the composite material and its handling, alternative operative techniques may be selected: the use of core-forms in association with self-cure or dual-cure composites is claimed to fasten chair-side procedures. Forms are pre-shaped, facilitating the subsequent prosthetic preparation of the abutment and permitting a single-bulk material application. There is always a concern about the possible microleakage from polymerization shrinkage of a large volume of material cured in one increment, even if it should not be considered a factor if the core will receive a crown coverage. Tachibana and colleagues (2004) recently reported no significant differences in contraction stress of composites when incremental and bulk build-up restorations were compared.

Apart from the material, however, practitioner's experience and the amount of residual coronal structure (to ensure retention of the restoration) are of crucial importance to the quality of core build-ups. The following SEM study evaluated the influence of two different direct restorative techniques on the morphological aspect of post and core build-up restorations.

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3.2. Micromorphology of the fiber post-resin core unit: a scanning electron microscopy evaluation.

Francesca Monticelli, Cecilia Goracci, Marco Ferrari. *Dental Materials* 2004; 20: 176-83.

Introduction

It often happens that teeth meant to serve as abutments in fixed prostheses are extensively destroyed as a result of caries, trauma, or of a previous restoration. In the face of a conspicuous horizontal loss of the clinical crown, such that only a ferrule of minimal thickness could be created in the remaining tooth structure, a post-and-core build-up is needed (Sorensen and Engleman, 1990) (Libman and Nicholls, 1995) (Shillingburg *et al*, 1997). In particular, the described clinical situation has traditionally been regarded as a specific indication for a cast gold post-and-core (Shillingburg *et al*, 1997).

However, some alternatives to cast posts and cores have been developed over the recent years, such as the use of prefabricated posts in combination with composite resins to directly build up the core. This technique offers the advantages of simplifying chair-side procedures. In addition, the use of the new tooth-coloured fiber posts (Mentik *et al*, 1993) (Trope and Ray, 1992) in combination with a matching resin-based material for core build-up, is expected to yield a more natural and aesthetic appearance of the final restoration, as compared with a cast post-and-core (Trope and Ray, 1992). As regards mechanical properties, cores directly built-up with composite resin have shown a fracture resistance comparable to that of cast gold cores (Reagan *et al*, 1999) (Sirimai *et al*, 1999) (Akkayan and Gulmetz, 2002) (Fredriksson *et al*, 1998).

For building up the abutment after luting a fiber post, all the types of composites, from the microhybrids (Ferrari *et al*, 2000) (Ferrari *et al*, 2000a) (Ferrari *et al*, 2001) to the flowables (Ferrari *et al*, 2001a) (Ferrari *et al*, 2002) in the light-activated or the self-curing formulation, could virtually be used. These materials differ among themselves in terms of strength, stiffness or elasticity, and other properties that can affect the longevity of the final restoration (Ferrari *et al*, 2002) (Asmussen *et al*, 1999).

The question as to the type of composite that performs best in directly building up a core on a posted tooth has not yet been systematically addressed.

Furthermore, pre-shaped shells have recently been marketed, which are claimed to speed up and simplify the procedure of building up the abutment. However, it has not yet been verified whether the addition of this matrix as a support in composite layers stratification actually results in an improved structural integrity of the abutment, or in an enhanced adaptation of the core material around the post.

The method of scanning electron microscopy has already been applied to investigate the morphologic characteristics of post-and-core units made of resin-based materials (Ferrari *et al*, 2002). In the present study, scanning electron microscopy was used to get an insight of the structural homogeneity of cores made up of different composite resins, with and without the support of preformed shells, as well as to assess the degree of structural continuity between the build-up material and the post. More precisely, the study was aimed at detecting and quantifying the presence of voids/bubbles and gaps, both within abutments made with and without pre-shaped shells, and at the interface between the post and the build-up material. It tested the null hypothesis that different restorative materials and build-up techniques cannot determine morphological differences of the abutments.

Materials and Methods

Forty human maxillary incisors selected for the study were placed, right after the extraction, in a physiologic solution, where they were kept for the whole experimental period. Only sound teeth with an average length of 23 mm \pm 1mm were included in the sample (Jordan *et al*, 1992).

Endodontic treatment was performed through stepwise filing with reamers and Handstrom files to Int. Standard Organization size 60. A 2.5% sodium hypochlorite solution was used to deterge the root canals, which were finally obturated with laterally condensed guttapercha and a resin sealer (AH plus; De Trey, Kostanz, Germany). With a diamond bur mounted on a high-speed handpiece and used under continuous water cooling, the teeth were decoronated 2 mm coronally to the most incisal point of the cementum-enamel junction (CEJ), and sectioned perpendicularly to the long axis of the tooth. On all of the teeth a 2 mm butt shoulder finishing line was prepared. Guttapercha was removed from the root canals with a Largo drill, leaving 4 mm of root canal filling in the apical portion. Several drills provided in a kit together with the posts were used in the recommended sequence to prepare the canal for post-insertion.

Then, in each tooth a fiber post (Aesthetic Post Plus, St. Egreve, France) was placed. In order to bond the post to the root canal walls, the dentin at this level was etched with 36% phosphoric acid, rinsed after 15 s, and dried with paper points. The One-Step adhesive system (Bisco, Schaumburg, IL, USA) was applied as a bonding material, and the C&B resin cement (Bisco) was used to lute the post. The post surface was silanized (Bisco) for 5 min. All of the materials were handled according to the manufacturers' instructions.

Composite core build up

The specimens were randomly divided into eight groups of five teeth each. On the posted roots, composite cores were built up to reach an abutment height of 6 mm, as measured from the buccal CEJ. Two millimetres of material created a ferrule on the dentin substrate, whereas the remaining 4 mm of composite actually made up the abutment. In each group of teeth a different composite resin was tested as a core build-up material. Three hybrid composites, Z100 (3M ESPE, St Paul, MN, USA, Group 1), Lumiglass (RTD, Group 2), and Gradia (GC Corporation, Tokyo, Japan, Group 3) were compared to Build-it! (Jeneric Pentron, Wallingsford, CT, USA), a fiber-reinforced dual-cure material, specifically developed for core build-ups (Group 4).

In the specimens of Groups 5 to 8, a preformed shell (Composipost Core Form, RTD, St. Egreve, France) was placed onto each posted root. According to the dimensions of the residual tooth portion, a shell of appropriate size was chosen for each specimen. The shell was then completely filled with a composite resin, the excess material was removed, and the resulting abutment was light-cured through the transparent shell with a Visilux light unit (3M ESPE,). The light was irradiated on the buccal, lingual, mesial, and distal aspect of the shell, each time for 40 s. The same four composites used to build up the cores in the absence of any matrix, were tested also in combination with the preformed shells. They were Z100 (3M ESPE, Group 5), Lumiglass (RTD, Group 6), Gradia (GC Corporation, Group 7), Build-it! (Jeneric Pentron, Group 8). The materials were always used strictly following manufacturers' instructions.

The cores built up without any shell (Groups 1-4) had to be slightly prepared to make their shape apt to support a hypothetical crown, whereas the abutment built up within the shells (Groups 5-8) already presented with the adequate shape.

On all of the abutments a 1-2 mm butt shoulder was prepared, and a wall convergence of approximately 6° was created.

Then, the specimens were cut perpendicularly to their long axis 1-, 2- and 3mm from the top of the abutment. Each section was gold sputtered with an Edwards Coater S150B device (London, UK), mounted on a metallic stub, and observed under a Philips 505 Scanning Electron Microscope (Amsterdam, The Netherlands) at different magnifications. The presence of voids/bubbles within the abutments, as well as the finding of gaps at the post-core interface were recorded, and scored according to indexes thus defined:

Scores within the abutment: 0: no voids/bubbles; 1: few voids/bubbles of a small size (100-200 μm); 2: voids/bubbles of a size between 200-500 μm ; 3: voids/bubbles of a size between 500 μm and 2 mm; 4: voids/bubbles larger than 2 mm (Fig. 1).

Scores at the adhesive interface: 0: no gaps; 1: gap no longer than 200 μm ; 2: gap between 200 and 500 μm ; 3: gap between 500 μm and 1mm; 4: gap longer than 1 mm (Fig. 2).

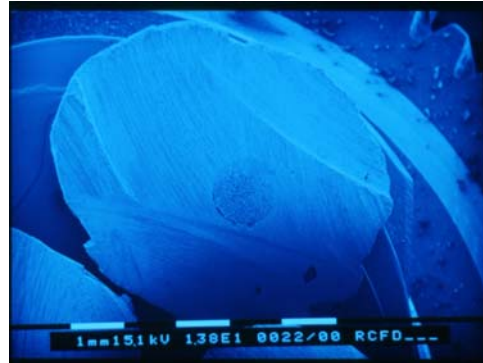
The sections were separately observed by two investigators. In case of a disagreement between the two investigators on the score assigned to a certain specimen, the worse score was chosen for the statistical analysis.

Statistical Analysis

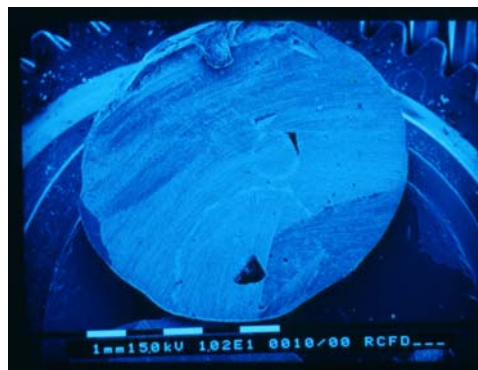
The Kruskal Wallis Non Parametric Analysis of Variance was applied to test the significance of the differences among all of the tested groups. The Mann-Whitney U test was used for post-hoc comparisons. The Mann-Whitney U test was also applied to verify the significance of the differences between the results yielded by one same material used with and without shells (comparison between Group 1 and 5, Group 2 and 6, Group 3 and 7, Group 4 and 8). For all the statistical tests the level of significance was set at $p < 0.05$.



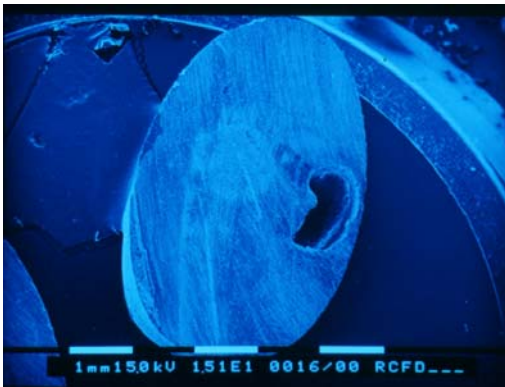
Score 0



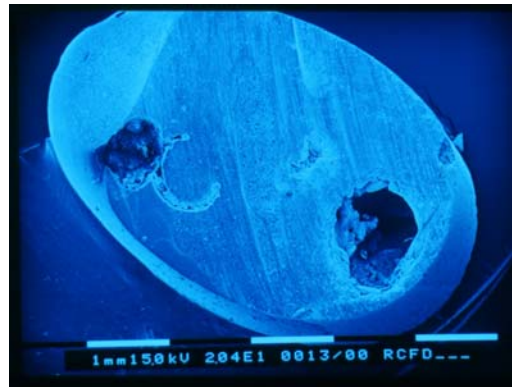
Score 1



Score 2

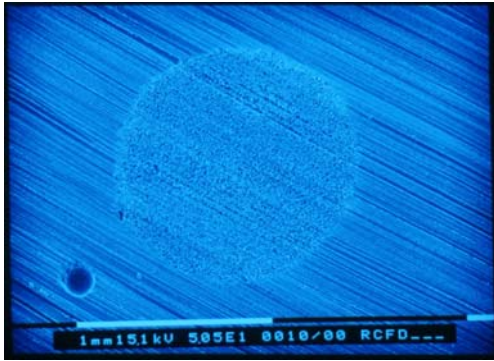


Score 3

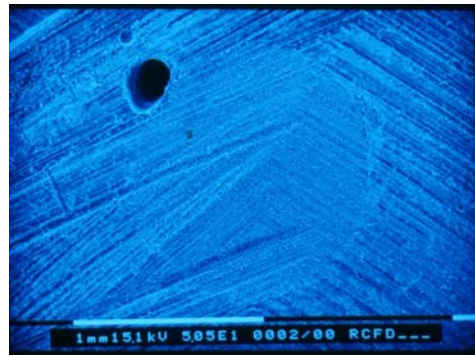


Score 4

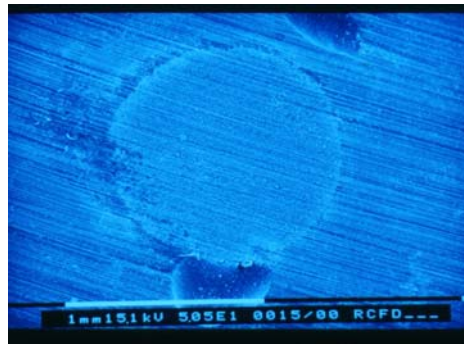
Fig.1. Abutment scores. (a) Score 0: no voids/bubbles are detectable within the abutment; (b) Score 1: only few voids/bubbles of a small size (100-200 μm) are visible; (c) Score 2: voids/bubbles of a size between 200-500 μm are present; (d) Score 3: voids/bubbles of a size between 500 μm and 2 mm are seen within the abutment; (e) Score 4: voids/bubbles larger than 2 mm are evident.



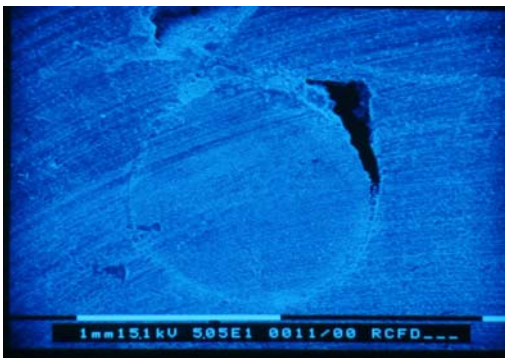
Score 0



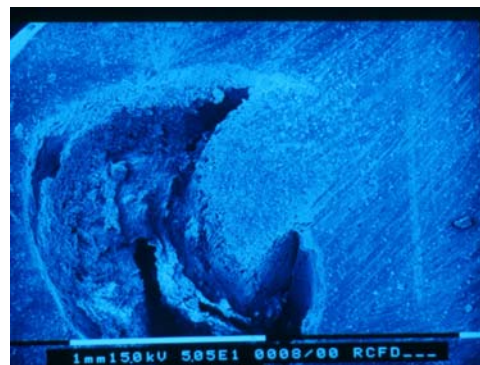
Score 1



Score 2



Score 3



Score 4

Fig. 2. Scores at the adhesive interface. (a) Score 0: no gap is detectable. (b) Score 1: a gap no longer than 200 μm is visible at the interface. (c) Score 2: a gap between 200 and 500 μm long is evident. (d) Score 3: a gap extending for 500 μm – 1 mm along the interface is detected. (e) Score 4: the gap along the interface extends longer than 1 mm.

Results

Among the four materials used without shells, Gradia (Group 3) showed the least presence of voids/bubbles within the resin abutments. The difference was statistically significant (Fig. 3a). When the scores at the interface were compared among the same four materials, Z100 (Group1) gave proof of a significantly better adaptation on the post than the other three materials (Fig. 3b).

When plastic shells were used, the specimens of Group 8 (Build-it!) yielded the best result, showing a very good structural integrity of the abutments, and a continuous adaptation of the core material around the post (Figs. 4a and b). Also Gradia (Group 7) performed significantly better than Lumiglass (Group 6) as far as the continuity of the material around the post was concerned (Fig. 4b).

The comparison between the results given by each composite used with and without shell revealed that Z100, Lumiglass, and Gradia yielded a better core homogeneity and a more satisfactory adaptation around the post when they were used without shells. With Build-it! similar results in terms of core integrity were achieved either with or without a shell, whereas the presence of the matrix significantly improved the adaptation of the material around the post.

Fig. 3a

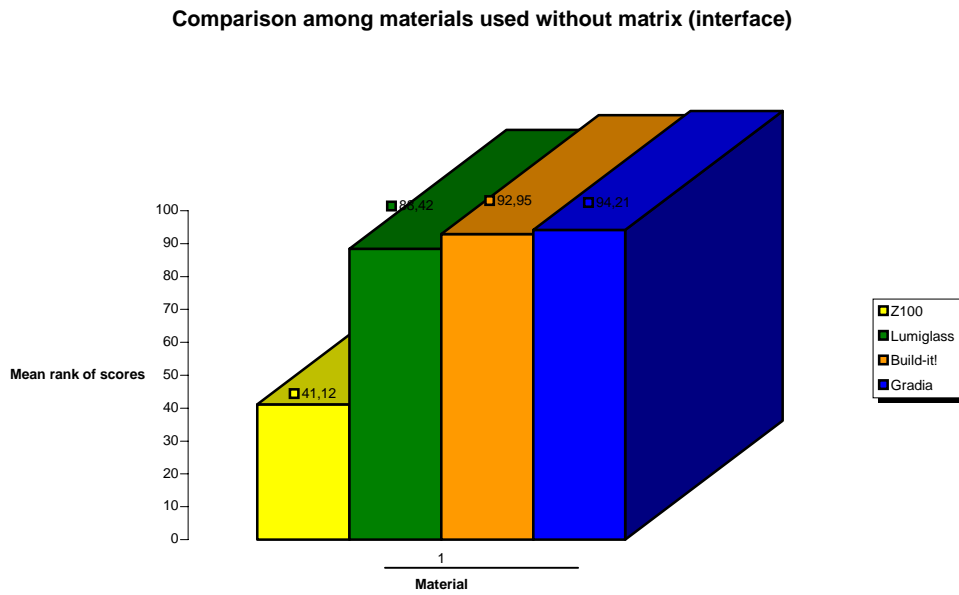


Fig.3b

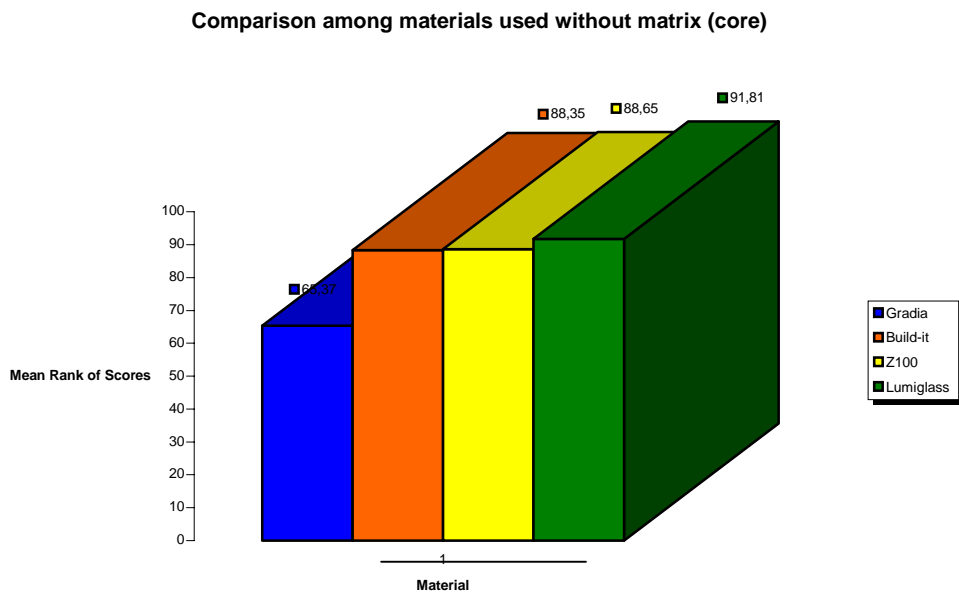


Fig.3. Comparison among the scores recorded for the four materials, tested without the use of a plastic shell. The y axis of the graphs indicates the mean rank that was assigned to each material through the statistical elaboration of the ordinal data. The materials that yielded statistically similar results have their columns

underlined by the same segment. (a) Evaluation of the core integrity. (b) Assessment of the integration between core material and fiber post.

Fig. 4a

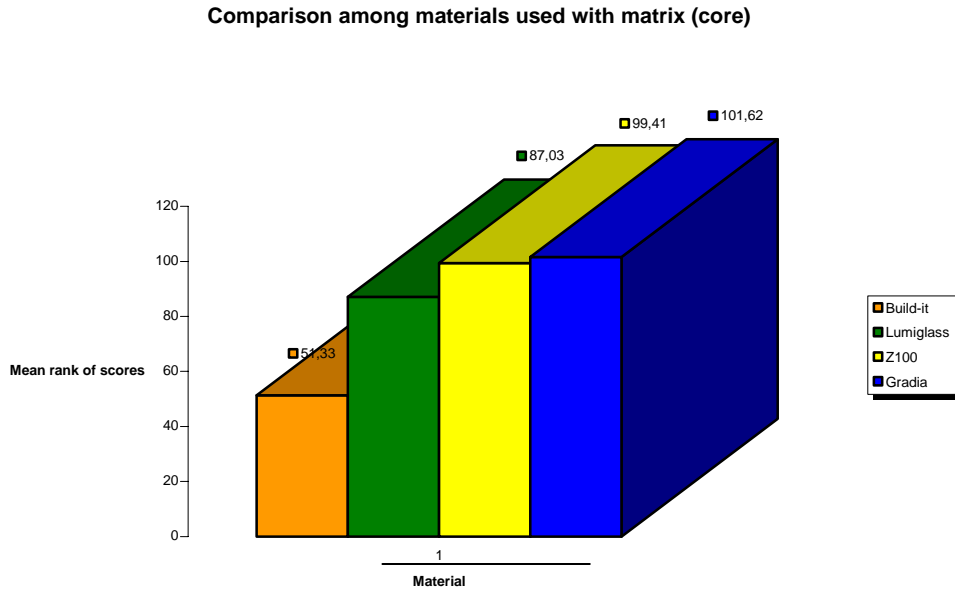


Fig. 4b

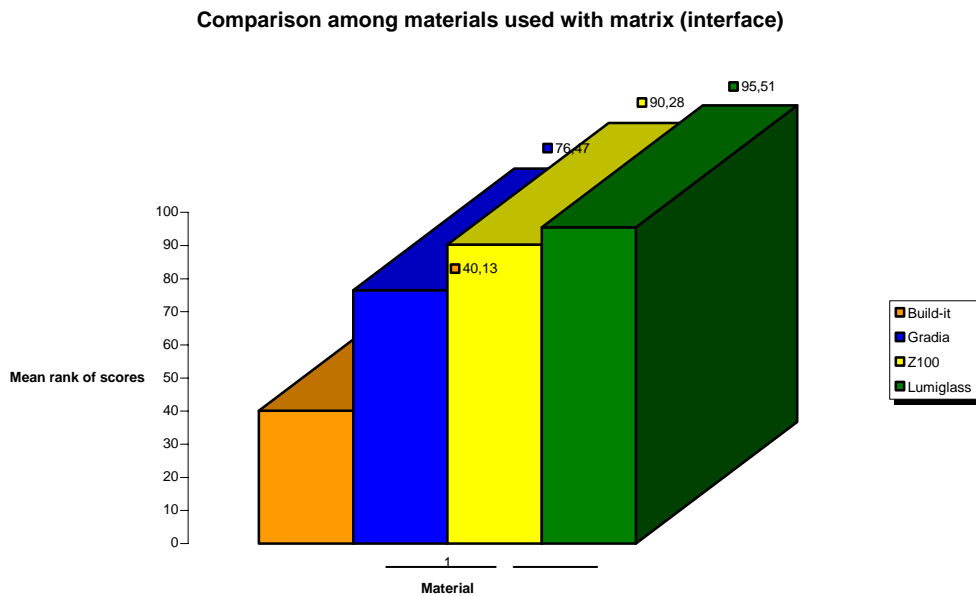


Fig. 4. Comparison among the scores recorded for the four materials, tested with the use of a plastic shell. The y axis of the graphs indicates the mean rank that was assigned to each material through the statistical elaboration of the ordinal data. The materials that yielded statistically similar results have their columns underlined by the same segment. (a) Evaluation of the core integrity. (b) Assessment of the integration between core material and fiber post.

Discussion

Among the several studies that over the past 10 years have evaluated *in vitro* (Duret *et al*, 1990) (Reagan *et al*, 1999) (Sirimai *et al*, 1999) (Asmussen *et al*, 1999) (Akkayan and Caniklioglu, 1998) (Drummond *et al*, 1999) (Mannocci *et al*, 1999) (Akkayan and Gulmez, 2002) and *in vivo* (Fredriksson *et al*, 1998) (Ferrari *et al*, 2000) (Ferrari *et al*, 2000a) (Ferrari *et al*, 2002) the performance of fiber posts, none has so far focused on the homogeneity of the resin abutment, and on the continuity of the bonding interface between the post and the core material. These structural characteristics of the fiber post–resin core unit can play an important role on the longevity of the final restoration of the endodontically treated tooth.

The presence of voids/bubbles within the resin cores and the development of gaps along the interface with the post negatively affect the strength of the abutment, thus increasing the risk of its fracture under the functional loading (Ferrari *et al*, 2001).

The occurrence of limited ditching of the core portion is usually overlooked in clinical reports, as it is considered unlikely to result in a clinical failure of the restoration. However, the detachment of small fragments of material from the core can alter the shape or the profile of the abutment, thus lowering the retention of the crown.

Even in the case the fracture has taken off the most part or the total of the core portion, the event still does not have to be considered as an irreversible failure of the restoration, since the abutment can be re-built. However, the procedure would be time-consuming, and possibly also involving the replacement of the post.

In order to strengthen the post-and-core unit, it seems advisable that the coronal end of the post be surrounded by the core material for at least 1 mm of length. In order to achieve this proper integration between post and core, the step of cutting the end of the post protruding from the root becomes a critical step (Grandini *et al*, 2002).

Different types of composite resins currently on the market could be used to build up a core onto a fiber post. Relatively stiff self-curing resins would have the advantage of providing a stable support to the crown on top. On the other hand, more elastic composites, such as flowable and light-activated materials, tend to have an easier handling and a better integration with the fiber post surface, thus leaving little room for bubbles/voids within the abutment, and for discontinuities along the core-post interface.

In addition, it is easy to prepare these materials with diamond burs for crown adaptation.

Self-curing resin should in any case be preferred in the presence of a plastic shell. Although transparent shells are claimed to let the light pass through, however the results of the present study seem to point out that the light-curing process of the core material can be negatively affected by the interposition of the plastic matrix. That would explain the finding of a more frequent occurrence of voids/bubbles within the abutments and of interfacial gaps along the post surface, when the core was built up in the presence of a shell. In this investigation, Build-It!, a reinforced resin composite, was the only material to yield better results when used in combination with shells. As a matter of fact Build-it! is, among the tested materials, the only one specifically conceived for core build-ups in combination with shells, whereas the other three materials on trial are most commonly used for direct restorations.

Microscopic investigations usually provide a qualitative appraisal of the characteristics of a material or an interface (Van Meerbeek *et al*, 2000). In the present study, an original method was proposed to quantify through an ordinal scale the integrity within the resin abutment and the adaptation of the core material onto the post surface. The expression of qualitative aspects through an ordinal scale allows for the application of relatively more elaborate and powerful statistical analysis. In addition, once an index is defined, it can also be applied in subsequent studies and, with reference to it, it becomes possible to compare different materials or to evaluate new products. The use of pre-definite scores has recently been proposed for assessing the quality of endodontic and restorative procedures (Barbakow *et al*, 1999) (Dagostin *et al*, 2001).

The Aesthetic Post Plus type of fiber post (RTD, St. Egrevé, France) was chosen as being one of the most popular on the market, and because several clinical trials have already been conducted on it (Ferrari *et al*, 2001) (Ferrari *et al*, 2002) (Ferrari *et al*, submitted). Many types of prefabricated posts are currently available to the clinician, from the carbon and quartz posts, to the wide variety of glass fibers and resin matrix posts. The various categories of posts differ for chemical composition, physical properties, and handling. It should be of interest to carry out an investigation with the same purpose as the present study on the different varieties of posts available.

In this trial the post surface was only coated with a silane agent. It can be expected that other types of post surface treatments, such as sandblasting and/or coating

with an adhesive material, would be able to enhance the integration between post and core material.

For a deeper more complete understanding of the properties of resin composites that can be used to build-up a core, the microscopic data should be complemented by the results of microtensile bond strength tests, and ideally by the findings of *in vivo* tests.

The results of this study rejected the null hypothesis tested: type of restorative materials and techniques determine morphological significant differences.

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Chapter 4

4.1. Clinical evaluation of fiber posts and core build-up restorations

It's well known the importance of clinical trials for producing reliable information on a material or an operative technique. The time required for completing an *in vivo* study together with economical and ethical aspects, tend to limit their applicability. Thus, laboratory tests still remain useful to give fresh information of new products.

Most of the recent clinical studies on posts have examined teeth restored with fiber posts, evaluating the survival rate and the main cause of failure of the treatment (Ferrari *et al*, 2000) (Glazer, 2000). In a 4-year retrospective study, Paul and Werder reported 100% success rate in posted teeth restored with direct composite cores before crowning (2004).

When prospective clinical studies are performed, operative procedures, materials and number of patients to be treated are evaluated in advance to obtain a rigorous and repeatable protocol. Although the studies on fiber posts have relatively short recall periods, the initial results seems to be promising revealing a high survival rate and a low percentage of failure mainly due to debonding (Ferrari *et al*, 2002). Malferrari and colleagues recently reported 98.2% success in teeth restored with quartz fiber posts after 2.5 years of clinical service (2003).

Crown coverage after post placement and core build-up is the traditional choice to restore structurally compromised endodontically treated teeth (Fredriksson *et al*, 1998) (Ferrari *et al*, 2000) (Ferrari *et al*, 2000a).

In this chapter, a prospective study on pre-prosthetic post and core restorations was performed to ascertain if the promising *in vitro* data of the previously reported investigations would be reflected in a satisfactory clinical performance.

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4.2. Clinical Behaviour of Translucent Fiber Posts: a 2-year Prospective Study

Francesca Monticelli, Simone Grandini, Cecilia Goracci, Marco Ferrari. *International Journal of Prosthodontics* 2003;16: 593-6.

Introduction

The restoration of endodontically treated teeth with fiber-reinforced post systems has been drawing the attention of a constantly growing number of clinicians. The progresses in the technology of fiber-reinforced materials, addressing structure, shape, and optical properties of the posts, have led to the development of materials that have overcome some of the limitations of metallic posts (platinum, alloys or titanium), concerning aesthetic appearance, mode of failure, and clinical performance (Duret *et al*, 1990) (Frejlich and Goodacre, 1992) (Siebert and Thiel, 1996) (Rinaldi, 1996) (Mutobe *et al*, 1997) (Paul and Sharer, 1998) (Takehashi *et al*, 1998) (Sakkal, 1998) (Kwaiatkowski and Geller, 1999) (Koutayas and Kern, 1999) (Hochman and Zalkind, 1999) (Asmussen *et al*, 1999) (Drummond *et al*, 1999) (Vichi *et al*, 2000) (Heydecke *et al*, 2002) (Akkayan and Gulmez, 2002).

As far as aesthetics is concerned, several brands of translucent glass fiber posts have been put on the market and are being preferred to carbon fiber posts, especially for anterior roots meant to provide support to an all-ceramic coronal restoration. Also, the mechanical behaviour and the related mechanisms of failure of fiber post were recently compared to those of metallic posts (Reagan *et al*, 1999) (Ukon *et al*, 2000) (Cornier *et al*, 2001). While metallic posts tend to produce an irreversible root fracture on failure, if a root fracture occurs in the presence of a fiber post, it is usually located more coronally and is more easily retreatable. This type of failure may be due to the greater amount of tooth structure that must be removed when a metallic post is placed (Stankiewicz and Wilson, 2002).

In general, in the need for endodontic retreatment (Sakkal *et al*, 1998), fiber posts are more easily removed than the metallic or ceramic ones.

Only a small number of case reports (Ferrari *et al*, 2000) (Ferrari *et al*, 2001) and few retrospective studies (Fredriksson *et al*, 1998) (Ferrari *et al*, 2000a) (Ferrari *et al*, 2000b) have so far been published on fiber posts. It then seemed of interest to conduct a

prospective clinical trial on three types of translucent fiber posts currently available on the market.

Flowable composites were used for core build-ups in order to verify whether these materials, which have proved capable of satisfactory integration with both the post surface and the residual coronal structure, are also able to efficiently withstand the functional loads transmitted through the crown placed on top (Ferrari *et al*, 2002). The restorations thus made were followed-up clinically for up to 24 months, with the aim of testing the null hypothesis that there is significant difference in the clinical service yielded by the three types of posts, and the use of materials from the same manufacturer when preparing fiber post-composite core units does significantly add to the clinical performance of the restoration.

Materials and Methods

Over 3 years, 225 patients treated in the Department of Restorative Dentistry were judged eligible for the study; they presented with a premolar that, according to the clinical and radiographic exam, needed endodontic treatment, followed by restoration with a fiber post and a single unit all-porcelain crown. This treatment was planned for premolars that presented with only two coronal walls left. Because the palatal root has a more favourable anatomy for receiving a post, in each premolar only one post was placed (Fig. 1).

On the selected teeth, endodontic treatment was performed, and the roots were prepared for receiving a post. The root canal walls were enlarged with low-speed burs provided by the manufacturer and a post space about 9 mm deep was created. Then, the post was tried into the root canal and cut to the adequate length with a diamond bur.

Seventy-five patients treated with Aesthetic Plus posts (RTD; group 1), 75 patients who received DT Posts (RTD; group 2), and 75 patients who had FRC Postec posts (Ivoclar-Vivadent; group 3) placed were included in the study. The age of the patients ranged between 18 and 78 years (mean 51 years). The distribution of the different types of premolars among the three groups is shown in Table 1.

In groups 1 and 2, fiber posts were bonded with the One-Step bonding system and the Duo-Link dual-cure resin cement (Bisco Co., Schaumburg, IL, USA). The root dentin was etched with 37% phosphoric acid (Uni-Etch, Bisco) for 15 seconds, rinsed with a water spray, and gently air-dried. The remaining humidity was absorbed with

paper points. Then, the One-Step adhesive was applied in three consecutive coats with a microbrush, air-dried, and the excess left in the post space was removed using a paper point. The bonding material was light-cured for 20 seconds with a Visilux 2 light-curing unit (3M ESPE, St. Paul, MN, USA, intensity 500 mW/cm²). The two components of the Duo-Link resin cement were mixed together and brought into the canal with a lentulo drill. Lastly, the post was inserted, and the resin cement was light-cured through the translucent post for 40 seconds.

In group 3 Excite DSC self-curing adhesive and Multilink resin cement (Ivoclar-Vivadent, Schaan, Liechtenstein) were used for the luting procedure, as they were materials produced by the same manufacturer as FRC Postec posts. The adhesive was applied in two consecutive layers with a brush tip, and dried with an air blast. Then, the excess left in the post space was removed using a paper point. After etching with phosphoric acid and applying two consecutive layers of Excite DSC with the self-activating microbrush, MultiLink resin cement base and catalyst were mixed and carried into the post space with a lentulo. The post was finally inserted into the canal and left undisturbed until the complete setting of the cement occurred.

Periapical radiographs were taken to check for the accuracy of the endodontic treatment and the post placement.

Following this, the core build-up was performed using the proprietary flowable resin composite material (AEIite Flo, Bisco in groups 1 and 2; Tetric Flow, Ivoclar-Vivadent, in group 3).

Then, the abutment was prepared with diamond burs, and a polyether impression (Impregum, 3M ESPE) was taken for the fabrication of a single-unit all-porcelain crown (Empress 2, Ivoclar-Vivadent).

Six, 12, and 24 months after post insertion, patients were recalled for a radiographic assessment of the results of endodontic treatment, as well as for a clinical evaluation of the restoration's condition. Clinically, treatment was considered successful when, in the absence of any radiologic sign of periapical pathology, the post and core were soundly retained and neither a root nor a post fracture had occurred. Mobility and possible opening of margins were carefully examined. When provisional restorations were in service, the integrity of the resin core was checked. Also, the occurrence of any post dislodgment during debonding of the crown was recorded.

Two observers carried out the clinical and radiologic examinations independently. These two investigators were different from the operator who had

performed the restorations and were blinded as to the types of materials that had been used. No episodes of disagreement between the two examiners occurred in the evaluation of clinical and radiological signs.

The Actuarial Life Table statistical analysis and the Mantel-Haenszel survival curve were elaborated at a 95% level of confidence.

Table 1. Distribution per jaw of the teeth in the 3 experimental groups.

<i>Type of post</i>	<i>Maxilla</i>	<i>Mandible</i>	<i>Total</i>
AP Posts	34	41	75
DT Posts	33	42	75
FRC Postec	49	26	75



Fig. 1. A first premolar restored with a fiber post and a porcelain crown at 2-year recall.

Results

Among all of the 225 teeth on trial, 14 (6.2%) failures were reported during the follow-up period, similarly distributed among the three tested groups (Table 2). Eight

restorations failed because of debonding of the post (3.5%). Six (7.2%) of the recorded failures were due to the recurrence of endodontic periapical lesions, as revealed by the radiographic examination. Endodontic failure was reported but not regarded as post failure.

Table 2. Frequency and type of failures recorded in each experimental group (n= 75).

<i>Type of post</i>	<i>Debonding</i>	<i>Periapical pathology</i>
AP Posts	3	3
DT Posts	2	2
FRC Postec	3	1

Discussion

Some retrospective studies on the clinical performance of fiber posts have recently appeared in the literature (Ferrari *et al*, 2000) (Ferrari *et al*, 2000a). However, most of them were unable to ensure an adequate control of all the variables that might come into play under clinical conditions. Also, the clinical procedures were rarely described in details.

On the other hand, in a prospective study design, many of the variables possibly involved are already controlled at the stage of case selection, and experimental groups can be made homogeneous in all but the variable under study. Such a study model, by limiting the effect of confounding factors, delivers more truthful, reliable, and therefore valuable information. For instance, in the present research, the experimental groups were controlled for type of tooth since only premolars were included, for single operator, and for type of endodontic treatment, which was performed following the same technique in all the teeth on trial. Thus, the variables under study, i.e. the different materials used for the restoration, became the factors most crucially responsible for the variability in the clinical performance of the teeth over time.

In any case, the results of the present clinical trial were in line with the findings of the previous retrospective studies (Dallari and Rovatti, 1996) (Ferrari *et al*, 2001) (Ellner *et al*, 2003), which also reported a survival rate of the tested posts comparable to

the 96% value recorded in this investigation. Essentially, all the clinical investigations on fiber posts have provided the same indications regarding fiber posts:

1. The failure of fiber posts is never due to a root fracture (Dallari and Rovatti, 1996) (Ferrari *et al*, 2000), as opposed to cast posts (Ferrari *et al*, 2000a) (Ellner *et al*, 2003), in which root fracture becomes a real risk. A possible reason for the quite common occurrence of root fracture with cast posts has been seen in the friction created along the walls, as well as in the rigidity of the metallic materials in comparison with dentin (Ferrari *et al*, 2002).

2. The most frequent cause of failure of luted fiber posts is debonding. This is usually the result of an adhesive failure at the interface between dentin and resin cement.

In the present study, no fracture of the root or abutment was seen, and no post dislodgement or crown debonding was recorded. Failures consisted only of post debonding, which were ascribed to a loss of integrity at the adhesive interface between dentin, adhesive, resin cement, and post.

All of the debondings occurred during the removal of provisional crowns, and in teeth with less than 2 mm of dentin structure left at the coronal level. This observation is in agreement with previous findings (Ferrari *et al*, 2002) that in the amount of residual coronal tooth structure is the most influential factor in predicting fracture resistance.

All of the debonding failures could be solved. In four of the cases the post had to be replaced, otherwise the same post was simply rebonded. Almost half of the failures were due to the recurrence of periapical lesions that required endodontic retreatment.

This study evaluated three types of translucent fiber posts, used with different combinations of adhesive and restorative materials under similar clinical conditions. No significant difference emerged in the performance of the three classes of posts. Frequency and patterns of failure were similar to those already reported in some previous studies (Ferrari *et al*, 2000a).

In two groups, the combination of light-curing adhesive and dual-curing cement was tested, whereas in the third group, self-curing materials were used. As no statistically significant differences were found among the three groups, it follows that the selection of the adhesive-cement combination basically becomes a matter of personal preference of the clinician, based on his experience and habits. The study also demonstrated that using materials from the same manufacturer for luting fiber posts and

building up composite cores does not significantly add to the clinical performance of the restoration.

This study evaluated two flowable resin composites used for core build-ups. Given the relatively low stiffness of flowable composites, the question of whether their mechanical properties make them reliable as core materials can reasonably be raised. However, in the present trial, flowable materials proved able to provide a valid support to porcelain crowns for at least two years of clinical service. This satisfactory performance encourages the daily use of flowable composites for building up abutments around fiber posts (Ferrari *et al*, 2002) (Bayne *et al*, 1998).

The null hypothesis tested in this study were not confirmed: there was no significant difference in the clinical service yielded by the three types of posts, and the use of materials from the same manufacturer when preparing fiber post-composite core units does not significantly add to the clinical performance of the restoration.

Longer follow-up data on translucent fiber posts and different composite resins used for core build-ups are expected from a prospective multicenter study currently in course.

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Chapter 5

5.1. Exploring the application of superficial treatments to improve bond strength to fiber posts.

Retention of post and core restorations depends on the quality of bond established at different interfaces. Several studies have already been conducted to evaluate the interface with dentin, both at the coronal and radicular level (Ferrari *et al*, 2001) (Ngoh *et al*, 2001) (Ari *et al*, 2003).

Since the introduction of fiber posts, a continuous effort has been made to improve bonding inside the root canal: despite the introduction of novel adhesive systems, radicular dentin still offers less favourable conditions for bonding than coronal dentin (Ferrari *et al*, 2002) (O'Keefe and Powers, 2001).

Chersoni and colleagues (2005) recently reported that the bonding efficacy of simplified hydrophilic adhesives to auto-cured composites/cements is hampered by the intrinsic permeability of these adhesives to water still present in pulpless teeth.

As matter of fact, the most frequent cause of adhesive failure is debonding of post restoration at the resin cement/ dentin interface (Ferrari *et al*, 2000a) (Ferrari *et al*, 2000b) (Malferrari *et al*, 2003).

Although the adhesion into the root canal represents the weakest point of the restoration, also the post/composite adhesion has to be considered. Immediately after fiber post cementation and core build-up, the restoration has to resist to the stresses transmitted during core trimming to adapt the provisional crown (Goracci *et al*, 2005). At the coronal level, the amount of residual tooth structure still offer more favourable conditions for ensuring strong adhesion and retention (Castellucci and Becciani, 2004) (Schwartz and Robbins, 2004).

At the post-core interfacial level, only the chemical interaction between the fiber post surface and the resin composite may ensure the bond of the core material around the post. Treating the post surface with a silane coupling agent is advisable for enhancing adhesion (Aksornmuang *et al*, 2004) (Goracci *et al*, 2005).

Silanes are able to improve surface wettability and chemically bridge organic and inorganic substrates (Plueddemann, 1991). Although the clear benefit of silane coating in enhancing post-core bond strength, it still remains a weak bond.

In an attempt to overcome this problem, an insight in fiber reinforcement technology may be of help. To achieve desirable properties in composite components, adhesion between fibers and composite is usually optimized through a preliminary superficial preparation to provide a chemical and micromechanical interaction (Iglèsias *et al*, 2002).

It was of interest to verify whether and to what extent the adhesive potential of the fiber post could be improved through these treatments. A study on the chemicals and the laboratory and industrial techniques is reported in this chapter in the attempt of finding a possible application in dentistry. Different *in vitro* studies, involving both bond strength tests and microscopic analysis evaluated different aspects of the post-core interface with the purpose of enhancing adhesion.

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5.2. A simple etching technique for improving the retention of fiber posts to resin composites

Francesca Monticelli, Manuel Toledano, Franklin R. Tay, Fernanda T. Sadek, Cecilia Goracci, Marco Ferrari. *Journal of Endodontics* 2005, in press.

Introduction

Etching procedures for natural and artificial substrates have been developed to enhance adhesion (Buonocore, 1955) (Nakabayashi, 1982) (Thompson, 1984) (Nakabayashi *et al*, 1991). They include acid treatments of enamel (Buonocore, 1955), dentin (Nakabayashi, 1982) (Nakabayashi *et al*, 1991) or non-noble alloys (Thompson, 1984) that partially dissolve the substrates and generate micro-porosities where resin can penetrate creating micromechanical interlocking (Nakabayashi, 1982).

Fiber posts are extensively used in clinical practice to restore endodontically-treated teeth (Duret *et al*, 1990) (Mannocci *et al*, 1999) (Ferrari *et al*, 2000) (Dammaschke *et al*, 2003) (Gesi *et al*, 2003) (Schwartz and Robbins, 2004) (Ettinger and Quian, 2004) (Grandini *et al*, 2005). The clinical longevity of endodontically-treated teeth restored with fiber posts and resin composite cores was recently evaluated (Ferrari *et al*, 2000) (Monticelli *et al*, 2003). Satisfactory adaptation at the post/core interface could be achieved using flowable composites as core materials (Monticelli *et al*, 2003) (Monticelli *et al*, 2005).

The effect of post silanization on the interfacial strength between fiber posts and core build-up composites was recently evaluated with the microtensile test (Goracci *et al*, 2005). Silane coupling agents can achieve chemical bonds with OH-covered inorganic substrates such as glass. A chemical bond may be achieved between the core resin matrix and the exposed glass fibers of the post at the interface level (Goracci *et al*, 2005) (Aksornmuang *et al*, 2004). However, the interfacial strength is still relatively low when compared to the values normally achieved with coronal dentin or enamel (Cardoso *et al*, 2002) (Sahafi *et al*, 2003), due to the absence of chemical union between the methacrylate-based resin composites and the epoxy resin matrix of fiber posts.

Hydrogen peroxide (H₂O₂) is commonly employed in immunological electron microscopy to partially dissolve the resin surface of epoxy resin-embedded tissue sections, and expose tissue epitopes for immunolabeling enhancement. The etching

effect of H₂O₂ depends on its capacity to partially dissolve the resin matrix, breaking epoxy resin bonds through a mechanism of substrate oxidation (Holm *et al*, 1989) (Erneström and Kniola, 1995) (Bronson *et al*, 2001) (Baskin *et al*, 1979). A similar H₂O₂ etching procedure may be employed to improve the micromechanical retention between the epoxy resin matrix of fiber posts and methacrylate-based resin composites.

The aims of this study were to evaluate the effect of H₂O₂ on the morphological aspects of the post surface, and the influence of different surface treatments on the interfacial strengths between fiber posts and composites. The null hypothesis tested was that different types of post surface treatment and the type of flowable core build-up composites do not affect the interfacial strength between fiber posts and resin composites.

Materials and methods

Experimental Design

Sixty DT Light-Posts (RTD, St. Egrève, France), each with a 2.1 mm maximum diameter were used in this study. These posts are made of quartz fibers (60% vol) previously processed to a unidirectional axial tensile stress and embedded in an epoxy resin matrix (40% vol). They were divided into five groups (n=12) according to the different surface pre-treatments performed.

In group 1 the posts were immersed in 24% H₂O₂ for 10 min at room temperature (RT). In group 2 they were immersed in 10% H₂O₂ for 20 min at RT. In Group 3, after immersion in 24% H₂O₂ for 10 min at RT, the posts were silanized for 60 sec. In Group 4, after immersion in 10% H₂O₂ for 20 min at RT, the posts were silanized for 60 sec. In Group 5 (control) the posts were silanized for 60 sec.

After the application of H₂O₂, the posts were rinsed with water and air-dried. A silane coupling agent (Monobond-S, Ivoclar-Vivadent, Schaan, Liechtenstein) was applied in a single layer on the post surfaces of groups 3-5 and gently air-dried after 60 sec, according to manufacturer's instructions. Monobond-S is a single-component silanizing agent that contains 1 wt% of 3-methacryl-oxypropyltrimethoxysilane in an ethanol/water-based solvent.

Scanning Electron Microscopy (SEM)

After H₂O₂ treatment, two posts from groups 1 and 2 were examined with an SEM to study their surface conditions. One post from each group was observed longitudinally, while the other was cross-sectioned using a water-cooled diamond blade (Isomet, Buehler, Lake Bluff, IL). The prepared specimens were ultrasonicated for 5 min in deionized water, immersed in 96% ethanol and gently air-dried. Each post was sputter-coated with gold-palladium alloy (Polaron Range SC7620, Quorum Technology, Newhaven, England) and examined with an SEM (JSM 6060 LV, JEOL, Tokyo, Japan).

Core Build-up and Microtensile Testing

Core build-up was performed for the remaining ten posts in each group using either one of the two flowable composites *ÆliteFlo* (A) (Bisco Inc., Schaumburg, IL) and *UniFil Flow* (B) (GC Corporation, Tokyo, Japan) using an *in vitro* technique that was described in detail by Goracci *et al.* (2005). Each post was positioned perpendicularly on a glass slab and secured with a needle holder at the apical end. A plastic matrix of 10 mm in diameter was placed around the cylindrical portion of the post (i.e., the part with uniform diameter) and adjusted so that the post would be exactly in the middle. An incremental technique was used for the core build-up with each composite increment cured for 20 sec using a halogen curing light with an output of 600 mW/cm² (VIP, Bisco Inc.). An additional 20 sec polymerization was subsequently performed from the bottom side of the cylinder previously in contact with the glass slab.

Two longitudinal cuts were made on the two opposite sides of the post/composite assembly with the Isomet saw under water cooling exposing the post surface throughout its length. A slab of uniform thickness, with the post in the centre and the core build-up on each side was created. Beams of 1-mm in thickness were serially sectioned from each slab. Every slab yielded 4-5 beams for microtensile testing that was performed with a universal testing machine at a cross-head speed of 0.5 mm/min until failure. Interfacial strength was calculated using the mathematical formula previously described by Bouillaguet *et al.* (2003).

The normally distributed data were analyzed with a two-way ANOVA design, to assess the effect of surface treatment and core build-up material on the interfacial strengths of the fiber posts. Post-hoc comparisons were performed using the Tukey test.

Statistical significance was set at $\alpha=0.05$. The sample size was checked to ensure a minimum power of 0.8 in the statistical analysis.

Results

SEM Evaluation

Pre-treatment with 24% H₂O₂ -10 min or 10% H₂O₂ -20 min were comparable in their efficacy in modifying the fiber post surfaces. Both groups created rough surfaces along the entire post lengths (Fig.1A). The exposed quartz fibers were not damaged or fractured by the oxidative action of H₂O₂ (Fig.1B). Cross-sectional views of both groups revealed surface dissolution of the epoxy resin matrix to a depth of 50 μ m (Fig.2A), exposing approximately five layers of quartz fibers (*ca.* 10 μ m in diameter) for micromechanical retention (Fig.2B). The underlying epoxy resin remained intact and exhibited no signs of cracking or damage.

Interfacial Strength

The results of microtensile bond testing are shown in the Table. The type of surface pre-treatment had a significant influence on interfacial strength ($p<0.0001$). The results achieved with 24% H₂O₂-10 min and 10% H₂O₂ -20 min were comparable and significantly better than no treatment. Silane application in combination with H₂O₂ pre-treatment produced the best overall results ($p<0.0001$). The type of core material also had a significant influence ($p=0.014$), as did the interaction of this factor with the type of pre-treatment ($p<0.01$). Post treatment with either 24% or 10% H₂O₂ enhanced the bond strength, particularly of UnifilFlow.

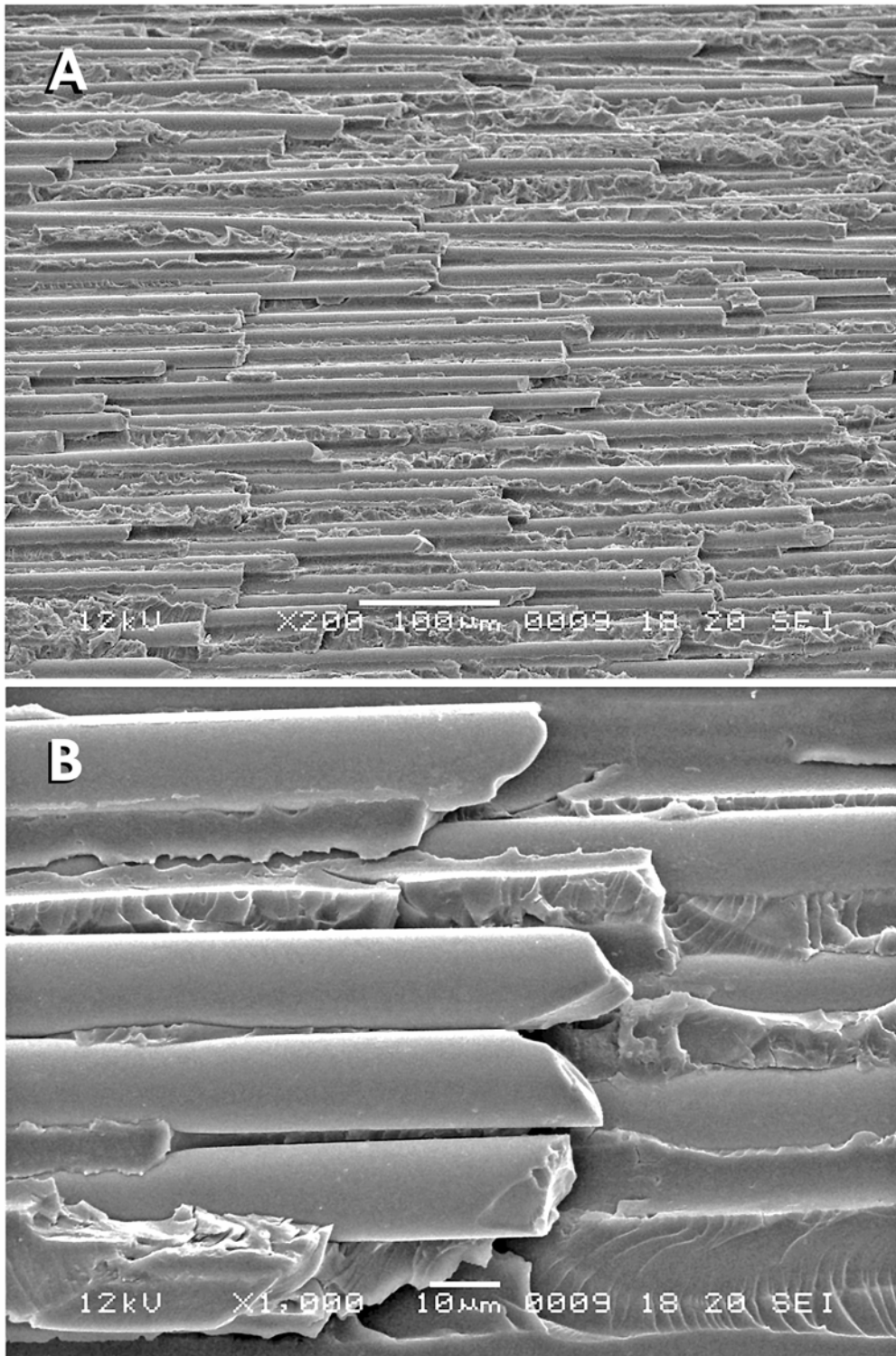


Fig. 1. Representative SEM micrographs of a post-surface after etching with 10% hydrogen peroxide for 20 min. **A.** At a low magnification, exposure of the quartz fibers could be seen, as a result of the dissolution of the surface epoxy resin. **B.** A high magnification view of the exposed quartz fibers, showing that they were not damaged by the etching treatment. Similar results were achieved when etching was performed with 24% hydrogen peroxide for 10 min (not shown).

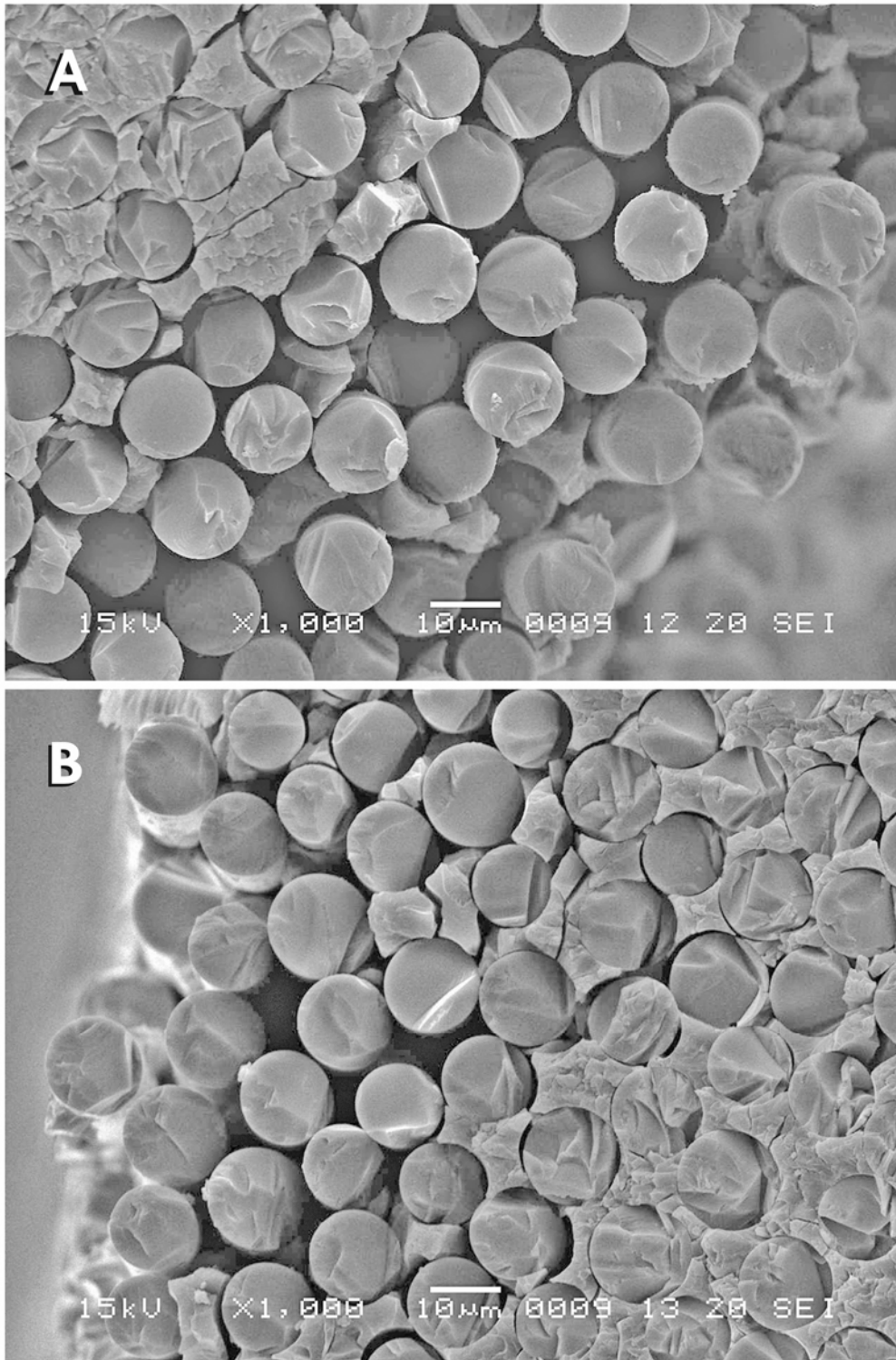


Fig. 2. SEM images of the cross sections of fiber posts after treatment with 24% Hydrogen Peroxide for 10 min (Fig 2a) and 10% Hydrogen Peroxide for 20 min (Fig 2b). Dissolution of the epoxy resin from the post-surfaces exposed the quartz fibers to a depth of 50 μm for silanization as well as created additional spaces for micromechanical retention of the flowable composites.

Table. Interfacial strengths of the experimental groups after different types of fiber post-surface treatment.

Core material	Type of treatment *			
		24% H ₂ O ₂ -10 min	10% H ₂ O ₂ -20 min	No treatment
Eliteflo (A)	No silane	Group 1A (n=24) 8.3 (4.2) ^{def}	Group 2A (n=24) 6.1 (4.8) ^{ef}	
	Silane	Group 3A (N=25) 11.7 (3.9) ^{bc}	Group 4A (n=26) 13.0 (4.6) ^{ab}	Group 5A(n=27) 6.7 (3.0) ^{def}
UniFil Flow (B)	No silane	Group 1B (n=26) 9.1(3.2) ^{cde}	Group 2B (n=25) 9.5 (4.0) ^{cd}	
	Silane	Group 5B (n=24) 15.3 (3.2) ^a	Group 4B (n=25) 13.8 (3.3) ^{ab}	Group 5B(n=24) 5.3 (1.7) ^f

* Values are mean (standard deviation) in MPa. Groups connected with the same letter superscripts are not statistically significant (p>0.05).

Discussion

The use of H₂O₂ pre-treatment and silane application significantly enhances interfacial strength between fiber posts and core materials. Interfacial strength was also influenced by the choice of flowable composites for core build-up. Thus, the null hypothesis tested in this study is rejected.

Unlike the use of more corrosive forms of industrial epoxy resin etching techniques such as sodium ethoxide or permanganate etching (Roizard *et al*, 2002) (Bronson, 2001), H₂O₂ etching provides an easy, effective and clinically feasible method for enhancement of interfacial strengths between fiber posts and resin composites, without the need of employing extremely corrosive liquids in a clinical setting. By removing a surface layer of epoxy resin, a larger surface area of exposed quartz fibers is available for silanization. The spaces between these fibers provide additional sites for micromechanical retention of the flowable resin composites. This retention concept is reminiscent of the creation of hybrid layers in dentin and cementum (Vichi *et al*, 2002), as the interface is contributed by both the quartz fibers from the fiber post and the methacrylate resin matrix.

A more aggressive etching procedure has recently been recommended that involves etching fiber posts with hydrofluoric acid (Dallari and Mason, 2004). However, this technique considerably affected the integrity of the fiber posts. Microscopic analysis revealed uneven removal of the epoxy resin matrix and extensive damage to the quartz fibers as they were simultaneously attacked by the hydrofluoric acid. On the contrary, H₂O₂ etching is a considerably milder technique with the exposed quartz fibers remaining smooth and leaving the underlying epoxy resin matrix intact after the etching procedures.

The use of silane coupling agents improves surface wettability and creates a chemical union between the glass fibers and resin-based materials (Park and Jin, 2001). In case of epoxy matrix glass-reinforced composites, amino-silanes are typically applied, since epoxy resin and glass can adhere through an amino-coupling reaction (Iglesias *et al*, 2002). The improved interfacial strength achieved following silanization may depend on the reaction between the silane molecules and the silica component in the quartz fibers (Cheng *et al*, 1993) (Debnath *et al*, 2003). In this study a prehydrolyzed silane coupling agent containing 3-methacryl-oxypropyltrimethoxysilane was used. The methacryloxy group in this silane coupling agent probably enables a

chemical union to be achieved between the silane-treated quartz fibers and the methacrylate-based resin composite matrix, thereby improving interfacial strength.

Although the mechanical properties of flowable composites are generally inferior to those of hybrid composites or core materials (Bayne *et al*, 1998)(Labella *et al*, 1999), they were selected for this study by virtue of their previously reported adaptation to post surfaces (Monticelli *et al*, 2003) (Monticelli *et al*, 2005) (Goracci *et al*, 2005). It could be seen from the interfacial strength results that the two flowable composites investigated did not differ significantly from one another in the absence of any surfaces pre-treatment. Conversely, significant differences were observed between the two materials when post-surface were pre-treated with H₂O₂ etching and silane. The statistically significant interaction with the two factors “pre-treatment” and “composite” suggests that these differences may depend on the variation in viscosity between the two flowable composites. It is possible that the lower viscosity of UniFil Flow enables a better penetration of this material within the 50 µm thick zone of denuded quartz fibers. Incomplete penetration of this zone may create a scenario that approximates the incompletely infiltrated hybrid layers that were observed when some dentin adhesives were applied to acid-etched dentin (Spencer *et al*, 2004). Unlike denuded collagen fibrils within incompletely infiltrated dentin hybrid layers, quartz fibers cannot degrade via hydrolysis by endogenous metalloproteinases (Pashley *et al*, 2004). Nevertheless, a weak zone may be created that could have accounted for the lower interfacial strength observed when the more viscous *Æ*liteFlo was applied to the surface-treated fiber posts. In the future, it would be necessary to examine whether the use of low viscosity hydrophobic resins such as those employed as pit-and-fissure sealants will further improve the novel concept of fiber post surface hybridization introduced in this study. The extent of resin impregnation within the zone of hybridized fiber post should also be examined with tracer penetration techniques. The use of hydrophobic resins in thin layers instead of dentin adhesives can prevent unnecessary water sorption and may alleviate the use of mechanically less favorable flowable composites as core build-up materials.

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5.3. Post surface conditioning improves interfacial adhesion in post/core restorations

Francesca Monticelli, Manuel Toledano, Franklin R. Tay, Alvaro H. Cury, Cecilia Goracci, Marco Ferrari. *Dental Materials* 2005, in press.

Introduction

The use of fiber posts in combination with composite core build-up materials has increased in popularity in the restoration of endodontically treated teeth. Many laboratory and clinical studies investigated their mechanical properties and clinical behaviour (Mannocci *et al*, 1999) (Ferrari *et al*, 2000) (Schwartz and Robbins, 2004) (Grandini *et al*, 2005). The clinical success of a post/core restoration depends on the selected composite, but also on the quality of the post/core interface, where materials of different compositions are in intimate contact (Aksornmuang *et al*, 2004).

Studies on fibre-reinforced composite materials have led to the modification and improvement of the properties of the interface between the resinous matrix and the fibres. In many cases, interfacial failure was attributed to chemical incompatibility or the plasticising phenomenon where impurities (commonly water) penetrate the interface (Cheng *et al*, 1993) (Park and Jin, 2001) (Iglesias *et al*, 2002). In dentistry, the durability of a composite core restoration depends on the formation of a strong bond between the resin composite and the residual dentine (O'Keefe and Powers, 2001), as well as between the composite and the fibre post (Park and Jin, 2001), enabling the interface to transfer stress under functional loading (Isidor *et al*, 1999).

Surface treatments are common methods to improve the general adhesion properties of a material, by facilitating chemical and micromechanical retention between different constituents. Advances in adhesive dentistry have resulted in the development of surface conditioning techniques for natural substrates (i.e. enamel, dentine) (Buonocore, 1955) (Nakabayashi, 1982) (Nakabayashi *et al*, 1991) and restorative materials. The latter includes the use of acids to condition the surfaces of non-noble alloys (Thompson, 1984) or ceramics (Horn, 1983), to partially dissolve the substrate and generate a rough surface to enhance adhesion (Özcan and Vallittu, 2004).

With respect to post/core restorations, most studies were designed to improving the performances of these restorations via enhancing the mechanical properties of the

composite core build-up materials (Combe *et al*, 1999) (Chutian *et al*, 2004). However, from acid-etching and silanization of the glass fibre phase of these posts (Özcan and Vallittu, 2004) (Sphor *et al*, 2003), little is known on how the resin phase of these fibre posts may be improved to interfacial adhesion. The application of a silane coupling agent as adhesion promoter in fibre post/core units was recently investigated (Aksornmuang *et al*, 2004) (Goracci *et al*, 2005). Nevertheless, adhesion of composites to fibre posts was still inferior when compared with the results achieved on dental substrates (Sahafi *et al*, 2003) (Cardoso *et al*, 2002). This is probably due to the absence of chemical interaction between methacrylate-based resin composite and the epoxy resin matrix of fiber posts (Özcan and Vallittu, 2004). We hypothesize that surface pre-treatment of the resin phase of fiber posts may improve their adhesion to methacrylate-based resin composites. For industrial applications such as epoxy resin-based circuit boards, many chemical treatment techniques have been introduced to improve the adhesion between the components of fibre-reinforced resin composites (Cheng *et al*, 1993) (Crasa *et al*, 1999) (Roizard *et al*, 2002). Different solutions and solvents are known to be effective on epoxy resin (Bronson *et al*, 2001) (Bronson, 2001). It is speculated that a similar approach may be applied in dentistry for surface pre-treatment of fibre posts to increase their responsiveness to silanization.

Thus, the aims of this study were: 1) to evaluate the effect of different chemical etching procedures on the morphological aspects of the fibre post surface, and 2) to examine the influence of different post surface treatments on the interfacial strength between epoxy resin-based fibre posts and methacrylate-based resin composites that are employed as core build-up materials. The null hypothesis tested was that different types of post surface treatment do not affect the interfacial strength between fibre posts and composite core build-up materials.

Materials and methods

Sixty DT Light Posts, #3 with a maximum diameter of 2.14 mm (batch no. 120US0401A, RTD, St. Egrève, France) were used for this study. The DT Light Posts are made of unidirectional pre-tensed quartz fibres (60 vol%) bound in an epoxy resin matrix (40 vol%). Five different chemical treatments were tested for their efficacy on etching the resin phase of the fibre post surface (n= 12 in each group):

- Group 1: etching with potassium permanganate;
- Group 2: etching with 10% hydrogen peroxide for 20 min;
- Group 3: etching with 21% sodium ethoxide for 20 min;
- Group 4: etching with potassium permanganate and 10 vol% HCl for 1 h;
- Group 5: silanization of the post surface for 60 s without chemical treatment of the resin phase (control group).

The etching procedure of group 1 consists of three successive steps: 1) immersion of the posts in a conditioning solution (60 vol% of methyl-pyrrolidone in deionised water) for 3 min at 50-60°C (E-K Hole Cleaner, Elkem, Torino, Italy). This initial step enhances the removal ability of permanganate with the epoxy resin being swollen and its surface chemical structure altered. Extensive rinsing with tap water was performed for 3 min; 2. etching in an alkaline potassium permanganate solution (20 vol% in deionised water; pH 12-13) (E-K Hole Oxidizer, Elkem) for 10 min at 70-80°C to oxidise and remove the epoxy resin matrix previously degraded by the solvent; 3. immersion in a sulphate neutraliser solution (10 vol% in deionised water) (E-K Hole Reducer, Elkem) for 5 min at 40-50° C to reduce and neutralise the excess permanganate and clean the surface of the post. Each surface-treated post was rinsed with deionised water for 3 min, followed by air-drying.

The fibre posts in group 2 were immersed in 10% hydrogen peroxide (Sigma, Aldrich Chemic, GmbH, Steinheim, Germany) for 20 min at room temperature and then rinsed with deionised water. The posts in group 3 were immersed in a 21 wt% sodium ethoxide solution (i.e. sodium hydroxide in ethanol; Sigma, Aldrich Chemic, GmbH, Steinheim, Germany) for 20 min at room temperature, then rinsed with pure ethanol, 50% ethanol solution in deionised water and finally deionised water (5 min for each cleaning bath) to reach a stable pH of 7.

For the posts in group 4, the same potassium permanganate treatment employed in group 1 was initially performed. This was followed by immersing the permanganate-treated post in a 10 vol% solution of hydrochloric acid (HCl; Panarec Quimica SA, Barcelona, Spain) in deionised water for 1 h.

Before performing the composite core build-ups, an additional exhaustive rinsing procedure was performed. All the posts were ultrasonically cleaned for 10 min in deionised water (P Selecta S.A. Abrera, Spain), immersed in 95% ethanol and dried with an air stream.

A single layer of silane coupling agent (Monobond-S, batch no. E53184 Ivoclar-Vivadent, Schaan, Liechtenstein) was then applied with a brush to the post surface of each of the five experimental and control groups, and gently air-dried after 60 s, according to manufacturer's recommendations. Monobond-S is a pre-hydrolyzed single component silanizing agent and contains 1 wt% of 3-methacryloxypropyl trimethoxysilane (3-MPS) in an ethanol/water-based solvent.

Core Build-up Procedure and Microtensile Test

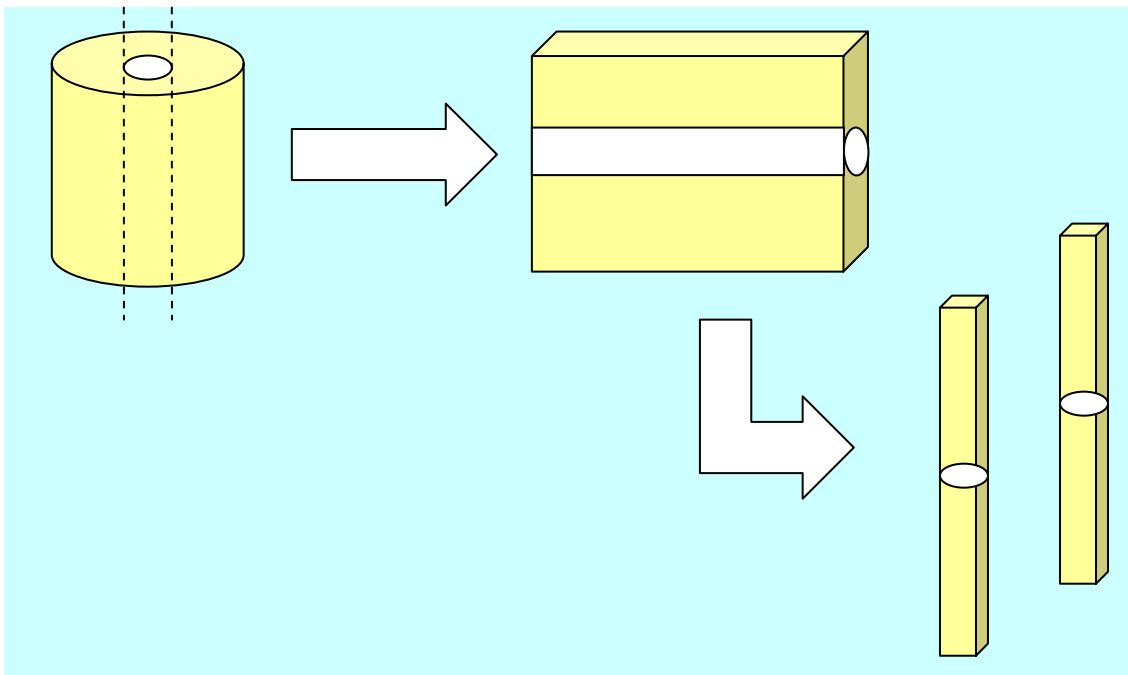
Core build-up was performed using (A) a dual-cured composite core material (Core Paste XP batch no. 030653101, Den-Mat, Santa Maria, CA, USA), and (B) a flowable resin composite material (UniFil Flow, batch no. 04001061, GC Corp., Tokyo, Japan), using an *in vitro* technique previously reported by Goracci *et al* (2005) for similar purposes. Each post was positioned perpendicularly on a glass slab and maintained with a needle holder at the apical end. A cylindrical plastic matrix was placed around the post and an incremental technique was followed to build up the core. Each 2-mm increment of the core composites was cured for 40 s with a halogen light-curing unit (Optilux, Demetron Res. Corp, Danbury CT, USA) with an output of 600mW/cm². The material was polymerised directly from the upper side of the matrix. The matrix was subsequently removed after being filled completely with polymerised composite. This resulted in a cylinder of resin composite that was built up around the fiber post. The bottom side of the cylinder that previously in contact with the glass slab was light-cured for an additional 20 s to ensure complete polymerisation of the composite material.

After storing in distilled water for 24 h, each bonded specimen was mounted on the holding device of a slow-speed diamond saw (Isomet 4000, Buehler, Lake Bluff, USA). Two longitudinal cuts were initially made on the two opposite sides of the post/composite assembly with the diamond saw under water cooling, to expose the post surface throughout its length. A slab of uniform thickness, with the post in the centre and the core build-up composite on either side was created. Each slab was then serially sectioned to obtain 4-5 beams of 1-mm in thickness (Fig. 1). Each beam was secured to the flat grips of a Bencor Multi-T device (Danville Engineering, San Ramon, CA) with cyanoacrylate adhesive, and subjected to a tensile load at a cross-head speed of 1 mm/min until failure, using a universal testing machine (Model 4411 Instron Corp.,

Canton MA). Interfacial strength was calculated using the mathematical formula previously described by Bouillaguet *et al* (2003).

The normally distributed data (Kolmogorov-Smirnof test) were analysed with a two-way ANOVA to examine the effect of surface treatment and choice of core material on the interfacial strength to fiber posts. Tukey test was performed for post-hoc comparisons. Statistical significance was set at $\alpha= 0.05$.

Fig. 1. Schematic illustration of the specimen preparation procedure for Microtensile bond strength test.



Scanning Electron Microscopy (SEM)

Two specimens from each group were examined with an SEM (JSM 6060 LV, JEOL, Tokyo, Japan) to study the characteristics of the post/core interface after different surface chemical treatments. The specimens were cross-sectioned by means of the water-cooled diamond saw to produce 2-mm thick slabs. The prepared specimens were mechanically polished with wet, 600-, 1200- and 4000-grit silicon carbide papers. Final polishing was achieved using a diamond polishing-paper (Lab-pol 8-12, Bantec Leicester, UK) for 10 s. The polished specimens were ultrasonicated for 5 min in deionised water (CP-104 EIA Intern. Paris, France), immersed in 95% ethanol, and gently air-dried. Each specimen was sputter-coated with gold-palladium (Polaron Range

SC7620, Quorum Technology, Newhaven, England) and examined with an SEM at different magnifications.

Results

SEM Analysis

SEM evaluation revealed surface modification of the epoxy resin matrix of the fibre posts in groups 1-4 that were comparable in their efficacies. Dissolution of the epoxy resin matrix resulted in the exposed quartz fibre component of these posts being surrounded by the core build-up resin composite. Partial removal of the epoxy resin matrix of the fibre posts created “retention spaces” among the fibres that appeared to be completely infiltrated by the core materials (Figs. 2A and 2B). No cracking was evident from the underlining untreated epoxy resin of these four groups of fiber posts, and the exposed quartz fibres were not damaged by the chemical treatments.

Low magnification micrographs revealed the absence of any defect and/or discontinuities along the interface between the fibre post and the composite (Fig. 3).

Interfacial Bond Strength

The results of the microtensile bond strength test are shown in Table 1. Surface chemical pre-treatment had a significant influence on the interfacial bond strengths of these fibre posts ($p < 0.001$). The results achieved with potassium permanganate were significantly better than those obtained with other surface chemical treatment techniques for both of the composite materials tested. The type of core material did not have a significant influence on the results. However, post surface treatments enhanced the bond strengths of the core build-up materials, especially for Core Paste XP ($P < 0.05$).

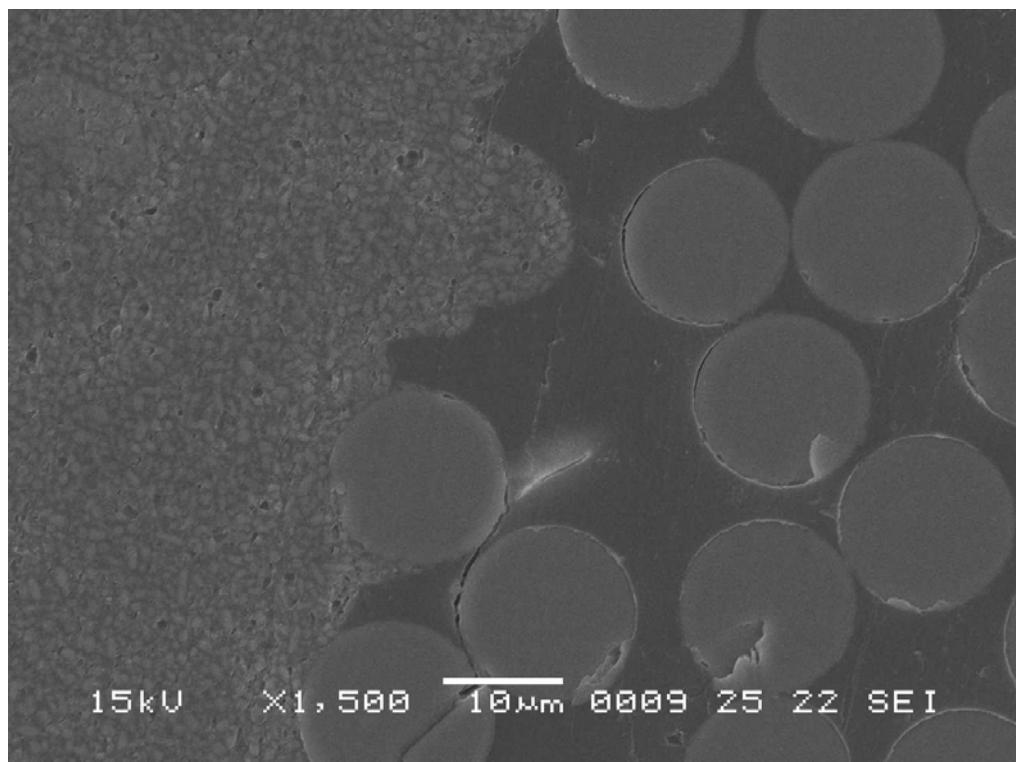
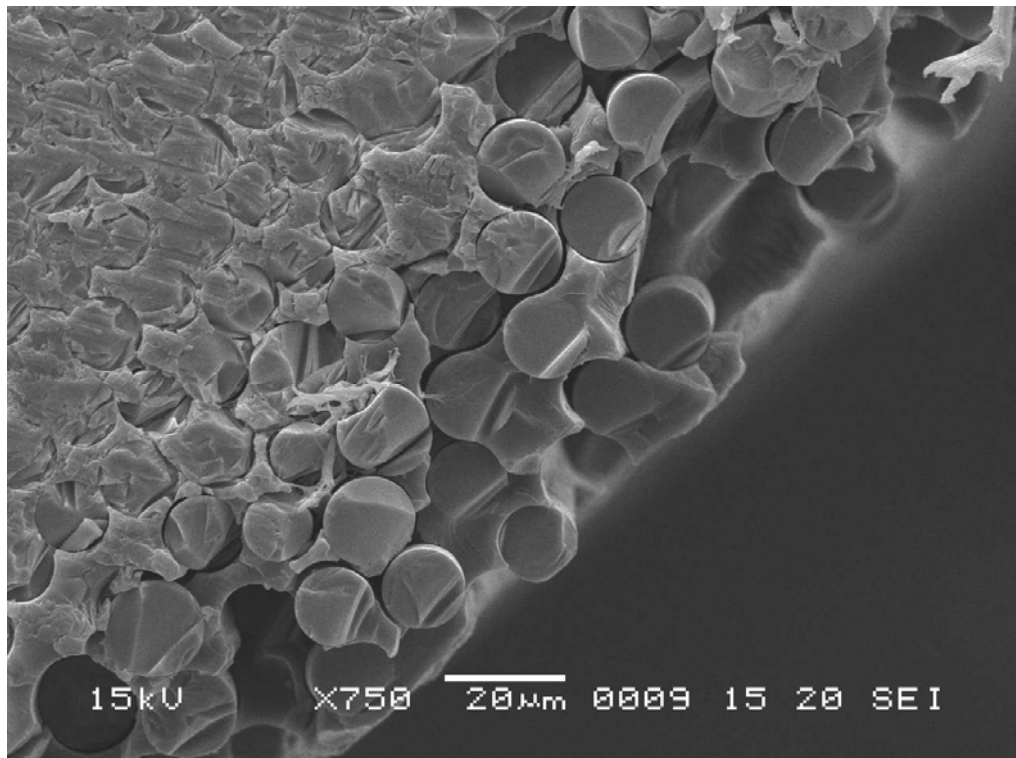


Fig 2. Representative SEM micrographs of the cross sections of fiber posts after etching with potassium permanganate. **A.** At a low magnification, exposure of the quartz fibres could be seen, as a result of the dissolution of the superficial epoxy resin (750X; Bar = 20 μm). **B.** A high magnification of the “free spaces” between the fibres that appeared to be completely infiltrated by the core material (1500X; Bar = 10 μm).

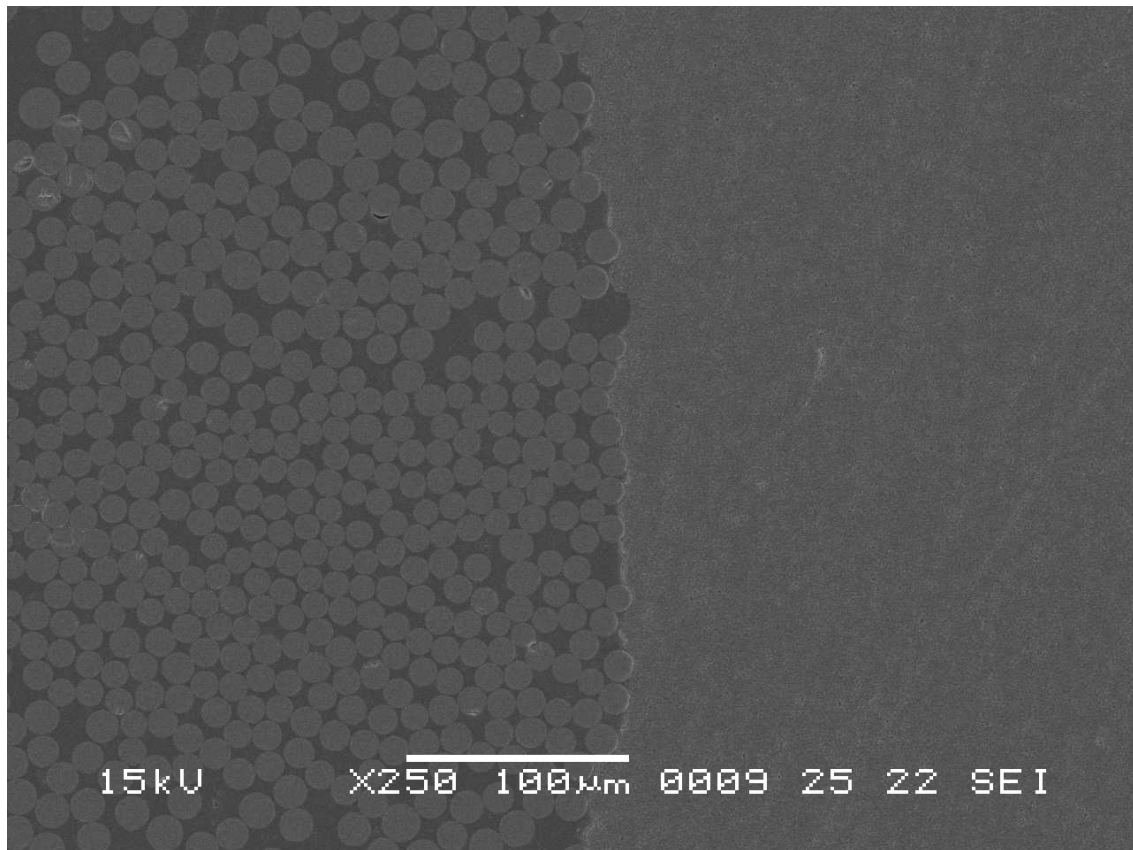


Fig 3. Low magnification micrographs revealed the absence of any defects and /or discontinuities at the interface between the fibre post and the resin composite (250X; Bar = 100 µm).

Table 1. Interfacial strength of the experimental and control groups after different post surface treatments.

Group designation	Surface chemical treatment	Core material	N*	Mean	SD	Statistical significance ¶
1	potassium permanganate + silane	Core Paste XP	25	14.50	4.07	a
		UniFil Flow	26	13.02	2.49	ab
2	sodium ethoxide + silane	Core Paste XP	27	11.04	2.98	bc
		UniFil Flow	26	8.48	2.42	cd
3	10% H ₂ O ₂ + silane	Core Paste XP	27	10.70	2.74	bcd
		UniFil Flow	24	8.18	3.20	d
4	potassium permanganate + HCl + silane	Core Paste XP	25	10.11	3.60	cd
		UniFil Flow	25	10.20	2.81	cd
5 (control)	silane only	Core Paste XP	26	8.49	2.91	cd
		UniFil Flow	25	10.66	2.22	bcd

N: number of microtensile sticks prepared from the bonded fiber post-composite assemblies.

¶ Groups identified with the different letters are statistically different (P<0.05).

Discussion

The use of chemical surface treatments influenced the interfacial bond strength between fibre posts and core build-up materials. Potassium permanganate significantly enhanced the interfacial bond strength between fibre posts and both the tested composite materials. Thus, the null hypothesis is rejected.

Epoxy resin etching techniques are commonly employed in industrial and laboratory fields. Sodium ethoxide and hydrogen peroxide are frequently used in transmission electron microscopy for immunolabelling enhancement, by partially removing the resinous superficial layer of resin-embedded tissue sections (Bronson *et al*, 2001) (Bronson, 2001) (Baskin *et al*, 1979). Potassium permanganate is usually applied in an industrial process designed for conditioning epoxy resin surfaces for metal plating of printed circuits boards (Mann *et al*, 1991) (Kirmann *et al*, 1998) (Thorn and Walsh, 1991). Epoxy polymers exhibit a high degree of conversion and highly cross-linked structures (Lassilla *et al*, 2004). These agents exert their etching activity by oxidising the substrates and breaking the epoxide bonds (Bronson *et al*, 2001) (Baskin *et al*, 1979).

In terms of application time of potassium permanganate, a mild treatment protocol was chosen in this study, as we initially speculated that the superficial etching developed during this conditioning process would render it suitable for the adaptation of resin composite to enhance the adhesion of fibre posts.

The concept of conditioning artificial substrates to improve bond strength has antecedents in dentistry that are exemplified by the etching of Maryland bridge (Thompson, 1984) (Thompson *et al*, 1983) and felspathic porcelain restorations (Horn, 1983) (Özcan and Vallittu, 2004) (Borges *et al*, 2003). Based on this principle, different conditioning procedures initially proposed for ceramics have also been tested on fibre posts. Hydrofluoric acid has recently been proposed for etching glass fibre posts (Dallari and Mason, 2004). However, this technique produced substantial damages to the glass fibres that affected the integrity of the post. This is due to the extremely corrosive effect of hydrofluoric acid on the glass phase of a ceramic matrix (Özcan and Vallittu, 2004) (Addison and Fleming, 2004).

Sahafi *et al*. recently tested the efficacy of blasting the surface of zirconia and fibre posts with silica oxide (Co-Jet System) (Sahafi *et al*, 2003) (Sahafi *et al*, 2004). Despite the satisfactory bond strengths achieved, the treatment was considered too

aggressive for fiber posts with the risk of significantly modifying their shape and consequently, their fit within the root canals (Sahafi *et al*, 2004a).

On the contrary, the etching procedures proposed in this investigation only affected the superficial part of the epoxy resin matrix of the fibre posts, leaving exposed smooth quartz fibres intact in all the tested groups. This differential etching effect reflects the difference in reactivity between the epoxy resin phase and the quartz fibre phase of the fibre posts (Fig. 2A) (Roizard *et al*, 2002). It is further speculated that potassium permanganate, besides exposing the quartz fibres, may also activate the latter by improving their hydrophilicity. Such a condition may be due to the use of a specific neutralising solution after conditioning in order to “clean” the fibres from residual MnO_2^- ions (Thorm and Walsh, 1991). An increased deposition of silane may take place in the presence of a surface with more hydroxyl groups (Crasa *et al*, 1999).

Amino-silane coupling agents are generally used as adhesion promoters in the presence of epoxy resin polymers, as they provide both a chemical bond between inorganic substrates and the polymer, as well as increase surface wettability (Park and Jin, 2001). MPS silanes are commonly applied in dentistry (Özcan and Vallittu, 2004) (Matinlinna *et al*, 2004). Since MPS silane does not bond well with the epoxy matrix, the bond strength between the epoxy resin phase of the fibre post and the methacrylate-based resin composite should not be enhanced. With the removal of the superficial layer of epoxy resin via the chemical treatments, more exposed quartz fibres in terms of surface area are available for reacting with the silane molecules. The increased chemical union between the silanized quartz fibres and the methacrylate-based core material would significantly improve the interfacial bond strength (Matinlinna *et al*, 2004). When the surface of the post is etched and rinsed, a more reactive surface is thus generated for both chemical and micromechanical retention. As quartz fibres are comparable in chemical structure with ceramic materials, it is reasonable to speculate that siloxane bonds would be achieved, as they represent the binding sites for the coupling agents to the post surface (Özcan and Vallittu, 2004).

Two different resinous core materials were evaluated in this study: a flowable composite and a low-viscosity core material that are commonly employed for this purpose (O’Keefe and Powers, 2001) (Chutian *et al*, 2004) (Glazer, 2000). Although flowable resin composites exhibit lower mechanical properties (Labella *et al*, 1999) (Bayne *et al*, 1998), good adaptation to the post surface was recently reported (Monticelli *et al*, 2003) (Goracci *et al*, 2005) (Monticelli *et al*, 2005). These previous

results were also confirmed in this investigation, showing that the flowable composite was able to achieve a satisfactory bond with the fibre posts in the absence of any surface chemical treatment (Table 1).

Moreover, the two core build-up materials may be considered as comparable by the virtue of the similar interfacial morphology achieved; their low viscosity enabled a satisfactory penetration into the porosities created between the exposed quartz fibres. This micromechanical interlocking between the infiltrating composite and the exposed glass fibres created a structure that is reminiscent of dentine hybridisation, in which adhesive resin monomers infiltrate a partially demineralised collagen matrix and polymerise in situ to create a mechanism for dentine adhesion (Nakabayashi, 1982) (Nakabayashi *et al*, 1991). Likewise, the simultaneous increase in the surface roughness of the post and the surface area of the quartz fibres also provide additional frictional resistance and sites for silanization, further enhancing the bonding of the fibre posts to the methacrylate-based composites (Roizard *et al*, 2002).

The main goal of fibre-reinforced composite material science is to obtain a strong bond between the different components, so as to obtain a unique material with improved performances (Gonzàles-Benito, 2003). A similar concept may be applied in dentistry to improve the quality of the post/core unit, with the objective in creating a “monoblock” between the tooth and the restorative material.

Conclusions

Surface chemical treatments of the resin phase of fibre posts enhance the silanization efficiency of the quartz fibre phase, so that the adhesion in the post/core unit may be considered a net sum of chemical and micromechanical retention.

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5.4. Effect of temperature on the silane coupling agents when bonding core resin to quartz fibre posts.

Francesca Monticelli, Manuel Toledano, Raquel Osorio, Marco Ferrari. Dental Materials 2005, in press.

Introduction

Several studies suggested the use of silane coupling agents in coating applications to promote adhesion between inorganic surfaces and polymeric molecules (Mittal, 1992) (Daniels and Francis, 1998).

Organosilanes have the formula $R'-Si-(OR)_3$ with an organic functional group (R') and three alkoxy groups (R): the chemical reaction begins with the hydrolysis of the alkoxide groups (R) into silanols ($SiOH$) that may condense forming siloxane bonds (Daniels and Francis, 1998) (Matinlinna *et al*, 2004).

Many factors (pH, presence of solvents, molecule size etc.) may exert an influence in the way silane molecules can absorb, condense or interact with the substrate, influencing coupling effectiveness (Wu *et al*, 1997) (Liu *et al*, 2001).

To accelerate the mechanism of chemical interaction between the silane and the inorganic surface, the reaction may be catalyzed by acid treatment or heating (Plueddemann, 1991) (Shen *et al*, 2004). Heat treatment of silanated glass is routinely performed in the glass industry to maximize bond strength (Barghi *et al*, 2000). Silane has been proven to increase ceramic-composite bond strength during luting procedures or when repairing chipped ceramic restorations (Della Bona *et al*, 2003) (Barghi, 2000) (Özcan and Vallittu, 2004). Drying with hot air increases the effectiveness of some silane coupling agents when bonding ceramics to composite resins (Shen *et al*, 2004) (Nogami *et al*, 2004).

High temperature silane heat treatment (70-80°C) is not feasible for chair-side procedures, but a stream of warm air (38 °C) may be used for this purpose (Shen *et al*, 2004).

Fibre posts are extensively used in combination with composite resins to directly restore endodontically treated teeth (Monticelli *et al*, 2003) (Ferrari *et al*, 2000). The efficacy of silane coupling agents increasing bond strength between fibre post and composite core restorations have been recently reported (Goracci *et al*, 2005)

(Aksornmuang *et al*, 2004) (Sahafi *et al*, 2003) (Sahafi *et al*, 2004). However, no information is available concerning the possible influence of different silane coupling agent composition or silanizing modalities on post/composite bond strength. In particular, the possible influence of heating on the condensation reaction of silane molecules on the post surface is still unknown. The aim of the study were to determine the effect of warm air drying and different silane coupling agents on the achieved bond strength between fibre posts and resin composite.

The null hypothesis is that silane composition and air-drying temperature do not influence the microtensile bond strength between fibre posts and composite resin.

Materials and Methods

Thirty quartz fiber posts, with a maximum diameter of 1.80 mm in the cylindrical coronal portion and 1.0 mm at the radicular end (DT Light Post #2, batch no.100US0311A, RTD, St. Egrève, France) were used for this study. DT Light posts are made of unidirectional pre-tensed quartz fibres (60%) bound in an epoxy resin matrix (40%).

The posts were etched in 10 vol % hydrogen peroxide solution (Panreac Química, Barcelona, Spain) for 20 min at room temperature (Monticelli *et al*, 2005). They were rinsed with tap water and ultrasonically cleaned for 10 min in deionised water (P Selecta S.A. Abrera, Barcelona, Spain), subsequently immersed in 96% ethanol and dried with an air stream.

Six experimental groups (n=5) were formed and three different silane coupling agents were used: a pre-hydrolyzed silane coupling agent containing 3-methacryloxypropyltrimethoxysilane (3-MPS) (Monobond-S, batch no. E53184, Ivoclar-Vivadent, Schaan, Liechtenstein); a two-component silane coupling agent containing 4-methacryloxyethyl trimellitate anhydride (4-META) and trimethoxysilyl propyl methacrylate (γ -MPTS) (Porcelain Liner M, batch no.GF1, Sun Medical Co Ltd, Japan); a pre-hydrolyzed silane coupling agent containing glycid-oxi-propyl-trimethyl-oxi-silane (GPS) (Porcelain Silane, batch no. 4101PFS, BJM Lab, Or-Yenuda Israel) at two different air-drying temperatures (21° and 38° C). The tested materials were applied following manufacturer's recommendations. The composition and application mode of the tested materials are described in Table 1.

pH measurements were performed for all tested silane coupling agents with a digital pH-meter and a glass electrode calibrated with standard buffer solutions. (Micro pH 2000, Crison Instruments, Alella, Spain).

After etching and silanizing the post surface, the composite build-up was performed following a technique previously described by Goracci *et al.* (2005) and using a dual-curing resin composite (Core Paste XP, batch no.030653101, Dent Mat, Santa Maria, CA, USA). Core Paste XP is a low viscosity core material and contains glass fillers in a methacrylate matrix. Samples were stored 24h at room temperature before testing.

Microtensile test specimens were prepared by sectioning each sample with a diamond saw under water cooling (Isomet 4000, Buehler, Lake Bluff, IL). A medium of 29 beams of 1-mm in thickness were tested for each group. For the microtensile bond strength test, each beam was glued with cianoacrylate (Zapit, Dental Ventures of America, Corona USA) to the flat grip of a testing device (Bencor, Multi-T, Danville Engineering, San Ramon, CA) and loaded in tension at a cross-head speed of 1mm/min until failure (Instron Model 4411 Instron, Canton MA). The modes of failure were evaluated after testing under a stereomicroscope (Olympus SZ-CTV, Olympus, Tokyo, Japan) at 40x magnification. Failure modes were classified as adhesive (at the post/core interface), cohesive (within the resin composite) or mixed (combination of the two modes on the same surface).

Interfacial bond strength values were expressed in MPa using a mathematical formula previously described by Bouillaguet *et al.* (2003). Data were analyzed by two-way ANOVA to evaluate the effect of the factors (silane composition and air-drying temperature) on the dependent variable (microtensile bond strength). Interactions were included in the analysis. Multiple comparisons were performed with Student-Newman-Keuls test. Statistical significance was set at $\alpha = 0.05$. The sample size was calculated to ensure a power of 0.8 in the statistical analysis.

Table 1. Silane coupling agent compositions and procedures tested in the study.

Material	Composition	Application
Monobond-S (Ivoclar-Vivadent, Schaan, Liechtenstein) pH \approx 3.8	3-MPS (1%) Ethanol/water-based solvent, Acetic acid	Apply with a brush; Leave undisturbed for 60s; Gently air-dry.
Porcelain Liner M (Sun Medical Co Ltd, Japan) pH \approx 4.5	<u>Liquid A</u> MMA 4-META (10%) <u>Liquid B</u> MMA γ -MPTS (10%)	Apply with a brush; Gently air-dry.
Porcelain Silane (BJM Lab, Or-Yenuda Israel) pH \approx 1.8	GPS (3%) Ethanol-based solvent	Apply with a brush; Gently air-dry.

3-MPS: 3-methacryloxypropyltrimethoxysilane; MMA: metilmethacrylate;
4-META: 4-methacryloxyethyl trimellitate anhydride; γ -MPTS: trimethoxysilyl propyl
methacrylate; GPS: glycid-oxi-propyl-trimethyl-oxi-silane.

Results

Mean microtensile bond strength values are shown in Table 2. Microtensile bond strength was influenced by the air-drying temperature ($p < 0.001$), and by the application of different coupling agents ($p < 0.001$) on the bond strength. Interactions were also significant ($p < 0.001$). 3-MPS performed similarly regardless air-drying temperature. If dried at 21° C, GPS and 4-META/ γ -MPTS silanes exhibited lower bond strengths than 3-MPS. Similar results were attained for all coupling agents when dried at 38° C. GPS and 3-MPS had a similar acidic pH (4.5 and 3.8, respectively), while 4-META/ γ -MPTS silane recorded a lower value (1.8).

The distribution and percentages of failures are described in Table 2. A percentage of adhesive failure between 90 and 100% was recorded in all tested groups.

Table 2. Mean (standard deviation) of microtensile bond strength values (MPa) and percentage of failure mode obtained for each tested group.

	21° C			38° C		
	Mean(SD)	Adhesive	Cohesive	Mean(SD)	Adhesive	Cohesive
Porcelain Liner M	(n=30) 8.8 (3.1) ^{a1}	90%	10%	(n=27) 11.7(2.7) ^{c2}	97%	3%
Porcelain Silane	(n=30) 6.9(2.0) ^{b1}	90%	10%	(n=30) 11.6(2.8) ^{c2}	100%	-
Monobond -S	(n=28) 11.0(2.9) ^{c2}	100%	-	(n=30) 11.4(2.5) ^{c2}	97%	3%

Superscript letters show differences within the same column and numbers within the same row ($p < 0.05$).

Discussion

The use of different air-drying temperatures on the silanated posts and the different composition of the silane coupling agents affected the bond strength of composite to fibre posts. Thus, the null hypothesis is rejected.

Silane enhances post-resin bond strength by promoting the wetting of the etched post surface and facilitating the diffusion of the fluid composite resin into the retentive spaces among the exposed fibres (Monticelli *et al*, 2005). When silane is applied on the post surface and dried, two phases are created (Hossmann *et al*, 2002) (Ishida, 1985): an outermost physisorbed layer with few siloxane bonds and a hydrolytically stable chemisorbed layer on the post surface (Daniels and Francis, 1998) (Shen *et al*, 2004).

Further reactions between silane molecules and the organic surface (fibre posts) have been proven to occur enhancing condensation and providing a more tightly packed configuration of the coupler molecules on the post surface (Daniels and Francis, 1998) (Yang *et al*, 2003).

Two different silane agents were tested in this study: two single-phase pre-activated solutions based on different silane molecules (3-MPS and GPS, respectively) and a two-component system. In the latter hydrolysis occurs when mixing the silane coupler (γ -MPTS) with the acidic monomer (4-META) just before its application (Filho *et al*, 2004) (Kato *et al*, 2001). Pre-activated silane solutions are expected to exhibit a higher rate of hydrolysis compared to the two-component systems in which an

incomplete reaction may eventually take place if the solvent is not completely evaporated, affecting bond strength (Barghi *et al*, 2000) (Kato *et al*, 2001).

3-MPS and GPS are considered neutral silanes with a similar degree of coupling power: under mildly acidic concentration, they condense rapidly to reach an equilibrium composition in a short time (Shen *et al*, 2004).

GPS silane is appropriate for epoxy resin coupling: however, its application on the post surface has been proven to decrease bond strength at least if dried at 21° C (Debnath *et al*, 2003).

Pre-activated silane primers have a limited shelf-life due to the rapid formation of oligomers (Barghi *et al*, 2000) (Daniels and Francis, 1998). At high silane concentration (3% GPS in the tested silane) the potential of oligomer formation increases: moreover, the physisorbed silane tends to form a weak boundary layer onto the quartz fibres that may act deficiently in the post/composite interface, causing a lubrication effect (Daniels and Francis, 1998) (Liu *et al*, 2001) (Matsumura *et al*, 1997). It explains the lower μ TBS attained by the pre-hydrolyzed GPS-based silane when compared to 3-MPS silane when dried at 21° C. The results from this study indicated that by using a GPS coupling on the post surface, 38° C air-drying is advisable to facilitate solvent removal and to achieve chemical stability (Hossmann *et al*, 2004).

Silane coupling is considered a technique-sensitive step. Among factors affecting its effectiveness, solvent evaporation plays an important role: a small amount of solvent may be beneficial in promoting silane wetting, but an incomplete removal may compromise coupling (de la Fuente *et al*, 1999). Ethanol evaporates easily without affecting the reactivity of silane, especially in pre-hydrolyzed coupling agents (Shen *et al*, 2004) (Garcia *et al*, 2005), however, if evaporation is not completed at 21° C, warm air-drying may help ethanol and other by-products evaporation, facilitating the elimination of the detrimental outer physisorbed silane layer (Berg and Jones, 1998). The higher bond strength results achieved for GPS dried at 38° C may confirm this aspect. Ethanol/water-based silanes had a more stable behaviour, probably due to the mixing ratio of the two solvent components (Abate *et al*, 2000). At the two selected temperatures, no significant differences in the evaporation rate of water are expected to occur. Higher values may facilitate water removal and could increase bond strength.

The selection of a warm air stream rather than heat treatment, seems to be more effective on bonding to polymers and it may be considered a clinically-feasible chair-side procedure to overcome some of the problems related to silane composition and/or

application (Plueddemann, 1991) (Shen *et al*, 2004) (Hossmann *et al*, 2002). Some results recently achieved on ceramics seem to confirm this aspect, in particular for the two-component systems that have been proven to be more sensitive to heating (Hossmann *et al*, 2002) (Barghi *et al*, 2000).

Pre-hydrolysed MPS silanes yielded a more stable bond to the post surface as shown by the higher bond strength achieved regardless of the drying temperature (Chan and Brauer, 1982). The hydrolysis and condensation reactions as well as the thickness of the siloxane film are pH dependent (Daniels and Francis, 1998). Hydrolysis rate increases at higher pH. In this sense the durability overtime of the GPS silane bond (pH around 4.5) may be questionable (Olmos *et al*, 2003).

The presence of acetic acid in the 3-MPS tested silane solution may guarantee pH stability and effectiveness (Olmos *et al*, 2003).

Despite of a significant increase in bond strength after warm air-drying, the failure mode registered in the all tested groups was predominantly adhesive, revealing the relative weakness of the post/composite bond. Chemical adhesion between methacrylate-based resin composites and fibre posts cannot be completely obtained by silane (Monticelli *et al*, 2005). Further studies should be performed to evaluate the possibility of combining silane solutions and bonding agents to improve bond strength and stability of silane coupling during aging.

Conclusions

Air-drying at 38° C promotes the condensation process of the silane on the post surface and removes some of the loosely absorbed molecules probably by simple evaporation. Water-based silane coupling solutions showed to be less influenced by air-temperature during drying.

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5.5. Improving the quality of the quartz fiber post-core bond using sodium ethoxide etching and combined silane/adhesive coupling.

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Introduction

Fiber posts are commonly employed for restoring endodontically treated teeth (Heydecke *et al*, 2002) (Schwartz and Robbins, 2004). Although *in vivo* studies of post/core restorations demonstrated satisfactory clinical results (Monticelli *et al*, 2003), further improvement in the adhesion between fiber posts and composite resins is desirable to optimize the durability of these restorations (O'Keefe and Powers, 2001).

The use of silane coupling agents as adhesion promoters in fiber-reinforced materials is well established (Connel *et al*, 1998) (Plueddemann, 1991). Trialkoxy silanes have the general formula RYSiX_3 , where R is a non-hydrolyzable functional group, Y a linker and X the hydrolyzable group. The non-hydrolyzable group (e.g. methacrylate) can polymerize with resin composite monomers containing C=C double bonds. The alkoxy groups (X) can form chemical bonds with inorganic substrates with hydroxyl groups, such as the silica surface, forming chemical bonds (Park and Jin, 2001) (Matinlinna *et al*, 2004). Recent studies on the silanization of translucent fiber posts before core build-up have been promising (Goracci *et al*, 2005) (Aksornmuang *et al*, 2004).

Bonding to etchable ceramics is usually achieved by etching the ceramic surface to increase the surface area for micromechanical retention and silane coupling (Barghi *et al*, 2000). A similar approach may be used to render the surfaces of epoxy-resin based fiber posts more conducive to silanization. Etching solutions such as sodium ethoxide or hydrogen peroxide commonly employed for partially removing the resinous superficial layer of resin-embedded tissue sections to enhance immunolabeling may be adopted for surface etching of fiber posts containing epoxy resin matrix (Bronson, 2001) (Baskin *et al*, 1979). The possibility of combining a silane/primer solution and a bonding agent has been recently shown to improve bond strength to ceramics (De Munck *et al*, 2004) (Blatz *et al*, 2004).

Microtensile bond strength and nanoleakage evaluation have generally been used

for evaluating the integrity of bonded dentin interfaces (Pashley *et al*, 1999) (Sano *et al*, 1997). Silver nitrate is a powerful tracer used to assess the existence of a microporous zone within or beneath dentin-resin hybrid layers (Tay *et al*, 2002). The purpose of the present study was to examine the effect of different combinations of silane coupling and bonding agents on the adhesion of etched fiber posts to a resin composite core material. The null hypothesis tested was that there are no differences on the adhesive strengths of different silane/resin-based couplings when they are applied for bonding fiber post to a composite core material.

Materials and Methods

Experimental Design

Forty-two quartz fiber posts (DT Light Post #3, RTD, St. Egève, France) were used for testing. DT Light Posts contain pre-tensed quartz fibers (60%) embedded in an epoxy resin matrix (40%).

All posts were pre-treated with 21 wt% sodium ethoxide solution in ethanol (Sigma, Aldrich Chemicals, GmbH, Steinheim, Germany) for 20 min to remove the surface epoxy resin and expose the quartz fibers (Bronson, 2001) (Baskin *et al*, 1979). After etching, the fiber posts were rinsed consecutively with absolute ethanol (Panreac Quimica SA, Barcelona, Spain), 50% ethanol and deionised water (P Selecta S.A. Abrera, Barcelona Spain) until a stable pH of 7 was achieved. To ensure thorough cleaning, all posts were ultrasonically cleaned for 10 min in deionised water, immersed in 96% ethanol and dried with an oil-free air.

The posts were divided into six experimental groups (n=7 posts each) according to the silane/adhesive coupling agent tested on the post surface:

Group 1: Clearfil Porcelain Bond Activator (PBA; Kuraray Medical Inc.) in combination with Clearfil SE Bond (Kuraray Medical Inc.): PBA was mixed (1:1) with the *Primer* solution, air dried after 5 s. The *Bond* was applied, gently dried and light cured for 10 s;

Group 2: PBA in combination with Clearfil Protect Bond (Kuraray Medical Inc.): PBA was mixed (1:1) with the *Primer* solution, air dried after 20 s. The *Bond* was applied, gently dried and light cured for 10 s;

Group 3: PBA in combination with Clearfil Tri S Bond (Kuraray Medical Inc.): PBA was mixed (1:1) with the adhesive, air dried after 5 s and light cured for 10 s;

Group 4: PBA in combination with Clearfil Photo Bond (Kuraray Medical Inc.): PBA, *Universal* and *Catalyst* solutions were mixed proportionally, dried after 2-3 s and light cured for 10 s;

Group 5: PBA in combination with Clearfil New Bond (Kuraray Medical Inc.): PBA, *Universal* and *Catalyst* solutions were mixed proportionally and dried after 2-3 s;

Group 6: Application of a pre-hydrolyzed silane in aqueous solution, dried after 60 s (Monobond-S; Ivoclar-Vivadent, Schaan, Liechtenstein).

Core build-up procedure and microtensile test

After silanization, core build-up was performed following a technique described by Goracci *et al.* (2005). Each post was positioned perpendicularly on a glass slab and maintained with a needle holder at the apical end. A cylindrical plastic matrix (10 mm in diameter) was placed around the coronal portion of the fiber post (with constant diameter), filled by a hybrid composite (Clearfil AP-X, Kuraray Medical Inc.) and light-cured for 40 s (output 600mW/cm²; Optilux, Demetron Res. Corp, Danbury CT). After removing the matrix, this resulted in a cylinder of resin composite built up around the fiber post.

Microtensile sticks were prepared by sectioning the specimens (n=5) with a diamond saw (Isomet 4000, Buehler, Lake Bluff, IL) under water cooling. Two longitudinal cuts were made on the two opposite sides of the post at its outermost periphery, exposing the post surface throughout its length. This created slab of uniform thickness, with the post in the centre and the core build-up on each side. Sticks of 1 mm in thickness were serially obtained sectioning each slab perpendicularly to the post.

A minimum of 25 sticks were tested per group. Each stick was attached with cyanoacrylate glue to a Bencor Multi-T testing assembly (Danville Engineering, San Ramon, CA) and loaded in tension in universal testing machine (Instron, Model 4411, Canton, MA) and at a cross-head speed of 1mm/min until failure. Failure modes were evaluated with a stereomicroscope at 40x magnification (Olympus SZ-CTV, Olympus, Tokyo, Japan) and classified as adhesive (at the post/adhesive interface or between the adhesive and the composite), cohesive (within the resin) and mixed (a combination of the two modes of failure at the same interface).

As the bonded interfaces represented a part of the circumference of a post with a circular cross section, bond strengths were derived using a mathematical formula reported by Bouillaguet *et al.* (2003). The data were analyzed with one-way ANOVA

and post-hoc Student-Newman-Keuls test with statistical significance set at $\alpha = 0.05$.

Micromorphologic examination

The remaining two post-core specimens from each group were evaluated for nanoleakage along the post/core interface. Each cylinder was sectioned into 1.5 mm thick slabs with the Isomet saw under water cooling. The slabs were immersed in a 50% ammoniacal silver nitrate solution for 24 h, according to the procedure described by Tay *et al.* (2002). The tracer-impregnated specimens were rinsed in deionized water and placed in a photodeveloping solution for 8 h to reduce the diamine silver ions into metallic silver grains (Tay *et al.*, 2002). They were subsequently cleaned for documenting the overall nanoleakage patterns using a digital camera attached to a stereomicroscope (Olympus SZ-CTV). Representative specimens were carbon-coated (E5000, Hitachi Ltd, Tokyo, Japan), and examined under a scanning electron microscope (SEM; 1430 VP, LEO Electron Microscopy Ltd., Cambridge, UK). SEM examination was performed using a combination of secondary electron (SEI) mode and back-scattered electron (BSE) mode (50-75% BSE).

Results

The groups in which silane/adhesive couplings were used (i.e. Groups 1-5) attained significantly higher interfacial strength ($p < 0.05$) when compared with the group using only pre-hydrolyzed silane Monobond-S (Table I). Clearfil SE Bond and Tri S Bond mixed with PBA produced the highest microtensile bond strengths.

Failure mode distribution for the different groups is presented in Table I. Higher percentages of mixed failure were encountered when the silane/adhesive coupling agent was applied with composite remnants present on the post surface after debonding (not shown). Conversely, when Monobond-S was used, adhesive failure occurred exclusively along the post/adhesive interface (not shown).

Evaluation of the post/core interface revealed partial removal of the surface epoxy resin matrix, creating “retentive micro-spaces” among the quartz fibres that appeared to be completely infiltrated by the silane/adhesive coupling in Groups 1 to 5 (Fig. 1A). In some areas the fibres were fully embedded by the coupling agent (Fig. 1B). When silane was used alone (Group 6), irregular interfaces with voids and discontinuities were frequently identified (Fig. 1C).

Differences in silver nanoleakage patterns could also be detected at the SEM

level within the silane/adhesive-coupled groups. Isolated silver deposits were present in the coupling layer of the two-step adhesives Clearfil SE Bond and Protect Bond (Figs.2A). Conversely, a more severe silver uptake in the form of silver-filled channels could be identified along the post surface when the silane was mixed with the simplified one-step adhesive Clearfil Tri S Bond (Fig.2B).

Table I. Mean (standard deviation) of microtensile bond strength (MPa) and distribution of failure modes obtained in the tested groups.

Coupling agent	Mean (SD)	Failure mode		
		Mixed	Adhesive	Cohesive
PBA+ Clearfil SE Bond	19.7 (4.9)^a	55 %	41 %	4 %
PBA+ Clearfil Protect Bond	14.5 (3.6)^b	68 %	28%	4 %
PBA+ Clearfil Tri S Bond	17.2 (4.9)^{a b}	80 %	16 %	4 %
PBA+ Clearfil Photo Bond	16.2 (5.1)^b	59 %	41 %	-
PBA + Clearfil New Bond	14.4 (3.8)^b	54 %	42 %	4 %
Monobond-S	10.4 (3.5)^c	15 %	85 %	-

PBA: Porcelain Bond Activator (silane); Clearfil SE Bond: two-step self-etch adhesive; Clearfil Protect Bond: two-step self-etch adhesive; Clearfil Tri S Bond: one-step self-etch adhesive; Clearfil Photo Bond: etch-and-rinse adhesive; Clearfil New Bond: etch-and-rinse adhesive.

Groups with the same superscripts are not statistically significant (p>0.05).

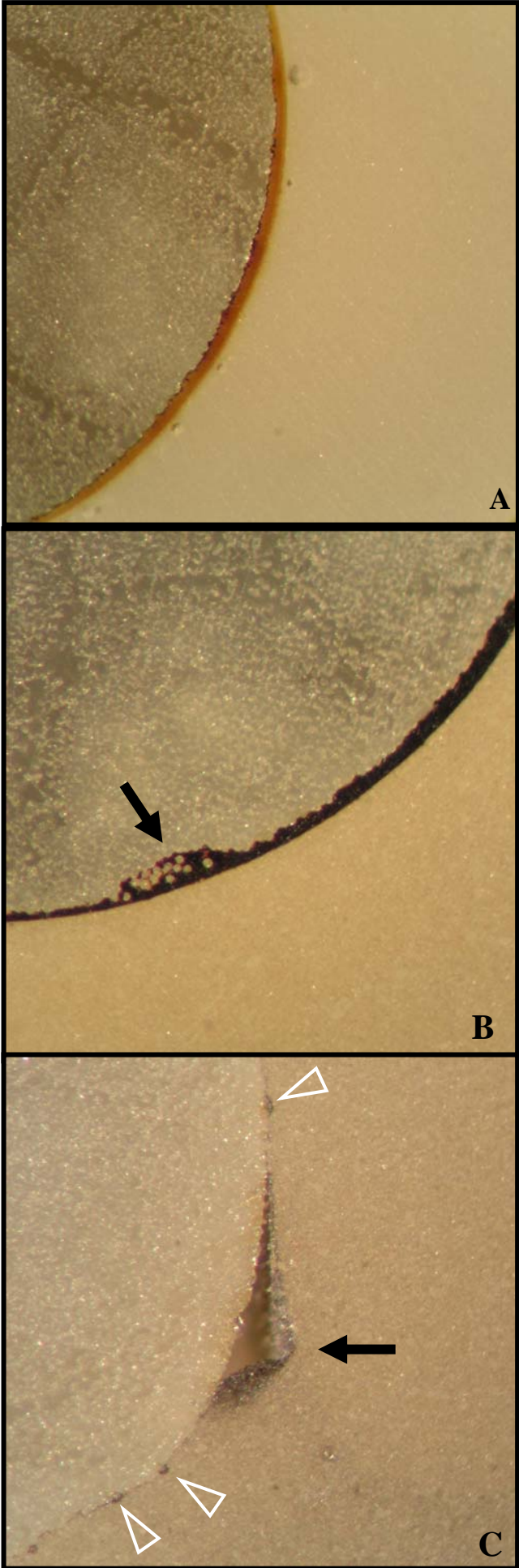


Fig. 1

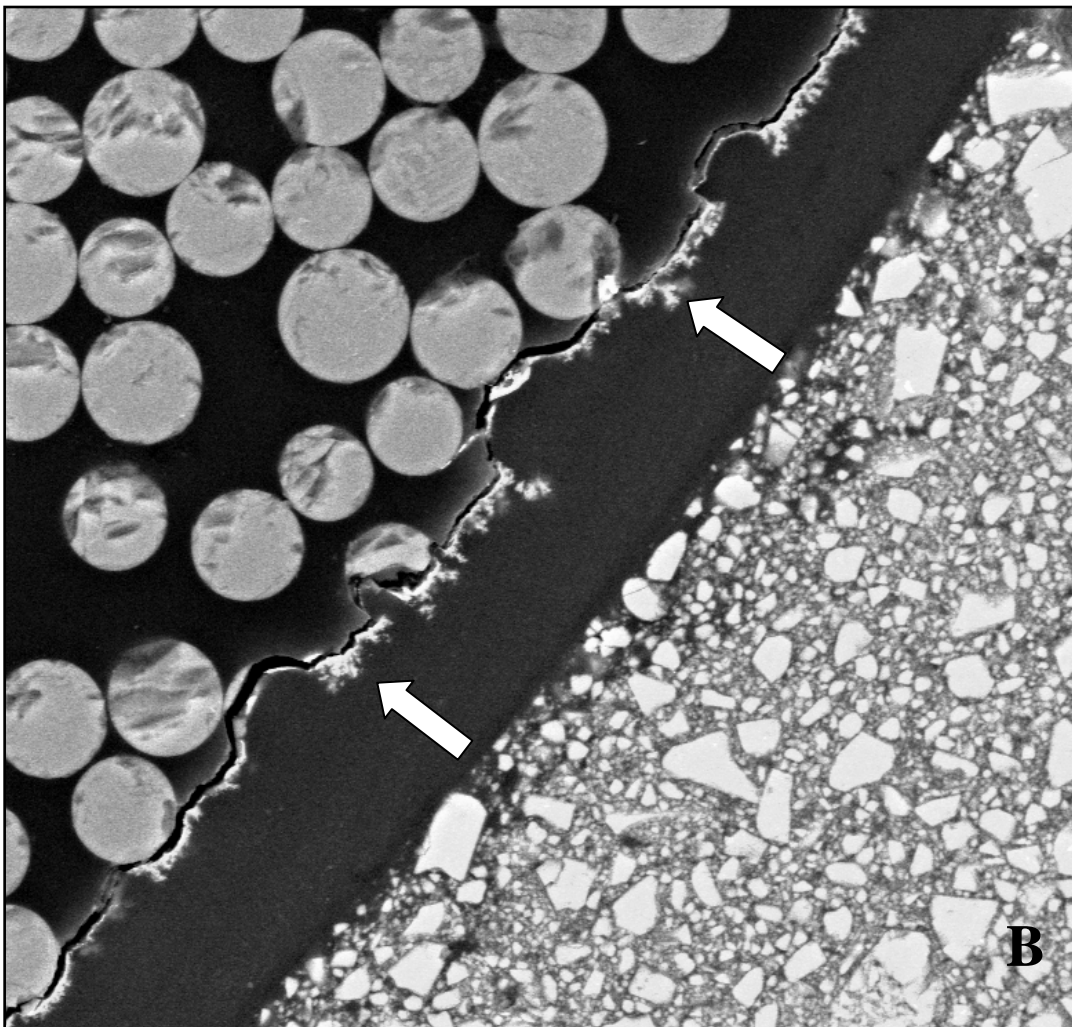
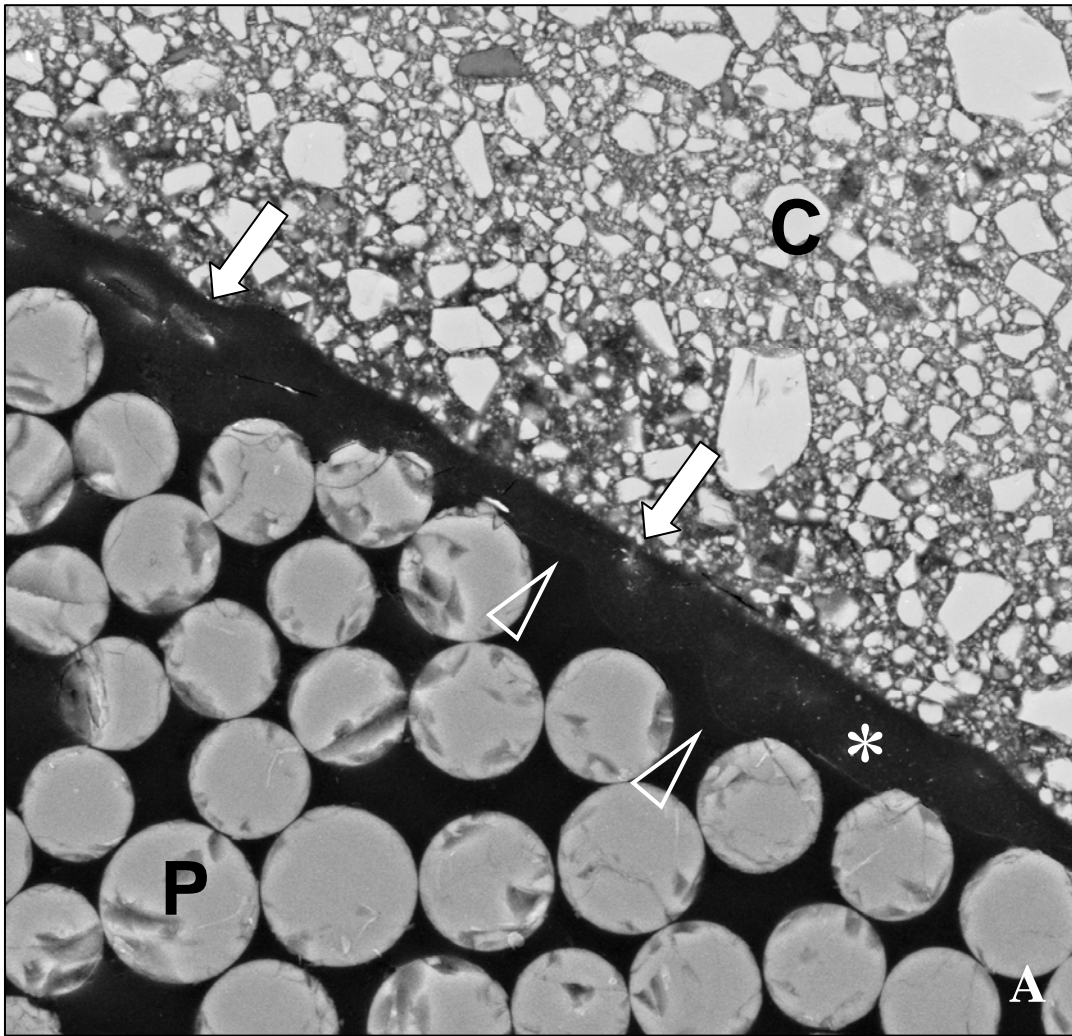


Fig.
2

Fig.1. A. Cross-section of a sample as observed under a stereomicroscope showing the diffusion of the coupling agent on the post surface (PBA+ Clearfil SE Bond). **B.** The silane/adhesive coupling could be detected between the exposed quartz fibers after etching with sodium ethoxide (arrow). **C.** Cross-section showing defects with poor approximation of the core composite (arrow) along the bonded interface when the pre-hydrolyzed silane Monobond-S was applied in the absence of a dentin adhesive. Additional voids (open arrowheads) could be observed even at the light microscopical level.

Fig. 2. Scanning electron micrographs of carbon-coated, silver-impregnated sections that were taken using a combination of 25% secondary electron mode and 75% back scattered electron mode. **A.** Only mild silver uptake (arrows) could be seen within the layer (asterisk) of PBA silane and the two-step self-etch adhesive Clearfil SE Bond (2500x bar= 10 μm). The undulations (open arrowheads) along post (P) surface represent the effect of pre-etching the post surface with sodium ethoxide, creating additional micromechanical retention and partially exposing the quartz fibers for chemical coupling. C: resin composite core. **B.** A continuous layer of tree-like silver channels could be seen along the post surface when PBA silane was mixed with the single-step adhesive Clearfil Tri S Bond (2500x bar= 10 μm).

Discussion

Application of the silane/adhesive couplings resulted in significantly higher microtensile bond strengths and a more uniform adaptation of the composite core to the etched post surface. The null hypothesis is rejected. Ideally, adhesion to fiber posts may be obtained by combining micromechanical retention with chemical bonding effectuated via a silane coupling agent (Monticelli *et al*, 2005). Sodium ethoxide (with formula $\text{CH}_3\text{CH}_2\text{ONa}$, obtained dropping sodium into ethanol) is able to partially dissolve the resin matrix, breaking epoxy resin bonds through a mechanism of substrate oxidation (Bronson, 2001) (Baskin *et al*, 1979), thereby exposing a superficial layer of undamaged quartz fibers.

Whereas the separate application of a silane and a dentin adhesive may lead to the formation of a thick multiphase layer that introduces flaws during each phase of application (Park and Jin, 2001) (Ferrari *et al*, 2005), the synergistic combination of two components creates a unique coupling agent that could have accounted for the better results. For the latter, failure modes were predominantly mixed failures, similar to those that occurred when this silane/adhesive combination was employed for coupling to dental ceramics (Nogami *et al*, 2004).

Two categories of coupling agents were evaluated: a single component pre-hydrolyzed silane (Monobond-S) and two-component systems for “on-demand”

hydrolysis in which the silane is rapidly hydrolyzed when mixed with the acidic phosphate monomer 10-MDP (10-methacryloyloxydecyl dihydrogenphosphate) present in the water-containing dentin adhesives. This results in a condensation reaction catalyzed by the acidic monomer, enabling the trialkoxy silane to perform more efficiently than completely pre-hydrolyzed solutions (Foxton *et al*, 2002). The additional presence of Bis-phenol A polyethoxy dimethacrylate in the coupling solution may also create a link between the composite and the epoxy matrix of the fiber post (Mozner and Salz, 2001).

Unlike bonding to dentin, no residual water was present on the etched post surface during coupling (Tay *et al*, 2003). Thus, differences in bond strengths among silane/adhesive couplings may be associated with the chemical composition of the tested adhesives. Several recently marketed adhesive systems included large amounts of water and other organic solvents, acidic monomers or 2-hydroxyethyl methacrylate (Hashimoto *et al*, 2004). Incomplete evaporation of the solvent during air-drying may occur, affecting the polymerization of the silane/adhesive coupling (Dickens and Beyong, 2005).

Even when high bond strengths were obtained, a diffuse silver tracer expression could be identified along the entire silane/adhesive layer when the PBA was used with the one-step self-etch adhesive Clearfil Tri S Bond. Unlike conventional adhesives (two-step self-etch or etch-and-rinse systems), simplified one-step self-etch adhesives contain hydrophilic and hydrophobic components (Pashley *et al*, 1998). Phase separation of the hydrophobic components may occur when the volatile solvent/water ratio is reduced during evaporation, creating a non-uniform interface. Retention of water-bound hydrophilic components was probably manifested as silver-impregnated channels along the interface (Fig. 2B), rendering the surface more susceptible to water sorption and subsequent hydrolytic degradation (Pashley *et al*, 1998) (Guillot *et al*, 2004) and possibly reducing the longevity of the fiber/composite bond. This issue represents the limitations of contemporary simplified one-step adhesives and their potential use with silane coupling agents is thus questionable. Conversely, the inclusion of a separate hydrophobic resin coating in the two-step adhesives that was applied after the silane/adhesive primer solution could have contributed to the reduction of nanoleakage, with the creation of a more reliable seal of the post surface.

The viscosity of the resin composite influences its adaptation to the post surface (Monticelli *et al*, 2005). The existence of defects along the post-core interface increases

the risk of micro-crack propagation and fracture of the abutment under functional loading. Thus, the application of a fluid resin with the silane agent is advantageous for improving surface wetting and interfacial adaptation of the more viscous resin composite core material (Monticelli *et al*, 2005).

The results of this study shed new lights on the issue that high interfacial bond strength between the fiber post and the core material may not be sufficient to ensure the quality of a restoration (Ferrari *et al*, 2005), as nanoleakage within the coupling layer represents potential sites of water sorption that may expedite the degradation of the post/resin interface.

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Chapter 6

6.1. Summary

Almost no procedure in dentistry has evolved more than the restoration of badly-broken pulpless teeth, both in philosophy and clinical procedures. The issues related to this topic include luting procedures, adhesive techniques, core materials and technique sensitivity in core placement. The continuous development of endodontics and adhesive dentistry is improving the quality and the survival rate of these restorations, according to the aim of the “Minimal Intervention” philosophy (Mjör *et al*, 2002). In this prospect, the use of fiber posts in combination with direct core build-up materials increased in the last few years.

In the initial part of this project (Chapter 1), an introduction of the main topic of the study has been presented. After a short description of the essential characteristics of pulpless teeth, some considerations have been made about the restorative techniques.

Arguments for replacing the standard of the custom cast post and core with fiber posts and composites have been discussed. Superior aesthetics and ease of use of direct materials are some of the major aspect of the clinical procedure (Vichi *et al*, 2000) (Heydecke *et al*, 2002).

Despite of their lower cost and the simplified chair-side procedure, particular attention has been given to the evolution of the restoring concept when using fiber posts. Only few decades ago, it was considered as necessary to place posts and cores in every tooth that received an endodontic treatment: at that time posts were thought to reinforce teeth (Shillingburg *et al*, 1997).

Subsequently, many *in vitro* and clinical researches concluded that posts have to be used primarily to retain the build-up material to replace the coronal portion of the tooth (Sorensen and Martinoff, 1984).

As far as post recommendation is concerned, it is the amount of residual coronal structure to determine when a post and core restoration is needed (Assif *et al*, 1994) (Cohen *et al*, 1996).

Moreover, during the last few years there has been a shift away from rigid materials to new materials whose properties are closer to sound dentin, allowing enduring the masticatory forces for prolonged periods.

The various components of the restoration (fiber posts, luting cement, core material and dentin) represent a more homogeneous complex: an uniform stress distribution to the residual tooth structure may take place, significantly reducing the risk of irreversible root fracture (Akkayan and Gulmetz, 2002) (Newman *et al*, 2003).

Regarding the final restoration of structurally compromised endodontically treated teeth, recent investigations revealed that fixed prosthodontics continues to be accomplished in significant quantities, even if the possibility of using fiber posts in conjunction with direct composite restorations (without additional crown coverage) is becoming a reliable alternative, ensuring long-term service (Grandini, 2004) (Creugers *et al*, 2005).

In this sense, special attention has to be given regarding the material selection. In order to choose the proper composite to be used in combination with fiber posts, an experimental work was carried out among the materials available commercially (Chapter 2). This research, based on the evaluation of the morphology of the core build-up, demonstrated that the quality of the restorations is dependent on the viscosity of the composite and its handling: flowable composites exhibited the best adaptation to the post surface. This statement is supported by some recent literature data indicating low viscosity composites as suitable for core build-up (Goracci *et al*, 2005) (Stober and Rammelsberg, 2005).

The author of this thesis, however, is more convinced that the low viscosity together with a higher filler content are the key properties of a resinous core material to ensure lower defects development and superior resistance of the abutment (Sadek *et al*, unpublished data) (Ferrari *et al*, 2005).

The application mode of the composite material also influences the final result. In Chapter 3, a comparison between two different direct restorative techniques was carried out revealing that the quality of the pre-prosthetic restoration is affected by the operative procedure: independently from the properties of the composite, a higher percentage of defects was recorded when pre-shaped forms were used to build-up the core onto fiber posts. The incremental technique is advisable also for restoring the abutment.

The various indications coming from the results of the studies described before found a confirmation in Chapter 4, in which a prospective study has been reported. This clinical evaluation was carried out during a follow-up period of 2-3 years, revealing that flowable composites may be of valid support to prosthetic crowns.

In Chapter 5, problems associated with bonding to fiber posts were considered. A series of preliminary observations suggested that the interfacial strength between composite core and fiber post is still relatively weak, mainly due to chemical incompatibility among different components. Silanes, commonly applied in dentistry to overcome these limits, may only partially solve the problem (Aksornmuang *et al*, 2004) (Goracci *et al*, 2005). The idea of applying on posts the concept of chemical and micromechanical bonding as well as on tooth substrates seemed to be of help and led to an insight into different scientific and technical disciplines.

More precisely, fiber reinforced material technology gave suggestion to test different fiber post superficial treatments with the aim of obtaining a proper adhesion. Different researches were carried out and described: microtensile bond strength test and microscopic analysis were performed for this purpose.

This approach successfully validated the effectiveness of different methodologies: among the tested chemical agents, hydrogen peroxide etching of fiber post surface provided an easy and clinically feasible method for enhancing bond strength.

An additional benefit may be reached applying silane in mixture with a bonding agent or optimizing its application mode on the etched post surface. However, when interpreting these favourable results in terms of bond strength, one should be aware of the limits of some materials. Simplified “single-bottle” adhesives in mixture with silane did not exhibit the satisfactory microscopic features of a reliable bond, revealing an intrinsic limit of these new systems, not completely solved (Tay and Pashley, 2003). However, microtensile test provides only a static assessment of interfacial bond strength (Goracci, 2004): fatigue tests, aging and clinical trials are needed to assess the durability of bond, contributing to validate the method.

Nevertheless, this last part of the project may open a new prospective in restorative dentistry, revealing that dental research cannot be apart from a multidisciplinary approach and critical evaluation of materials and techniques.

6.2. Conclusions

The following conclusions may be drawn from the basic and clinical evaluation of composites and fiber posts in the restoration of endodontically treated teeth:

- 1) Low viscosity composites can be used as core build-up materials thanks to their adaptability to the post surface and the integrity of the abutment.
- 2) The incremental technique is appropriate to build-up the core around fiber posts, ensuring better adaptation to the post surface and lower defects formation in the bulk of the abutment.
- 3) Fiber posts and flowable composite materials can be safely used to build-up the abutment in combination with ceramic crowns, providing good aesthetics and support to prosthetic restorations.
- 4) Reliable and predictable bonding of composites to fiber posts is significantly enhanced by etching the post surface. This will promote the coupling effectiveness of silane and the formation of a micromechanical interlocking between the two components.
- 5) As far as silane agents are concerned, the use of warm air-drying may be of help to achieve a more effective coupling of the etched post surface.
- 6) The combined application of a silane solution in mixture with adhesive systems on the etched post surface, may contribute to strengthen the chemical and micromechanical bond with the composite core.

Future directions

Dental research targeted at the development of ideal materials has been ongoing for many years. Since its introduction in the 50s, Adhesion has undergone considerable maturation, increasing the role exerted in daily practice and dental investigation.

The desire to achieve a long-lasting, natural-looking restoration is a driving force behind the continuous quest of clinicians and dental manufactures and leads researchers to continuously develop their thoughts.

To get this purpose, it is not possible to be aware from the inflow and the experience of different scientific disciplines. Chemistry, material science and engineering may be of help to understand materials properties trying to optimize them.

This approach is much more relevant if one considers that the behaviour of many synthetic materials is closer to biological tissues, included enamel and dentin.

An example of multidisciplinary approach in dentistry can be represented by implantology, considering how much surface characterization of titanium affect the biological response of bone and revealing how material science may assist biology.

The main goal of fiber reinforced material science is ensure a strong bond between different components to create a unique multiphase material with improved performances. A similar concept may be applied in restorative dentistry, with the purpose of creating a single unit between the tooth and the restoration.

In the next years, tissue engineering probably will indicate the future of dental materials research replacing missing teeth and lost tooth structure.

6.3. Riassunto, conclusioni e direzioni future

La ricostruzione dei denti trattati endodonticamente rappresenta forse uno degli aspetti dell'odontoiatria che più si è evoluto negli ultimi anni da un punto di vista teorico e delle procedure cliniche. Numerose sono le variabili legate a tale trattamento, quali la fase di cementazione, la selezione dei materiali da ricostruzione o della tecnica operativa. Le continue innovazioni in campo endodontico e dell'odontoiatria adesiva hanno migliorato la qualità e la durata di tali ricostruzioni con un'attenzione sempre crescente al concetto di odontoiatria minimamente invasiva (Mjör *et al*, 2002). In tale prospettiva, l'uso dei perni in fibra e dei materiali compositi per il restauro diretto dei denti trattati endodonticamente è significativamente aumentato negli ultimi anni.

Nella parte iniziale di questa tesi (Capitolo 1), è stata effettuata una introduzione generale all'argomento in studio. Dopo una breve descrizione delle principali caratteristiche dei denti trattati endodonticamente, sono state fatte alcune considerazioni circa la scelta delle tecniche di ricostruzione. Sono stati presentati e discussi i principi sulla base dei quali i tradizionali perni moncone fusi sono stati progressivamente sostituiti da tecniche più conservative di restauro pre-protetico. Miglior risultato estetico e facilità di uso sono alcune delle caratteristiche principali delle nuove procedure operative (Vichi *et al*, 2000) (Heydecke *et al*, 2002).

Oltre ai vantaggi derivanti da una riduzione dei costi e dei tempi di lavoro, una attenzione particolare è stata riservata anche all'evoluzione del concetto di ricostruzione derivante dall'uso dei perni in fibra. Solo pochi anni fa si pensava che tutti i denti trattati endodonticamente dovessero ricevere un perno moncone: si riteneva infatti che i perni potessero rinforzare la struttura dentale residua (Shillingburg *et al*, 1997).

Studi *in vitro* e clinici hanno dimostrato che l'indicazione principale all'uso dei perni in fibra è la ritenzione del materiale composito utilizzato per la ricostruzione coronale (Sorensen and Martinoff, 1984).

Per quanto riguarda poi i criteri di scelta, la quantità di dentina coronale residua costituisce un fattore determinante per valutare l'effettiva necessità di eseguire una ricostruzione diretta con perni in fibra. (Assif *et al*, 1994) (Cohen *et al*, 1996).

Inoltre, nel corso degli ultimi anni si è assistito ad un graduale superamento dei tradizionali materiali rigidi da ricostruzione da parte di materiali, quali le resine

composite, in grado di resistere alle forze masticatorie e le cui proprietà sono definite simili a quelle della dentina sana.

Le diverse componenti della ricostruzione (perno in fibra, cemento resinoso, composito e dentina) formano così un complesso più omogeneo: si realizza una uniforme distribuzione dello stress masticatorio alla struttura dentaria residua, riducendo di forma significativa il rischio di fratture radicolari (Akkayan and Gulmetz, 2002) (Newman *et al*, 2003).

Per quanto riguarda la ricostruzione finale dei denti trattati endodonticamente, studi recenti hanno dimostrato che la scelta del restauro protesico continua ad essere la più predicibile, anche se la possibilità di combinare l'utilizzo dei perni in fibra con una ricostruzione diretta in composito (senza la successiva cementazione di una corona protesica) sta diventando un'alternativa concreta anche nel lungo termine (Grandini, 2004) (Creugers *et al*, 2005).

A tal proposito, un'attenzione particolare deve essere riservata alla scelta del materiale da ricostruzione. Con l'obiettivo di individuare il composito più adeguato da essere utilizzato in combinazione con i perni in fibra, è stato eseguito uno studio sperimentale utilizzando alcuni dei materiali compositi presenti in commercio (Capitolo 2). Questo studio, basato sulla valutazione delle caratteristiche morfologiche di ricostruzioni perno/composito, ha dimostrato che la viscosità e la modalità di applicazione delle resine composite influenza la qualità del restauro finale: i compositi di tipo flowable si adattano in maniera migliore alla superficie del perno. Quanto affermato è confermato anche dai risultati di alcuni recenti lavori di ricerca in cui le resine composite a bassa viscosità vengono considerate adatte alla ricostruzione pre-protesica del moncone (Goracci *et al*, 2005) (Stober and Rammelsberg, 2005).

Alla luce di valutazioni successive l'autore di questa tesi ritiene che la bassa viscosità insieme ad un elevato contenuto di riempitivo siano due fattori chiave per garantire la resistenza del materiale da core assicurando al tempo stesso un buon adattamento alla superficie del perno (Sadek *et al*, unpublished data) (Ferrari *et al*, 2005).

Anche le tecniche operative possono influenzare la qualità della ricostruzione finale. Nel Capitolo 3, è stato eseguito un confronto tra due diverse tecniche di ricostruzione diretta del moncone rivelando che la qualità della ricostruzione pre-protesica dipende dalla selezione della metodica operativa: indipendentemente dalle proprietà del materiale composito, l'uso di matrici preformate provoca una percentuale

più elevata di difetti nel moncone e a livello dell'interfaccia con il perno. La tecnica incrementale al contrario, sembra fornire risultati più predicibili.

Le indicazioni pervenute dai lavori in vitro trovano una conferma nel Capitolo 4 in cui viene presentato uno studio prospettico. Tale ricerca clinica è stata condotta durante un follow-up di 2-3 anni, dimostrando che i compositi di tipo flowable ed i perni in fibra sono adatti alla ricostruzione pre-protetica del moncone ed offrono un valido supporto a corone in ceramica.

Nel Capitolo 5 sono state analizzate le problematiche relative all'adesione alla superficie dei perni in fibra. Alla luce di alcune osservazioni preliminari, si evince che la forza di adesione all'interfaccia perno/composito è relativamente debole, prevalentemente a causa di una incompatibilità chimica tra le diverse componenti. I silani sono comunemente applicati anche in ambito odontoiatrico allo scopo di superare questo limite, ma possono risolvere la problematica solo in parte (Aksornmuang *et al*, 2004) (Goracci *et al*, 2005). L'idea di applicare alla superficie del perno i principi di adesione chimica e micromeccanica applicati ai tessuti dentali, è sembrata di aiuto ed è servita come punto di partenza per valutare diverse discipline tecniche e scientifiche.

Più precisamente, la tecnologia dei materiali compositi rinforzati con fibre ha fornito dei suggerimenti per effettuare dei trattamenti della superficie del perno con l'obiettivo di migliorare il meccanismo di adesione. Sono stati realizzati diversi studi in vitro avvalendosi della tecnica microtensile e della microscopia elettronica a scansione.

Questo approccio ha permesso di evidenziare l'efficacia di diversi agenti chimici tra i quali il perossido di idrogeno ha rappresentato il più semplice e clinicamente attuabile metodo per mordenzare la superficie del perno in fibra, aumentando l'adesione dei materiali compositi.

Un miglioramento aggiuntivo può essere ottenuto combinando il silano con un appropriato sistema adesivo o ottimizzando la sua modalità di applicazione sulla superficie mordenzata del perno. Di fatto, questi risultati favorevoli in termini di forza di adesione non devono far dimenticare i limiti di alcuni materiali. I sistemi adesivi utilizzati in combinazione con il silano, pur determinando degli elevati valori di forza di adesione, possono non presentare soddisfacenti proprietà morfologiche, come nel caso degli adesivi di tipo one-bottle, rivelando un limite intrinseco di questi nuovi sistemi non ancora risolto (Tay and Pashley, 2003). Di fatto però il test microtensile fornisce solo una valutazione statica della forza di adesione a livello dell'interfaccia perno/composito e dovrebbe essere completato da studi successivi come test di fatica, di

invecchiamento e studi clinici allo scopo di valutare la durata del legame adesivo e di confermare l'efficacia del metodo (Goracci, 2004).

La parte finale della tesi può rappresentare una nuova prospettiva nell'odontoiatria conservativa, dimostrando come la ricerca in campo dentale non possa prescindere dalla valutazione critica dei materiali e delle tecniche nonché da un approccio di tipo multidisciplinare.

Alla luce delle valutazioni in vitro e cliniche sulla ricostruzione dei denti trattati endodonticamente possono essere tratte le seguenti conclusioni:

- 7) Le resine composite a bassa viscosità possono essere utilizzate per la ricostruzione del moncone protesico grazie alla capacità di adattamento alla superficie del perno ed all'inegria della ricostruzione.
- 8) La tecnica incrementale è adatta alla ricostruzione del moncone protesico, permettendo un buon adattamento del composito alla superficie del perno ed una minor formazione di difetti.
- 9) I perni in fibra ed i compositi di tipo flowable possono essere utilizzati clinicamente nelle ricostruzioni pre-protesiche garantendo un'estetica soddisfacente ed un adeguato supporto a corone protesiche in sola ceramica.
- 10) La qualità dell'adesione dei materiali compositi ai perni in fibra può essere migliorata con risultati predicibili mediante la mordenzatura della superficie del perno. Ciò permette la formazione di un'adesione di tipo micromeccanico tra le due componenti della ricostruzione.
- 11) La fase di silanizzazione può essere ottimizzata seccando con aria calda la superficie mordenzata del perno in fibra, così da potenziare l'efficacia dell'agente di accoppiamento.
- 12) L'applicazione del silano in combinazione con un appropriato sistema adesivo sulla superficie mordenzata del perno può contribuire a rinforzare il meccanismo di adesione chimica e micromeccanica tra resina composita e perno in fibra.

L'obiettivo principale della ricerca in campo dentale è da sempre rappresentato dalla creazione di un materiale ideale. Fin dalla sua introduzione all'inizio degli anni 50, l'Odontoiatria Adesiva ha subito un continuo sviluppo accrescendo la sua importanza nell'ambito clinico e della ricerca.

Il desiderio di poter ottenere delle ricostruzioni durature ed estetiche sta prendendo sempre più consistenza, anche alla luce delle richieste cliniche e del mercato, stimolando i ricercatori ad evolvere continuamente le proprie idee.

A tal proposito, appare oggi più che mai imprescindibile avvalersi delle conoscenze e dell'esperienza di altre discipline scientifiche. La chimica, la scienza dei materiali e l'ingegneria possono rappresentare un valido aiuto per comprendere le proprietà dei materiali ottimizzandole ed adattandole alla necessità dell'odontoiatria.

Ciò risulta ancora più rilevante pensando che il comportamento di molti materiali di sintesi è simile a quello dei tessuti biologici, come la dentina o lo smalto.

L'implantologia rappresenta probabilmente il primo risultato di un approccio multidisciplinare se si pensa a quanto le caratteristiche superficiali dell'impianto possano influenzare la risposta biologica del tessuto osseo, dimostrando come la scienza dei materiali possa assistere la biologia.

L'obiettivo principale che la ricerca nel campo dei materiali rinforzati in fibra si prefigge è quello di assicurare un legame forte tra le diverse componenti in modo da creare un materiale unico con caratteristiche superiori. Un simile risultato dovrebbe essere raggiunto anche dall'odontoiatria conservativa formando una unione tra il dente ed il materiale da ricostruzione. Sarà probabilmente compito dell'ingegneria tissutale fornire la risposta a tale richiesta indicando il futuro della ricerca in campo odontoiatrico.

6.4 Resumen, conclusiones y direcciones futuras

Casi no hay tramo en la Odontología que se han evolucionado más de la restauración de los dientes endodonciados, con respecto a la filosofía y a las técnicas operativas. Los aspectos que están relacionados con este tema incluyen la fase de cementación, las técnicas adhesivas, la selección de los materiales para la restauración y la misma técnica operativa. La calidad y la durabilidad de las restauraciones de los dientes endodonciados se ha mejorado por efecto del continuo perfeccionamiento de la endodoncia y de la odontología adhesiva de acuerdo con los objetivos de la filosofía de la “intervención mínima” (Mjör *et al*, 2002). Segundo esta perspectiva, el uso de los postes de fibra en conjunción a los materiales composites para la restauración directa de los dientes endodonciados aumentó en los últimos años.

En la parte inicial de esta tesis doctoral (Capítulo 1), se realizó una introducción al principal argumento en estudio. Después de una breve descripción de las características principales de los dientes desvitalizados, se comentaron las técnicas de restauración.

Se discutieron las razones por las cuales los postes metálicos tradicionales hayan sido sustituidos por los postes de fibra y las resinas compuestas. Las mejoradas propiedades estéticas y la facilidad de uso de los materiales directos son unos de los aspectos más importantes del tratamiento clínico (Vichi *et al*, 2000) (Heydecke *et al*, 2002).

A pesar de un costo inferior y de la simplificación de las fases operativas, la evaluación del concepto de restauración con postes de fibra merece una atención particular. Hace unos años se consideraba necesaria la reconstrucción con postes de todos los dientes endodonciados: se pensaba que los postes pudieran fortalecer el diente (Shillingburg *et al*, 1997).

En seguida, los estudios de laboratorio y clínicos llegaron a la conclusión de que los postes tienen que ser utilizados principalmente para la retención de la restauración coronal del diente (Sorensen and Martinoff, 1984).

Con respecto al uso de los postes, es la cantidad de estructura dentaria residual la que establece la necesidad real de recurrir a este tipo de restauración (Assif *et al*, 1994) (Cohen *et al*, 1996).

Además, en los últimos años se realizó un cambio gradual de los materiales de restauración más rígidos con materiales nuevos las cuyas propiedades son más cercanas

a las de la dentina sana, y que son capaces de resistir a las fuerzas masticatorias por un tiempo prolongado.

Las distintas componentes de la restauración (postes de fibra, cemento adhesivo, resinas compuestas y dentina) representan un complejo homogéneo: la distribución del estrés a la estructura dentaria residual se realiza de forma más uniforme, reduciendo significativamente el riesgo de fracturas radiculares irreversibles (Akkayan and Gulmetz, 2002) (Newman *et al*, 2003).

Con respecto a la restauración final de los dientes endodonciados, investigaciones recientes demostraron que el tratamiento protésico fijo sigue siendo el más utilizado y seguro, aunque el uso de postes de fibra y resinas compuestas para la realización de restauraciones directas (sin algún recubrimiento protésico) se pueda considerar una alternativa concreta con resultados positivos a largo plazo (Grandini, 2004) (Creugers *et al*, 2005).

La selección de los materiales merece una atención particular. Para una correcta selección de la resina compuesta da utilizar con los postes de fibra, se realizó un estudio experimental de comparación entre distintos materiales disponibles en el mercado (Capítulo 2). El estudio se propuso de evaluar las características morfológicas de las restauraciones, y demostró que la calidad de las restauraciones depende de la viscosidad de la resina compuesta y de su manipulación: los composites de tipo fluido mostraron una mejor adaptación a la superficie del poste. En literatura esta afirmación es sostenida por algunos resultados recientes que indican las resinas compuestas de baja viscosidad como adecuadas para la reconstrucción de muñones (Goracci *et al*, 2005) (Stober and Rammelsberg, 2005).

El autor de esta tesis doctoral es más convencido de que la baja viscosidad y el alto contenido de relleno sean las propiedades clave de una resina compuesta para la restauración de los dientes endodonciados al fin de garantizar un menor número de defectos y una mayor resistencia (Sadek *et al*, datos no publicados) (Ferrari *et al*, 2005).

La modalidad de uso de las resinas compuestas también influye sobre el resultado final. En el Capítulo 3, se realizó una comparación entre dos distintas técnicas operativas de restauración poniendo de manifiesto que la calidad de la restauración depende de la técnica utilizada: sin considerar las propiedades del composite, el uso de matrices determinó un porcentaje superior de defectos a nivel de la restauración y de la interfase con el poste de fibra. Mejores resultados fueron obtenidos con la técnica de estratificación.

Los distintos resultados de los trabajos de investigación *in Vitro* fueron confirmados en el Capítulo 4 en un estudio clínico prospectivo. La evaluación clínica se desarrolló durante 2-3 años y puso de manifiesto como dientes endodonciados restaurados en resina compuesta de tipo fluido puedan soportar coronas protésicas.

En el Capítulo 5, se consideraron los problemas relacionados con la adhesión a los postes de fibra. Observaciones preliminares sugirieron que la fuerza de adhesión entre la resina compuesta y el poste de fibra es aun débil, principalmente debido a la incompatibilidad entre los distintos materiales. El agente de acoplamiento silano, normalmente aplicado en odontología, consigue solucionar este límite solo parcialmente (Aksornmuang *et al*, 2004) (Goracci *et al*, 2005). Una solución posible puede derivar de la idea de aplicar a los postes de fibra los conceptos de adhesión química y micro-mecánica utilizados para la sustancia dental y implica la valoración de aspectos de distintas materias científicas y tecnológicas.

En particular, la tecnología de los materiales reforzados en fibra fue de sugerencia para el análisis de distintos métodos de grabado superficial de la superficie de los postes con el objetivo de mejorar la fuerza de adhesión.

Distintos proyectos de investigación fueron desarrollados y se utilizaron la prueba de microtensión y la microscopía electrónica de barrido.

Este enfoque validó con suceso el uso de métodos distintos: entre los agentes químicos testados, el grabado con peróxido de hidrógeno se mostró un tratamiento viable para el tratamiento de la superficie de los postes de fibra.

Una ventaja adicional puede ser alcanzada aplicando el silano y el adhesivo al mismo tiempo o mejorando las técnicas de aplicación sobre la superficie de los postes de fibra anteriormente grabados. Sin embargo, cuando se analizan los valores de fuerza de adhesión es importante ser consciente de las limitaciones de los materiales. El sistema adhesivo simplificado de “paso único” aplicado conjuntamente al silano, no mostró características microscópicas satisfactorias, demostrando que las propiedades de los nuevos sistemas adhesivos deberían ser perfeccionadas (Tay and Pashley, 2003).

La prueba de microtensión solo prevee una medida de la fuerza estática de una interfaz (Goracci, 2004): pruebas de carga, de envejecimiento e investigaciones clínicas deberían ser realizadas para establecer la durabilidad de la adhesión y para validar el método.

Sin embargo, la última parte de este proyecto puede abrir una nueva perspectiva en la odontología operatoria, demostrando como la investigación en materia dental no pueda presumir de un análisis multidisciplinario de las técnicas y de los materiales.

Las conclusiones y recomendaciones siguientes se pueden deducir de las evaluaciones básicas y clínicas en el uso de resinas compuestas y de postes de fibra en la restauración de los dientes endodonciados:

- 1) Las resinas compuestas de baja viscosidad pueden ser utilizadas para la restauración debido a la buena adaptación de las mismas a la superficie de los postes y a la integridad de la restauración;
- 2) La técnica operatoria de estratificación del composite puede ser utilizada en la restauración de los dientes endodonciados con postes de fibra, asegurando una superior calidad del resultado;
- 3) Los postes de fibra y las resinas compuestas de tipo fluido garantizan un válido soporte para la retención de coronas protésicas de sola cerámica y mejoradas propiedades estéticas;
- 4) El grabado superficial de los postes de fibra permite de obtener una adhesión mas fuerte y previsible a las resinas compuestas;
- 5) Con respecto a los agentes de acoplamiento silano, el uso de aire caliente para favorecer la evaporación del solvente contribuye a mejorar la adhesión a la superficie grabada del poste de fibra;
- 6) La adhesión química y micro-mecánica entre la resina compuesta y el poste de fibra aumenta por efecto de la aplicación conjunta del agente de acoplamiento silano y del sistema adhesivo.

La investigación en materia odontológica finalizada al desarrollo de materiales ideales para la restauración de los dientes sigue desde hace muchos años.

Desde que fue introducido al principio de los años cincuenta, el concepto de adhesión cambió de forma considerable, adquiriendo un papel importante en la odontología clínica diaria y en la investigación.

El deseo de obtener restauraciones durables y estéticas se considera el motivo clave que lleva los investigadores a mejorar siempre sus conocimientos detrás también de la continua demanda de los odontólogos clínicos y de la industria.

Para obtener eso es necesario tener en cuenta de la influencia y de la experiencia de distintas disciplinas científicas. La química, la ciencia de los materiales y la ingeniería pueden ser de ayuda para comprender las propiedades de los materiales de uso odontológico con el propósito de mejorarlas.

Esta aproximación es mucho más relevante si se considera que el comportamiento de algunos materiales sintéticos es muy parecido a lo de los tejidos biológicos, esmalte y dentina también.

En ejemplo de aproximación multidisciplinaria en odontología la representa la implantología, si se considera como las características superficiales del titanio influyan en la respuesta biológica del hueso y como la ciencia de los materiales pueda en este caso soportar la biología.

El objetivo principal que la ciencia de los materiales reforzados en fibra se plantea es lo de asegurar una adhesión fuerte entre las componentes distintas para crear un material único multifase con características superiores. Un concepto similar podría ser aplicado a la odontología operatoria con el objetivo de crear una unidad sola entre el diente y la restauración.

A lo largo de los próximos años, será la ingeniería tisútal la que probablemente nos indicará el futuro de la investigación en odontología y la posibilidad de sustituir los dientes o la estructura dental.

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Professional positions

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Membership in Dental Societies:

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