NEW PERSPECTIVES IN LUTING INDIRECT RESTORATIONS:
EVALUATION OF THE BONDING PERFORMANCE OF SELF-
ADHESIVE RESIN CEMENTS

Nuevas perspectivas en el cementado de las restauraciones indirectas:
Valoración de la capacidad de unión de los cementos resinosos auto-
adhesivos

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(Nuevas perspectivas en el cementado de las restauraciones indirectas: Valoración de la capacidad de unión de los cementos resinosos auto-adhesivos)

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CONTENTS

Chapter 1
1.1 General Introduction .......................................................... 7
1.2 Introducción general ............................................................ 10
1.3 An overview of the luting materials available for the cementation of indirect restorations ......................................................... 15
1.4 Self-adhesive resin cements: composition and properties ............. 19
1.5 Self-adhesive resin cements vs cements based on multi-step systems ...... 22
1.6 Dentin characteristics as adhesive substrate and the influence of the hydration state of dentin on the bonding performance of adhesive systems and cements ......................................................... 23
1.7 The use of fiber posts in dentistry .............................................. 26
1.8 Post surface treatments for improving the cement/post bond .......... 29
1.9 An overview of self-adhesive resin cements and their clinical applications ............................................................................. 31
References ..................................................................................... 35

Chapter 2
2.1 The interaction between self-adhesive cements and the dentin substrate .................................................................................. 52
References ..................................................................................... 54
2.2 Limited decalcification/diffusion of Self-adhesive Cements into Dentin ..................................................................................... 56

Chapter 3
3.1 Vital dentin as bonding substrate ................................................. 74
References ............................................................................................................. 76

3.2 Effect of simulated pulpal pressure on self-adhesive cements bonding to dentin ............................................................................................................. 77

Chapter 4
4.1 The role of smear layer on the bonding quality of self-adhesive resin cements ............................................................................................................. 99
References ............................................................................................................. 100
4.2 Dentin treatment effects on the bonding performance of self-adhesive resin cements ............................................................................................................. 104

Chapter 5
5.1 Self-adhesive cements and fiber posts ............................................................ 126
References ............................................................................................................. 128
5.2 Evaluation of the push-out bond strengths of self-adhesive resin cements to fiber posts ..................................................................................................... 130
5.3 Effect of thermocycling on the bond strength of self-adhesive cements to fiber posts ................................................................................................. 145

Chapter 6
6.1 Post surface treatments for improving the adhesive bonds ......................... 162
References ............................................................................................................. 164
6.2 Surface roughness analysis of fiber post conditioning processes ......... 166
6.3 Effects of post surface treatments on the bond strength of self-adhesive resin cements ......................................................................................... 182
Chapter 7

7.1 Summary, Conclusions and Future Directions………………………200
7.2 Riassunto, conclusioni e direzioni future…………………………208
7.3 Resumen, conclusiones y direcciones futures………………………217
7.4 Zusammenfassung, schlussfolgerungen und zukünftige ausrichtung………………………………………………………………………226
7.5 Resumé, conclusions et directions futures…………………………235
7.6 Resumo, Conclusões, Futuras perspectivas…………………………244

Complete list of References…………………………………………………………252
Curriculum Vitae……………………………………………………………………..285
Acknowledgements………………………………………………………………….296
Chapter 1

1.1 General Introduction

The clinical success of an indirect restoration is partially related to the material and technique used for the luting procedures (Hickel and Manhart, 2001). An inadequate marginal adaptation of the cement on the bonding interfaces and a decreased retention mainly cause the premature failure of a restoration (Mijör and Gordan, 2002) (Mijör et al, 2002).

A reliable adhesion is obtained when the luting agent and the bonding substrate intimately contact. New products are continually developed to improve and simplify luting procedures. When using cements based on a total-etch adhesive system, the bonding mechanism is based on the removal of the dentin mineral components as to create a demineralized area of 1.5 µm while leaving intact the collagen fibrils (Van Meerbeek et al, 2003). This condition is achieved using acidic solutions. Acid-etching the dental substrate promotes the complete removal of the smear layer thus creating unfilled spaces (Glasspoole et al, 2002). Ideally, the resin should fulfill the voids left by the removal of the mineral content, infiltrate the dentinal tubules and stabilize the collagen matrix, as to form an hybrid layer between dentin and resin (Nakabayashi et al, 1982) (Pashley et al, 1993) (Titley et al, 1994) (Van Meerbeek et al, 1992). Hybrid layer has been considered necessary for an effective adhesion when using cements based on multi-steps adhesive systems (Van Meerbeek et al, 2003). However, this technique is considered too operator- and material-dependent.
Some variables may influence the clinical outcome of a restoration: the operator, the design of the restoration, the materials, the intra-oral conditions and the patient (Bayne, 2007). The first two factors are directly related to the clinician ability. Regarding the material, it is indispensable to understand its characteristics as well as the mechanical/physical properties and clinical behaviours. The last two variables are related to the patient *per se* (Bayne, 2007).

Self-adhesive resin cements were firstly launched to satisfy clinician’s demands for simplification of luting procedures. These cements are directly applied on the restoration that is then seated in place and no treatments are necessary.

This thesis contains a study about several different aspects related to the bonding performances of self-adhesive resin cements used to lute coronal restorations on dentin, the influence of the hydration state of dentin and the retentive strength of simplified cements to fiber posts. The influence of fiber post surface treatments on the bond strength of self-adhesive cements was also taken into consideration.

The materials used for luting indirect restorations are associated to different adhesive characteristics and bonding performances. When using cements relying on multi-step bonding systems, limited adhesive penetration into demineralized dentin has led to post-operative sensitivity and decreased shelf-life of the restoration (Walshaw and McComb, 1996). A complete resin penetration into the depth of the acid-etched dental substrate is necessary to ensure long-lasting restorations (Toledano *et al*, 2004). Scanning electron microscope (SEM) is commonly used to observe the material/dentin interfacial characteristics. Good marginal adaptation of self-adhesive cements to dentin was observed in SEM evaluations,
although no hybrid layer nor resin tag formations were detected (De Munck et al, 2004) (Al-Assaf et al, 2007) (Behr et al, 2004). The first study was conducted to evaluate the degree of dentin demineralization, the depth of resin penetration and the quality of hybrid layer of different self-adhesive resin cements. A total-etch cement and a resin cement based on a self-etch system were used as controls. Two combined microscopic analysis were used: the Masson’s trichrome-staining technique for optical microscopy and the scanning electron microscopy.

Vital dentin is an heterogeneous substrate characterized by a water fluid flow through dentinal tubules (Marshall et al, 1997). The influence of dentin perfusion was previously investigated on self-etch or one bottle adhesive systems, showing detrimental effects during the initial setting phase of these materials (Sauro et al, 2007) (Hosaka et al, 2007). The study was conducted to evaluate the effect of an in vitro simulation of dentinal pulpal pressure on the bonding performances of different self-adhesive resin cements. For this purpose, a microtensile bond strength test and a scanning electron microscopy analysis were performed.

Due to the limited cement/dentin interactions, the influence of pre-etching steps on the bond strengths of the one-step cements to dentin was previously investigated. It was noteworthy how dentin conditioning with 35% phosphoric acid was detrimental (De Munck et al, 2004) (Hikita et al, 2007). Less aggressive acidic solutions could be proposed to partially remove the smear layer and improve the bond strengths of simplified cements to dentin. A microtensile bond strength test in combination with the Masson’s staining trichrome technique were used for this purpose.

Self-adhesive resin cements are alternative materials for fiber post luting. The effective sealing ability of self-adhesive as fiber post luting
agents is still a matter of concern (Zicari et al, 2008) (Simonetti et al, 2008). RelyX Unicem was undoubtedly the most investigated material while little information is present regarding the bonding performances of others differently branded auto-adhesive cements. Further studies of this thesis have focused on the bond strength and resistance to thermal stresses of different self-adhesive cements. In the attempt to improve the quality of the cement/fiber post bonds, several chemo/mechanical post surface treatments have been proposed. The topographic characteristics after the different conditioning approaches have been studied using a confocal microscopy and an atomic force microscopy (AFM). The self-adhesive cement/pre-treated fiber post combinations would result in improved adhesion and simplified techniques. A further object was to evaluate the effects of post surface treatments on the retentive strength of selected self-adhesive cements. Push-out bond strength test and scanning electron microscopic analysis were performed for this purpose.

1.2 Introducción general

El éxito clínico de una restauración indirecta está, en parte, relacionado con el material y la técnica de cementado utilizada para crear una unión entre la restauración y el sustrato dental (Hickel and Manhart, 2001). Entre los factores responsables de una posible reducción de su integridad, se considera una inadecuada adaptación marginal del cemento a nivel de las interfases adhesivas y una disminución de la retención de la restauración (Mijör and Gordan, 2002) (Mijör et al, 2002).

El mecanismo de adhesión se considera eficaz cuando se realiza una íntima relación entre cemento y dentina. La investigación en el área de los
El éxito clínico de una restauración indirecta se ve influenciado por cinco factores principales: el operador, el diseño de la restauración, el material, las condiciones intra-órales y la tipología del paciente (Bayne, 2007). Los primeros dos factores están directamente relacionados con la habilidad del odontólogo. Con respecto a la elección del material, el
conocimiento de todas sus características, incluyendo las propiedades física/químicas y clínicas se hace imprescindible. Los dos últimos factores están relacionados con el paciente (Bayne, 2007).

Los cementos auto-adhesivos han sido recientemente introducidos en el mercado dental para satisfacer las requestas de los odontólogos de simplificación de las técnicas de cementado. Estos materiales se aplican directamente en la superficie a adherir y no se prevé tratamiento alguno ni de las restauraciones ni de los sustratos dentales.

El objetivo principal que se plantea en esta thesis doctoral es de estudiar y determinar cual es el comportamiento adhesivo de los cementos simplificados recién introducidos, como se ven influenciados por el estado de hidratación de la dentina, y la capacidad retentiva que poseen como material de cementación de los postes de fibra, incluso tras el tratamiento superficial de los postes de fibra a base de resina epóxica.

Los materials utilizados para el cementado de las restauraciones indirectas mostraron diferentes características adhesivas y diferentes comportamientos. Una limitada penetración del adhesivo en el sustrato dental fue observado cuando se utilizaron cementos de pasos multiple. Esta situación provocó una sensibilidad postoperatoria y influenció negativamente el éxito de la restauración (Walshaw and McComb, 1996). La completa penetración de la resina en el sustrato desmineralizado es imprescindible para asegurar una duradera restauración (Toledano et al, 2004). La metodología más utilizada para la individuación de las características de las interfases adhesivas fue la microscopía electronica de barrido (MEB). Cuando este tipo de microscopía fue utilizada para observar la interacción entre los cementos auto-adhesivos y la dentina, se notó una buena adaptación marginal del cemento al sustrato adhesivo pero

La dentina vital es un sustrato altamente eterogéneo que está caracterizado por un continuo movimiento de fluido a través de los tubulos dentinarios (Marshall et al, 1997). Precedentemente, la influencia de la presión pulpar fue investigada sobre adhesivos simplificados one bottle o self-etch, revelando efectos negativos en la fuerza de adhesión, en particular manera durante las primeras fases de endurecimiento de los materials (Sauro et al, 2007) (Hosaka et al, 2007). El objectivo del segundo estudio se fijó en evaluar el efecto de una presión pulpar simulada en la capacidad adhesiva de los cementos simplificados. Para ello, fue utilizado un test de microtensión en combinación a un análisis de miscoscopia electronica de barrido (MEB).

La limitada interacción entre los cementos resinosos auto-adhesivos y la dentina recubierta de barrillo dentinario ha llevado algunos investigadores a testar la influencia de soluciones ácidas con el objetivo de eliminar el barrillo dentinario y permitir un contacto directo entre cementos y dentina. A pesar de las espectativas, condicionar la dentina con 35% de ácido fosfórico se ha relevado ineficaz cuando se evaluó la capacidad adhesiva de RelyX Unicem (De Munck et al, 2004) (Hikita et al,
Diferentes técnicas de remoción del barrillo dentinario, a través de soluciones ácidas más débiles (EDTA y ácido poliacrílico), se han evaluado, utilizando el test de microtensión asociado a un análisis de la interfase adhesiva a través de la técnica tricromica de Masson para la microscopía óptica.

Los cementos resinosos de paso único representan una alternativa para el cementado de los postes de fibra atraendo la atención de investigadores y clínicos. A pesar del grande interés, se observó una limitada habilidad de adherir a la dentina radicular (Zicari et al, 2008) (Simonetti et al, 2008), no obstante RelyX Unicem ha sido el material perteneciente a la clase de auto-adhesivos más investigado, mientras que algunas dudas se quedan acerca de la capacidad de unión de los demás cementos auto-adhesivos presentes en el mercado. Los siguientes estudios de esta tesis doctoral se focalizaron en evaluar la capacidad retentiva de estos materiales para el cementado de postes de fibra y sus resistencia a los éstreses termicos. Para ello, el push-out test se utilizó para evaluar la fuerza de unión de diferentes cementos auto-adhesivos y el efecto del termociclado en la dicha capacidad retentiva. Con la intención de mejorar la unión a los postes de fibra, fueron propuestos diferentes tratamientos de las superficies de los postes. Las características topográficas fueron evaluadas antes y post tratamientos químico/meccánicos a través de un análisis con microscopía confocal y con microscopía a fuerza atómica (AFM). La combinación entre los cementos auto-adhesivos y postes de fibra tratados en superficie puede resultar en una técnica de cementado simplificada alcanzando una adhesión mejorada. Un ulterior objetivo de esta thesis fue de evaluar el efecto de determinados tratamientos de superficie que no demostraron de dañar las fibras de vidrio, en la fuerza
An overview of the luting materials available for the cementation of indirect restorations

Several products are available in the dental market for the cementation of indirect restorations such as single crowns, bridges, fiber posts and screws. The selection of the luting agent should be based on the specific clinical situation, the type of the restoration and the physical, biologic and handling properties of the luting material itself (Jivraj et al, 2006). However, it cannot be possible to indicate one single product to be universally recommended in multiple situations. An ideal luting material should provide an effective marginal seal, it should possesses good mechanical and physical properties, it should be insoluble in the oral fluid, it should set in a short period of time and it has to be esthetic.

According to their chemical composition, dental cements can be divided into five main classess: zinc-phosphate cements, polycarboxilate cements, glass-ionomer cements, hybrid cements (resin-modified glass-ionomer cements and compomer) and resin cements (Diaz-Arnold et al, 1999). Clinicians should be aware of each material’s characteristics, its advantages and disadvantages, its chemical compositions and mechanical properties as well as the substrate to be bonded and the type of material used for the restoration (i.e. ceramic, zirconia, composite) should also be taken into consideration.
Zinc-phosphate cements, polycarboxilate cements and glass-ionomer cements are basically water-based. They are characterized by simple cementing techniques, but they possess limited mechanical properties and high solubility into the oral fluids. In general, these cements can be used for the cementation of metal and/or metal-ceramic crowns, but are not recommended for luting resins and all-ceramic restorations.

Resin cements have improved physical and mechanical properties and higher bond strength is established with the dental substrates. However, they need of an adhesive system to achieve high bond strengths and this makes the cementation technique more difficult and too operator-related. Resin cements can be divided according to their polymerization mode into dual-cured (recommended for the cementation of inlays, onlays, crowns and fiber posts), self-cured (for inlays, onlays, crowns and fiber posts) and light-cured (recommended only for luting laminate veneers).

Another classification of resin cements is based on the number of bonding steps required. From their first introductions in the late 1955, adhesive systems and techniques underwent to structural modifications and differentiations (Buonocore, 1955). At the beginning, the cements did not require any previous adhesive application as they could establish retention with the substrate to be bonded. Lately, adhesive systems were selectively recommended for enamel or dentin only. To date, bonding agents are combined to be suitable for all tooth substrates, and the classification can be made according to the steps necessary to condition the dental substrates (Heymann and Bayne, 1993) (Van Meerbeek et al, 2003). According to the data available into the dental literature, simplifying luting procedures would not always be related to higher bond
strengths and nowadays dental research is moving to optimize the adhesion mechanism of these materials to dentin.

The bonding mechanism is based on the concept of tissue hybridization between the dental substrate and the material (Nakabayashi et al, 1982) (Nakabayashi et al, 1991). The smear layer created during prosthetic preparations, can be completely removed or partially dissolved/modified and therefore considered as an intermediate bonding substrate (Ayad et al, 2001) (Toledano et al, 1999). Once the dental substrate is treated, dentin results demineralized and collagen fibers exposed: this situation would promote resin diffusion through dentinal tubules favouring the formation of an hybrid layer (Nakabayashi et al, 1982) (Moszner et al, 2005).

Cements relying on etch-and-rinse systems (such as Variolink, Variolink II and Calibra) follow a three step bonding process, that is the acid etching (necessary to remove the smear layer and to demineralize the inter-tubular dentin and the enamel prisms), primer application (to improve the wettability and the superficial characteristics of the substrate) followed by the use of the bonding (Bayne, 2005). Dentin would result more susceptible to the external physical changes after the etching process: over-drying or over-wetting in this stage would affect the resin diffusion and increase the probability of adhesive failures (Pioch et al, 1992) (Sano et al, 1995) (Van Meerbeek et al, 1998).

In the late 1990s, acidic monomers were incorporated into the primer in order to assemble the etching and the primer agents in a single solution (i.e. Panavia 21, Panavia F, Panavia F 2.0). The acidic monomers are intended to modify the smear layer and consequently the inter-tubular dentin; lately, the bonding should infiltrate the collagen fibrils as to create

More recently, self-etch one bottle adhesive systems have been introduced (i.e. Adper prompt L Pop) (Moszner et al, 2005) (Nishiyama et al, 2006). Although the interest for their easy to handling and user friendless, their main disadvantages is represented by the chemical incompatibility existing between the tertiary amine of the simplified adhesives and the catalizador of the dual-cured or light-cure resin materials (Carvalho et al, 2005b) (Tay et al, 2003a). Moreover, the incorporation of hydrophilic monomers make these adhesives more susceptible to the hydration state of dentin, as water can proceed through dentinal tubules reaching the bonding interface resulting in an improper setting reaction that would affect the durability of the restoration (Musanje et al, 2003) (Sauro et al, 2007) (Hosaka et al, 2007). The bonds established between highly hydrophobic adhesives and dentin would deteriorate over time (Pashley et al, 2004) (Toledano et al, 2007). Considering the total-etch adhesive systems, an incomplete resin diffusion into the opened dentinal tubules was noticed (Eliades et al, 2001), showing areas not completely impregnated at the bottom of the hybrid layer (Pashley et al, 2004) (Hashimoto et al, 2002). This problem could ideally be overcome with the self-etch adhesives, as a simultaneous demineralization/infiltration is expected (Toledano et al, 2004) (Toledano et al, 2007). However, dentinal tubules were only superficially infiltrated by simplified adhesives (Santini and Miletic, 2008) (Carvalho et al, 2005a) (Tay et al, 2002).
1.4 Self-adhesive resin cements: composition and properties

Self-adhesive cements are the latest subgroup of resin cements introduced in the clinical practice. These cements include in a single product the ease of handling of conventional cements, the auto-adhesion and the fluoride release of glass-ionomer cements as well as the mechanical properties, dimensional stability and micro-mechanical retention of resin cements (Radovic et al, 2008). The less technique sensitivity is pivotal of these one-step cements: after mixing base and catalyst or after capsule activation, the cement is directly applied on the adhesive substrate, hence limiting the errors that can occur with the cement relying on multi-step systems (i.e. overwetting or overdrying the dental substrate or the chemical incompatibility between simplified adhesive and light or dual polimerizable resin cements) (Tay et al, 2003c) (Pfeifer et al, 2003). Self-adhesive cements simultaneously demineralize/infiltrate smear layer and consequently the underneath tooth substrate. Accordingly, smear layer represents an intermediate bonding substrate which can reduce post operative sensitivity. In general, the bonding mechanism of self-adhesive cements is based on a chemical interaction and micro-mechanical retention with the adhesive substrate (De Munck et al, 2004) (Yang et al, 2006) (Abo-Hamar et al, 2005). A chemical reaction is established between the multifunctional monomers with the phosphoric acid groups of the cement and the hidroxiapatite; together, the acidic monomers interact with the alkaline fillers of the cement complementing the chemical reaction. The water produced during the acid-base reaction is necessary to neutralize the acidic monomers thus favoring the hydrophilic behaviour of the material during the initial phase.
of the reaction, resulting in improved marginal adaptation and limiting the influence of the intrinsic wetness of human dentin. The water also acts as a buffer solution developing more hydrophobic characteristics during the secondary setting reaction (Radovic et al, 2008). The setting reaction takes place following the radical polymerization that can be induced by light exposure or through a self-cure modality (De Souza Costa et al, 2006). However, according to recent literature data, concerns exist regarding the efficacy of these simplified cements to set in a solely light cure mode: an increase in monomer conversion and superior mechanical properties have been reported after the material was dual-cured (both chemically and light activation) (Vrochari et al, 2009) (Kumbuloglu et al, 2004) (Pedreira et al, 2009).

A wide range of self-adhesive products have been launched in the market by different manufacturers, although RelyX Unicem (3M ESPE) has been the first to be introduced and undoubtedly the product more investigated. Although based on a similar auto-adhesive technology, these materials show differences in terms of application modality, working and setting time and chemical compositions that may differentiate their mechanical properties and bonding performances (Saskalauskaite et al, 2008) (Han et al, 2007). Limited in vivo studies are present in literature (Naumann et al, 2007) (Behr et al, 2008), and the knowledge of the bonding potential and mechanical properties of self-adhesive cements is mainly based on laboratory investigations. According to in vitro reports, a decreased bond strength is registered when self-adhesive cements are applied on enamel (De Munck et al, 2004) (Abo-Hamar et al, 2005) (Hikita et al, 2007). This should be taken into consideration when luting inlays or partial crowns in presence of a considerable amount of enamel,
such as for brackets cementation. Bond strength to enamel can be increased after etching the dental substrate with 35% phosphoric acid (De Munck et al, 2004) (Duarte et al, 2008) (Vicente et al, 2006). On the other hand, the behavior of self-adhesive cements on coronal dentin remain uncertain and no hybrid layer no real resin tag formations could be observed at the dentin interface (Goracci et al, 2006) (De Munck et al, 2004) (Yang et al, 2006) (Al-Assaf et al, 2007). Although the presence of smear layer as adhesive substrate would represent a barrier against the direct interaction between cement and the underneath dentin, it was noteworthy how dentin conditioning with 35% phosphoric acid did not procure any bond strength improvement (De Munck et al, 2004). The high viscosity of the self-etch luting agents can limits their diffusion into opened dentinal tubules. Accordingly, Goracci et al. found that applying a sustained seating pressure during the setting reaction of the material would encourage the resin penetration into dentinal tubules, and increased bond strength values were registered than when the seating pressure was not present (Goracci et al, 2006). Although radicular dentin presents structural differences with coronal dentin (Ferrari et al, 2000), contemporary studies reported limited interaction between self-adhesive cements and root canal dentin which resulted poorly demineralized and no hybrid layer was formed (Zicari et al, 2008)(Goracci et al, 2004)(Simonetti et al, 2008)(Bitter et al, 2009).

From a mechanical point of view, self-adhesive cements showed good resistance to compression (Piwowarczy and Lauer, 2003) (Kumbuloglu et al, 2004) and microhardness even after immersion in water after 3 months (Pedreira et al, 2009). The film thickness recorded for some self-adhesive cements has been considered valid for the

1.5 Self-adhesive resin cements vs cements based on multi-step systems

Contrasting results were evidenced when the bonding performances of self-adhesive composite cements were compared to those of conventional and/or resin cements that rely on multi-step adhesive systems. When used for the cementation of ceramic crowns, the marginal adaptation of RelyX Unicem was comparable to that of a self-etch system, a total-etch cement and a compomer, revealing a lower dye penetration, notwithstanding the scarce hybrid layer and resin tags formations (Behr et al, 2004). The interfacial characteristics of the self-adhesive cement/dentin interfaces were inferior when compared to those of well-tried systems (Al-Assaf et al, 2007) (De Munck et al, 2004), although the bond strength of self-adhesive cements to dentin would be considered more crucial (Bitter et al, 2008).

Ageing simulations were claimed to be important sources for the understanding of biomaterials properties. According to the data present in literature, total-etch cements showed the higher resistance to thermo/mechanical stresses than self-etch systems and self-adhesive
composite cements. Conversely, results of other investigations revealed bond strengths of self-adhesive cements comparable or higher to conventional cements after thermocycling.

Compared to conventional resin cements, Max-Cem revealed to be highly dependent to mixing errors, that would jeopardize the final adhesion process (Behr et al, 2008). However, the bonding performances of a material seemed related to the material chemical composition itself. Comparisons are usually difficult to be done as the same self-adhesive cements showed differences in their chemical composition and different bonding behaviour (Han et al, 2007) (Skalauskaite et al, 2008). A more complete classification of self-adhesive cements is warranted in order to determine the class and to deeply understand their adhesion mechanism.

1.6 Dentin characteristics as adhesive substrate and the influence of the hydration state of dentin on the bonding performance of adhesive systems and cements

The dentin is an host substrate to be bonded, due to the structural differences existing between the different regions and the physiological problems. Dentin surface treatments and new materials are continuously experimented in order to improve the adhesive strength to this variable and heterogeneous substrate. The knowledge of the main components and mechanical properties of dentin is imperative to face to this substrate and develop dental biomaterials able to substitute the lost tissue and to physiologically integrate with the rest of the dental tissue (i.e. enamel, cementum). These procedures are intended to limit the effects of micro-
and nano-infiltration which can determine a premature failure of the restoration (Marshall et al, 1993a).

Different types of dentin can be distinguished: primary, secondary, reparative, tertiary, transparent, carried, demineralized, remineralized or hypermineralized (Marshall, 1997). In fact, dentin is an highly complex and heterogeneous substrate dissimilar from the others dental tissues. It is formed of its 50% of vol of mineral components, 30% of organic matrix (in particular, type I collagen fibers) and 20% of fluids (Ten Cate, 1994). Dentin is an elastic tissue for the above enamel, while it has to protect the underlying pulp tissue. Differences exist between the superficial and deep dentin in terms of morphology and chemical constituents (i.e. number of dentinal tubules and their dimensions, the peri-tubular dentin and the areas occupy by the inter-tubular dentin) (Urabe et al, 2000) (Toledano et al, 1999) (Pashley, 1989). Originating from the pulpal tissue, the dentinal tubules go along a radial orientation. Both tubules density and their diameter undergo to a reduction follow the proximity of the superficial dentin. The orientation of dentinal tubules can influence the mechanical properties of the substrate (i.e. being situated perpendicularly would result in a reduced strength to the occlusal forces) (Arola and Reprogel, 2006). The mineral component is found among the inter-tubular and peri-tubular dentin (Marshall et al, 1993a). The dentin hydroxiapatite crystals show differences when compared to those of the enamel, as they are smaller, contain an inferior percentage of calcium and a reduced amount of carbonate (4-5%) (Posner and Tannenbaum, 1984) (Jones and Boyde, 1984). The hydroxiapatite crystal anchor to the dentinal tubules (Hayashi, 1992) and this union play an important role in establishing the mechanical

Different from the enamel, vital dentin is known to be highly hydrated, as it is characterized from a continuous water fluid-flow through dentinal tubules (Sauro et al, 2007) (Hosaka et al, 2007a) (Elgalaid et al, 2004) (Ciucchi et al, 1995) (Tay et al, 2005) that originally are occupied by the odontoblastic processes. The water movement is high at the deep level while diminishes close to the superficial dentin (Pashley et al, 1987) (Fogel et al, 1988). These differences are related to the variety of diameter existing between the dentinal tubules of the deep (2.5 µm) and those of the superficial dentin (0.8 µm) (Pashley et al, 1987) (Pashley, 1991). Moreover, differences exist between the number of tubules that can be found in the two areas: 22% at the deep dentin against the 1% of superficial dentin (Pashley et al, 1987). Some researchers investigated the water fluid movement present in human being, and considered appropriate the application of a pulpal pressure of 15-20 mmHg that more likely could reproduce the clinical situation into the laboratory (Ciucchi et al, 1995) (Vongsavan and Matthews, 1992). From an adhesive point of view, the hydration state of dentin can influence the bonding mechanism of selected resinous materials and limit the durability of the restoration. In particular, the presence of water can negatively affects those materials containing an high percentage of hydrophilic monomers (Sauro et al, 2007) (Hosaka et al, 2007b) (Tay and Pashley, 2003), especially during their initial setting process (Hiraishi et al, 2008).

In general, it is possible to classify the adhesion mechanism to dentin into two types: chemical and mechanical-retentive. The chemical adhesion is realized between the restorative material and the mineral component of
dentin, the collagen fibers or its precipitates that are formed after the acidic pre-treatment (Bowen and Marjenhoff, 1992). The micro-retention is realized through resin tags formations into the opened dentinal tubules and the modification of the inter-tubular components (Nakabayashi, 1992) in a process that should be finalized by the complete polymerization of the material (Özok et al, 2004) (Erhardt et al, 2008).

Dentin demineralization causes structural modifications that determine an increase of dentinal permeability once the smear layer is removed and the dentinal tubules are opened (Musanje and Darvell, 2003) (Balooch et al, 2008). Nowadays, several chemical solutions are intended to remove the smear layer and to create a direct contact between the material and the dental substrate (Betolotti, 1992) (Lopes et al, 2003) (Garberoglio and Brännström, 1976). Acid etching of dentin permits to remove the peri-tubular dentin, to demineralize the intra-tubular dentin and to create a rough surface that can establish retention with the restorative material. Several investigations dealt with the morphological changes caused by chemical agents, that were based on different microscopic methodologies (Van Meerbeek et al, 1992) (Urabe et al, 2000) (Balooch et al, 2008) (Lopes et al, 2003) (Van Meerbeek et al, 1993) (Marshall et al, 1993b).

1.7 The use of fiber posts in dentistry

Teeth requiring an endodontic treatment often present an excessive loss of mineralized tissue, and thus, in comparison to sound teeth, they are weaker (Trope et al, 1985) (Morgano and Milot, 1993). This consideration become of great importance in terms of the subsequent prosthetic
restoration (Ferrari and Scotti, 2002) (Trabert et al, 1984) (Sorensen et al, 1984). Fiber posts are increasingly assuming importance for the restoration of endodontically treated teeth with massive coronal distractions. Different post systems have been proposed over the years, from the early cast metallic posts to the pre-fabricated metallic posts or the more recently introduced fiber posts. Fiber reinforced composite posts (FRC) have been introduced at the beginning of the 90s, as an alternative to conventional post systems (Duret et al, 1990)(Trabert et al, 1978). In general, fiber posts consist of unidirectional, pre-tensed fibers, whose diameter and density strictly influence the quality of the material and its adhesion process, embedded in a resinous matrix (in particular epoxy-based). In particular, differently branded fiber posts are characterized by differences in their quality, mechanical properties and clinical behaviour (Grandini et al, 2005) (Ferrari et al, 2007) (Goracci et al, 2008). Fiber posts present many advantages, that make their choice in the restoration of endodontically treated teeth secure and more reliable when compared to metallic posts. The biomechanical properties of fiber posts have been reported to be closer to that of dentin, diminishing the incidence of root fracture that would represent the loss of the tooth (Duret et al, 1990) (Asmussen et al, 1999) (Malferrari et al, 2004) (Sorrentino et al, 2006) as fiber posts allow for a better stress distribution compared to cast metal posts (Ferrari et al, 2000) (Akkayan and Gulmetz 2002) (Heydecke and Peters, 2002). Their esthetic properties could satisfy the growing patient’s esthetic demands (Martinez-Insua et al, 1998). Different factors may influence the clinical outcome of a post-restored tooth as post design (Nissan et al, 2001), length (Okamoto et al, 2008), diameter and root canal configuration (Morgano et al, 1996) (Innella et al, 2005). Fiber posts are
passively retained into root canal. Accordingly, their dislocation resistance is mostly ascribed to the luting agent and technique adopted for the cementation (Bitter et al, 2006) (D’Arcangelo et al, 2008). The fiber post/resin cement combinations have been preferred to conventional luting agents as they can additionally strengthen the root, and uniformly distribute stress along the entire root (Dietschi et al, 2008) (Rosenstiel et al, 1998) (Vichi et al, 2002). The fiber post/resin cement combinations would allow for an easily “debonding” mode of failure, as it can be solved by solely repeating the adhesive procedure without compromising the dental structure (Cagidiaco et al, 2007) (Mannocci et al, 2005).

The choice of the proper luting agent should be based on different considerations, such as the clinical situation, personal preferences and the quality of the material. No differences were found in terms of bond strengths between different resin cements, and Magni et al. recommended the use of material that would function both as cement and core material (Magni et al, 2006). This would simplify luting and restorative procedures, diminishing the number of materials and reducing the interfaces thus limiting the critical areas prone to be stressed (Magni et al, 2006) (Mazzitelli et al, 2006). Simplifying luting procedures would be convenient in terms of time-saving and less-incidence of operator variability. One-step, self-adhesive resin cements, revealed bond strength similar to that of well-tried systems, offering new perspectives for the cementation of fiber posts (Radovic et al, 2008).

The methodology most employed to test the dislocation resistance of fiber post into root canal is the push-out test. Stresses are created parallel to the adhesive interfaces (cement/post and cement/dentin) hence better simulating the clinical situation (Goracci et al, 2007). Microtensile test
was also used, but it showed some limitations that made the test less appropriate for the calculation of the bond strength into root canal (Goracci et al, 2007) (Goracci et al, 2005). Durability test are necessary to explore the dental biomaterial behaviour in laboratory in order to be then clinically recommendable. Thermocycling, cyclic loading and or chewing simulators are all methods accepted for testing the materials properties and their long-lasting bonding performances (De Munck et al, 2004).

1.8 Post surface treatments for improving the cement/post bond

Fiber post/cement/dentin complex should form a “monoblock” to ensure long-lasting restorations (Schwartz and Robbins, 2004). However, the different properties of the bonding substrates involved make the monoblock philosophy hard to be achieved (Tay and Pashley, 2007) (Zicari et al, 2008).

Post surface pre-treatment have been proposed to improve the retention of the restorative resinous materials to fiber posts (Magni et al, 2007) (Monticelli et al, 2006). According to the nature of the conditioning process, three main classes can be distinguished: chemical (silane and/or adhesives application), mechanical (sandblasting or acid etching) and chemical/mechanical (the combined used of the previous mentioned mechanisms) (Monticelli et al, 2008a) (Monticelli et al, 2008b). The adhesion between resin cements and epoxy-resin based fiber posts can only be realized through the methacrylic group of the cement and the glass fibers of the posts. As the epoxy matrix completely envelop the glass fibers, the chemical interaction would result limited. The goal of post treatments is that of eliminating the superficial incompatible epoxy matrix
and to expose the underlying glass fibers that can be then activated by
silanization. Most of these surface treatments create rough surfaces that
should enhance micro-mechanical retentions with the restorative materials
al, 2006) (Radovic et al, 2007). Silane coupling agents are undoubtedly
the most investigated treatments solutions for fiber post conditionings
(Perdigao et al, 2006) (Goracci et al, 2005). Silane solutions would
enhance the superficial wettability and enable for a stronger chemical
interaction between the two incompatible materials (Lassilla et al, 2004).
As silanes can only promote the adhesion between methacrylic groups and
glass fibers, their use would be improved after pre-treating fiber posts with
additional chemical and/or mechanical procedures (Monticelli et al,
2008b). In the attempt to improve the chemical adhesion between cements
and fiber posts, the combined use of silane/primer have been proposed by
some manufacturers (Monticelli et al, 2008b) (Okuda et al, 2002) (Ferrari
et al, 2002). The rationale of treating fiber post have antecedents on the
procedures previously proposed for dental substrates (Nakabayashi et al,
1991) (Buonocore, 1955). The superficial treatments should create
additional anchoring sites to be bonded and increase the mechanical
retention of resinous materials. Clinicians should take care of the nature of
the treatments, as some of them resulted too aggressive for the integrity of
the fibers, such as, hydrofluoric acid (Vano et al, 2006) and sandblasting
executed with alumina particles with excessive dimensions (Valandro et
al, 2006) (Sahafi et al, 2004). Nowadays, the dental market offers fiber
post already treated with a silicate/silane layer that would ulteriorly
simplify luting procedures (i.e. DT Light SL Post, VDW) with the
convenience of reliable bonds.
1.9 An overview of self-adhesive resin cements and their clinical application

According to literature data, RelyX Unicem was the most investigated self-adhesive material. Seventy-eight studies reported on the bonding behaviour and mechanical properties of RelyX Unicem, while 13 investigations dealt with Max-Cem, 7 were related to Multilink Sprint and 5 treated on the bonding ability of G-Cem. Only 2 articles focused on the bonding performance of Breeze, 5 were concentrated on Bis-Cem, 1 on Smart-Cem and 1 on I-Cem. Only one study compared the bonding potential of an experimental self-adhesive cements (Kuraray) to those of other auto-adhesive materials (Cantoro et al, 2009).

In the most part of the cases, control materials were represented by well-established total-etch systems (i.e. Variolink and/or Calibra) or self-etch cements (i.e. Panavia). Other comparisons were made with glass-ionomer cements (in particular Fujiy Plus) or compomers (i.e. Dyract).

Only one literature review is available regarding the bonding effectiveness and mechanism of self-adhesive cements (Radovic et al, 2007), although new reports were then published dealing with new materials. Due to the differences in their chemical compositions (Han et al, 2007), comparisons between the cited studies is not always possible, and dentist should refere to the material itself more than to the class they belong to. Only 2 clinical studies were conducted to explore the in vivo performance of a self-adhesive material. One study analyzed the clinical behaviour of RelyX Unicem used as fiber post luting agent (Naumann et al, 2007), while the second focused on the bonding ability of RelyX
imperative. The cement/dentin bond is more complicated, due to the heterogeneity of the dental substrate. According to the *in vitro* studies, the interfacial characteristics between self-adhesive cements and dentin are formed by scarcely distributed and sporadic resin tag formations, with no evident hybrid layer both in coronal and in root dentin (Yang *et al*, 2006) (Al-Assaf *et al*, 2007). The bonding process is though to be based on a chemical interaction between the acidic monomers of the cement and the minerals of the hydroxiapatite. A second reaction is established between the acidic monomers and the basic fillers of the cement that generate water necessary to balance the acid-base chemical reaction. However, pre-treating dentin with 35% phosphoric acid did not result in improved bond strengths of RelyX Unicem. Thus, acid etching dentin was not recommended during clinical procedures (De Munck *et al*, 2004) (Hikita *et al*, 2007).

Some authors tested the ability of self-adhesive cements to lute CAD/CAM ceramic restorations. While self-adhesive cements would show acceptable marginal adaptation in terms of continuous margins (Mormann *et al*, 2008) (Good *et al*, 2009), Nuria *et al* recorded the inferior microtensile bond strength values of RelyX Unicem when compared to Panavia F (Nuria *et al*, 2006). No information can be found regarding the other self-adhesive cements. Bonding to zirconia ceramic is hampered by the chemical incompatibility of the restorative material with most of the cements available (Casucci *et al*, 2009). Although different zirconia surface treatments are continuously proposed, Blatz *et al* found that of the self-adhesive cements available, those containing adhesive monomers such as MDP/4-META (i.e. G-Cem) would be adequate to obtain reliable bonds to zirconia frameworks (Blatz *et al*, 2009). Senyilmaz *et al*, found
that Max-Cem recorded the inferior shear bond strength when used on pre-treated zirconia, concluding that attention should be paid on the chemical composition of the material adopted (Senyilmaz et al, 2007). Dual curing self-adhesive cements resulted in superior push-out values when compared to other resin cements for the cementation of fiber posts (Toman et al, 2009) (Elsayed et al, 2009).
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Chapter 2

2.1 The interaction between self-adhesive cements and the dentin substrate

A reliable bond is achieved when an intimate contact is established between the dental substrate and the material (Moszner et al, 2005). The dental substrate should be ideally treated to remove the smear layer created during prosthetic preparations (Nakabayashi et al, 1992) (Nakabayashi et al, 1991). The material should then infiltrate the opened spaces, fulfill the voids and polymerized in a short period of time (Toledano et al, 1999). The hybridization of the dental substrate was supposed to represent the basis of the bonding mechanism. Several cements are available on the market for the cementation of indirect restorations. Resin cements can offer improved mechanical and physical properties and advanced adhesion mechanisms, thanks to the chemical interaction they can have with the dental substrates. A classification was made according to the adhesive systems used (Van Meerbeek et al, 2003). A discrepancy between the degree of dentin demineralization and depth of resin penetration has been observed with the cements that utilize the total-etch systems: the voids left at the bottom of the hybrid layer represent areas prone to premature degradation influencing the long-lasting outcome of the bonds (Wang and Spencer, 2005) (Spencer et al, 2004). The simplified version of these bonding agents is represented by the one-bottle adhesives, in which the acid, the primer and the bonding are accomunate in a single product. The goal was that of simultaneously demineralize and infiltrate the adhesive interfaces ideally improving the resin/dentin interdiffusion zone (Tay and Pashley, 2002). However, the technique simplification is not always accompanied with superior bond strength
values: although the acidic monomers contained into the solution, they were not strong enough to demineralize the smear layer and subsequently the dental substrate (Oliveira et al, 2003) (Breschi et al, 2008). The exposure of weak dentin/adhesive interfaces to the oral cavity would result in marginal discoloration, poor marginal adaptation and subsequent loss of retention of the restoration (Mijor and Gordan, 2002) (Mijor et al, 2002) (Breschi et al, 2008).

Self-adhesive cements introduced a simple luting approach and the smear layer is taken as an intermediate bonding substrate. Literature data report that self-adhesive resin cements only superficially interact with dentin: interfacial evaluations revealed sporadic and scarcely distributed resin tags with no hybrid layer formation (Al-Assaf et al, 2007) (Abo-Amar et al, 2005) (De Munck et al, 2004) (Blatz et al, 2009). Many investigators focused their attention on microscopic evaluations using scanning electron microscopy (SEM) and/or transmission electron microscopy (TEM) to evaluate the adhesive interfaces (Al-Assaf et al, 2007) (De Munck et al, 2004) (Behr et al, 2009). In particular, the only information still present is related to the bonding ability of RelyX Unicem, while a clear knowledge of the bonding efficacy and quality of the bond of others self-adhesive cements is necessary.

The following chapter deals with the observation of adhesive interfaces established between three classes of resin cements: total-etch, self-etch and four self-adhesive resin cements. In order to evaluate the effect of the pre-treatment regime on the demineralization/penetration of resin into dentin, a morphological analysis was performed using the scanning electron microscopy (SEM) and the Masson’s staining trichrome technique for light microscopy.
References


2.2 Limited decalcification/diffusion of self-adhesive cements into dentin.


Introduction

Resin-based dental luting agents, which are routinely used for luting gold, composite crowns and all-ceramic restorations have traditionally required a separate etching step to allow subsequent adhesive infiltration (Diaz-Arnold et al, 1999). Incomplete adhesive diffusion throughout the demineralized dentin has been reported for conventional dentin bonding agents (Spencer and Swafford, 1999). The discrepancy between etching depth and adhesive penetration led to a large area of exposed collagen at the interface between the adhesive and prepared dentin surfaces. If this discrepancy occurs with luting agents that require a separate etching step, it is conceivable that there may be post-operative sensitivity as a result of the exposed collagen (Walshaw and Mc Comb, 1996).

To overcome some of the limitations associated with dentin etching, resin cements that include self-etching primers have been proposed (Watanabe et al, 1994). This approach has reintroduced the concept of employing smear layer as bonding substrate, but with novel formulations that should etch beyond the smear layer into the underlying dentin (Reis et al, 2005).

A growing interest has been focused on the use of self-adhesive cements. These systems were designed with the purpose of combining the favorable characteristics of different cements in a single product. Trying to satisfy demands for simplification of luting procedures and supposedly

Self-adhesive cements do not require any tooth surface pretreatment and their application is accomplished through a single clinical step, similarly to more conventional zinc-phosphate and polycarboxylate cements (Diaz-Arnold et al, 1999).

Based on recent in vitro data, the behavior of the most investigated self-adhesive cement to dentin (RelyX Unicem) and different restorative materials should not differ from multi-step resinous cements (Fabianelli et al, 2005) (Bitter et al, 2006) (Piwowarczyk et al, 2004). However, concerns emerged regarding the bonding potential of these materials to enamel and dentin (Behr et al, 2004) (Gerth et al, 2006). Although the basic adhesion mechanism appears similar for all self-adhesive cements, these materials are still relatively new and detailed information on their composition and adhesive properties is limited.

The purpose of this study was to qualitatively compare the dentin/cement interfacial characteristics of six current commercial adhesive cements that differ as a function of pre-treatment regimen. Scanning electron microscopy (SEM) and a staining technique for optical microscopy, that specifically identifies depth of decalcification/infiltration or exposed collagen at the dentin/cement interface, were employed. This study tested the hypothesis that resin cement diffusion into the prepared dentin surfaces would differ as a function of the pre-treatment regimen.
Materials and Methods

Thirty extracted human third molars, stored at 4°C in 0.1% wt/vol Chloramine T solution were decoronated (Isomet 4000, Buehler, Lake Bluff, IL) obtaining mid-coronal dentin surfaces that were grinded with 600-grit wet silicon carbide papers creating a uniform thin smear layer. The use of human specimens was obtained following a protocol that was approved by the institutional review board (IRB) and with the informed consent of the donors.

Composite cylinders were made by layering 2 mm-thick increments of a micro-filled hybrid composite (Gradia Direct Anterior, GC Corp., Tokyo, Japan, shade A3) in a split aluminium mold (8 mm diameter/4 mm height). Each increment was light-cured for 40s (VIP, Bisco Inc., Schaumburg, IL, USA, output: 600 mW/cm²). The specimen was removed from the mold, additionally light-cured from five aspects for 40s each on the portions previously in contact with the metallic surface of the mold.

The prepared dentin surfaces (n=5 each group) were luted with: 1. Calibra dual-cured etch-and-rinse cement (Dentsply DeTrey GmbH, Konstanz, Germany); 2. Panavia F 2.0 dual-cured self-etch cement (Kuraray Co. Ltd, Osaka, Japan); 3. Multilink Sprint (Ivoclar-Vivadent, Schaan, Liechtenstein); 4. RelyX Unicem (3M ESPE, St. Paul, MN, USA); 5. G-Cem (GC Corporation, Tokyo, Japan) 6. Bis-Cem (Bisco, Schaumburg IL, USA) dual-cured self-adhesive cements.

pH measurements were performed for all tested luting agents. After mixing, they were dispensed on pH acid indicator strips with narrow ranges (0.0-1.8; 1.8-3.8; 3.8-5.5; Panreac Química, Barcelona, Spain). The
composition, pH and application mode of the tested resin cement systems are reported in Table 1.

The luting procedure of composite cylinders on dentinal substrates was performed exerting a constant pressure of 40 g/mm² during the initial 5-min self-curing period (Goracci et al, 2006).

### Table 1. Chemical composition and application mode of the resin cements tested in the study.

<table>
<thead>
<tr>
<th>Material</th>
<th>Composition</th>
<th>Application</th>
</tr>
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<tbody>
<tr>
<td>Calibra (H₃PO₄) pH= 0.4</td>
<td><strong>Prime&amp;Bond NT</strong>: acetone, Di- and Tri- methacrylate resins, Urethane Dimethacrylate, PENTA, Nanofiller- amorphous silicone dioxide, photoinitiators, stabilizers, cetylamine hydrofluoride. <strong>Calibra</strong>: Barium boron fluoroalumino silicate glass, bisphenol A, diglycidylmethacrylate, polimerizable dimethacrylate resin, hydrophobic amorphous fumed silica, titanium dioxide, dicamphoroquinone. <strong>Catalyst</strong>: barium boron fluoroalumino silicate glass, bisphenol A diglycidylmethacrylate, polimerizable dimethacrylate resin, hydrophobic amorphous fumed silica, titanium dioxide, benzoyl peroxide.</td>
<td>Apply etchant (30s). Water rinse (20s). Air-drying. Apply adhesive in a single coat. Gently air-drying after 5s. Light-cure for 20s. Mix base and catalyst (1:1). Apply and self-cure (5 min). Light-cure (40s).</td>
</tr>
<tr>
<td>Panavia F 2.0 pH: 2.4</td>
<td><strong>ED Primer II</strong>: Liquid A: 10- methacryloyloxydecyl dihydrogenphosphate; 2-hydroxyethyl methacrylate; N,N-diethanol-p-toluidine; N-methacryloyl 5-aminosalicylic acid; water. Liquid B: N,N-diethanol-p-toluidine; Sodium benzenesulphinate; N-methacryloyl 5-aminosalicylic acid; water. <strong>Panavia F</strong>: Paste A: Silanated barium glass; colloidal silica; Bisphenol A Polyethoxy Dimethacrylate; 10- methacryloyloxydecyl dihydrogenphosphate; Hydrophilic dimethacrylate; Hydrophobic dimethacrylate; benzoi peroxide; dl-camphoroquinone. Paste B: Silanated barium glass; Silanated titanium oxide; Sodium fluoride colloidal silica; Bisphenol A Polyethoxy Dimethacrylate; Hydrophilic dimethacrylate; Hydrophobic dimethacrylate; N,N-diethanol-p-toluidine; Sodium 2,4,6-Triisopropyl benzene sulfinate.</td>
<td>Mix ED Primer A+B (1:1). Apply on the tooth. Gently air-blow after 30s. Mix Paste A+B (1:1) for 20s. Apply and self-cure (5 min) Light-cure (40s)</td>
</tr>
<tr>
<td>RelyX Unicem pH: 2.1</td>
<td>Powder: Glass fillers, silica, calcium hydroxide, self-curing initiators, pigments, light-curing initiators. <strong>Liquid</strong>: methacrylated phosphoric esters, dimethacrylates, acetate, stabilizers, self-curing initiators, light-curing initiators.</td>
<td>Mix cement. Apply, self-cure (5 min) and light-cure (40s)</td>
</tr>
<tr>
<td>Multilink Sprint pH: 4.2</td>
<td>Dimethacrylates; adhesive monomer; Fillers; initiators / stabilizers.</td>
<td>Mix cement. Apply, self-cure (5 min) and light-cure (40s)</td>
</tr>
<tr>
<td>G-Cem pH: 2.7</td>
<td>UDMA; phosphoric acid ester monomer; 4-META; water; dimethacrylates; silica powder; initiators/stabilizers; fluoro-amino-silicate glass.</td>
<td>Mix cement. Apply, self-cure (5 min) and light-cure (40s)</td>
</tr>
<tr>
<td>Bis-Cem pH: 2.1</td>
<td>Bis (Hydroxyethyl methacrylate) phosphate (Base); Tetraethylene glycol dimethacrylate; dental glass.</td>
<td>Mix cement. Apply, self-cure (5 min) and light-cure (40s)</td>
</tr>
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</table>
**Trichromic stain and microscopic observation**

After 24h storage (37°C at 100% humidity), three samples from each group were sectioned perpendicularly to the bonded surface into 1-mm thick slabs using a water-cooled, low-speed diamond saw (Isomet 4000). A total of 12 sections were analyzed for each dentin treatment. Slabs were glued on methacrylate supports with photo-curing adhesive (Technovit 7200 VLC, Kulzer, Norderstedt, Germany) and grinded with an Exakt polishing machine (EXAKT Technologies, Inc., Oklahoma City, OK, USA) using SiC abrasive wet papers (800; 1200; 2500; 4000 grit) until getting a thickness of 5-6µm. Differential staining was accomplished with Masson’s trichrome, a classic bone stain (Erhardt *et al.*, 2008). After coverslipping with mounting media, they were examined using a light microscope (BH2, Olympus, Tokyo, Japan) at 100x magnification.

**Scanning Electron Microscopy**

Two additional specimens for each group were prepared for SEM evaluation. Samples were sectioned perpendicularly to the bonded surface (Isomet 4000). Each section was polished with wet abrasive SiC papers, gently decalcified (37% phosphoric acid/10s) and deproteinized (2% NaOCl solution/1 min) ultrasonicated in 96% ethanol for 2 min and air-dried. Samples were mounted in stubs, sputter-coated with gold (Polaron Range SC7620; Quorum Technology, Newhaven, UK) and observed under a scanning electron microscope (SEM; JSM-6060 LV, JEOL, Tokyo, Japan) at different magnifications to evaluate for resin tags and hybrid layer formation. Impressions of the restored teeth and positive
impression replicas were fabricated (Chersoni et al, 2004) and observed by SEM to control for artefact formation.

**Results**

According to the Masson’s trichrome-staining technique, the mineralized dentin stains green, whereas the resin cement is clear with filler particles. The staining reaction of proteins is non-specific and some non-collagenous proteins may have been marked. Type I collagen represents the 90% of dentin organic matrix. Thus, it is likely that the protein staining (red) resulted from dentin collagen unprotected by mineral and/or resin.

Using the conventional etch & rinse luting agent (Calibra) a distinct red zone of denuded collagen at the basis of the bonded interface was observed. Tubules were opened (Fig. 1A), and hybrid layer with resin tags formation were identified by SEM (Fig. 2 A). At the dentin/cement interface of teeth luted with the self-etching cement (Panavia) a narrow purple line representing mild collagen demineralization is detectable at the intact dentin surface (Figs. 1B). After Multilink Sprint application, dentin surface appeared in red (decalcified), but not resin infiltrated (Fig. 1C).

No demineralization/infiltration of dentin was evident for the self-adhesive cements Rely X Unicem (Fig. 1D), G-Cem (Fig. 1E) or Bis-Cem (Fig. 1F), that produced similar interfacial patterns. All light microscopy sections from Bis-Cem group debonded at the cement/dentin interfacial level during laboratory preparation procedures (Fig. 1F).

A scanning electron micrograph of a Bis-Cem bonded to dentin revealed an intimate adaptation with the substrate. However, no signs of hybrid layer formation with the underlying dentin could be noticed; no
resin tags were observed (Fig. 2 B). All the tested auto-adhesive luting cements recorded an acidic pH ranging from 2.1 and 4.2 after mixing (Table 1).
**Figure 1.** Representative light micrographs of cement/dentin interfaces stained with Masson’s trichrome: mineralized dentin (green), resin cement (clear with filler particles), exposed protein (red). A. A distinct red zone of exposed protein was identified in the sections recovered from specimens etched with phosphoric acid (Calibra). B-C. A slight purple line representing collagen partially reacted with resin cement is detectable at the interface between dentin and the self-etching primer (B; Panavia F 2.0) or Multilink Sprint (C). D-E-F. No signs of demineralization and/or exposed protein (red stain) are detectable at the cement/dentin interface of Rely X Unicem (D), G-Cem (E) and Bis-Cem (F). (Original magnification 100x, bar 10 μm).
Figure 2: Scanning electron micrographs of G-Cem (A); RelyX Unicem (B), Multilink Sprint (C), Calibra (D), Panavia F 2.0 (E) and Bis-Cem (F). When using the multi-step resin cement (Calibra), dentin was demineralized and consecutively infiltrated by resin. Resin tags and resin cement/hybrid zone are identified. When luting with self-adhesive cements, tubules were not infiltrated by resin, but intimate adaptation is patent, no distinct morphological manifestation of interaction with dentin may be observed.
Discussion

Within the limitations of this study, the combined application of trichromic technique and SEM examination provided information about the demineralized dentin depth, extent of adhesive diffusion and hybrid layer formation (Spencer and Swafford, 1999). Differences in resin cement diffusion into the prepared dentin surfaces as a function of the pre-treatment regimen were evidenced. The interfacial pattern of the tested simplified self-adhesive cements was not comparable to that of conventional resin-based systems. Thus, the null hypothesis has to be rejected.

The substantial zone of demineralization produced by the etch-and-rinse pre-treatment facilitates resin penetration, but infiltration of Prime&Bond NT was not complete, as it was encountered when compared to other etch&rinse systems (Spencer et al, 2004). It seems that the solvent (acetone) is not able to generate interfibrillar spaces wide enough to accommodate the infiltrating adhesive (Pashley et al, 2002). This adhesive does not contain monomers capable of enhancing diffusion and lowering the initial viscosity of the mixture (HEMA or TEGDMA) (Toledano et al, 2006).

Differing from the application of the etch & rinse system, the mild etching-priming blend (Panavia; pH=2.4) produced minimal dentin demineralization, but resin penetration is identified. It contains ambiphilic monomers (HEMA, 10-MDP, 5-NMSA), with low molecular weight, that may have selectively diffused into dentin (Al-Assaf et al, 2006), forming the hybridized complex (Walker et al, 2002) (Reis et al, 2005).
Similarly to self-etching primer formulations, self-adhesive cements contain multifunctional phosphoric acid methacrylates that are claimed to react with the hydroxyapatite of the hard tooth tissue (Moszner et al, 2005) (Fu et al, 2005) (Hikita et al, 2007). However, no evidences of decalcification/infiltration into dentin are found in any of the tested self-adhesive cements. To achieve a correct infiltration pattern, these cements should be able to etch the substrate in a relatively short time, requiring optimal wetting properties to ensure a fast interaction with dental hard tissues (Moszner et al, 2005). Ideally, the thin smear layer evaluated in this study should allow acidic monomers to freely reach the mineralized tissue underneath (Reis et al, 2005), but it did not occur. Despite of the initial acidic pH, Rely X Unicem and Bis-Cem did not produce any evidence of dentin demineralization and/or hybridization in a micrometer scale (Al-Assaf et al, 2004) (Yang et al, 2006). The adhesive joint appeared essentially similar to that of some conventional luting agents (silicate cements or zinc-phosphate) (Beher et al, 2004).

Some reasons may be advocated regarding the limited capacity of these cements to effectively diffuse up and decalcify the underlying dentin: 1) high viscosity (De Munck et al, 2004), that may rapidly increase as an acid-base reaction (ionic bond formation and setting), that is reminiscent of conventional cements (i.e. silicate or glass-ionomers), is supposed to occur (Fukuda et al, 2003); 2) a neutralization effect during setting, as these chemical reactions involve water releasing and alkaline filler that may raise the pH level (Behr et al, 2004) (Al-Assaf et al, 2006); this neutralization effect may also be exerted by dentin buffering components contained in the smear layer (Reis et al, 2005) (Olivera et al, 2003).
In the attempt of improving diffusivity, a reduction in the initial pH of the cement formulations may be proposed, but it would impair the hydrolytic stability of acidic methacrylates phosphates (Moszner et al., 2005), may reduce polymerization efficacy (Nunes et al.; 2006) and should leave an unprotected interface prone to degradation.

The presence of smear layer has been recognized as the “weak” link in bonding of glass ionomers to dentin, and may also be the case of self-adhesive cements (Al-Assaf et al., 2004). Phosphoric acid etching, prior to the application of the self-adhesive cement, has been shown to be detrimental for effective dentin bonding (De Munck et al., 2004). Most likely, the choice of milder acidic agents to remove the superficial loosely bound fraction of smear layer could somewhat enhance adhesion.

In the case of G-Cem, a self-adhering capacity to dentin may be supposed due to the incorporation of 4-META that bonds by a chelating reaction to calcium ions in apatite (Abo et al., 2004). Water in the cement composition is expected to help the conditioning reaction, reducing the time needed for interacting with the substrate. However, the relatively weak bonding potential and the high molecular weight of the functional monomer are expected to poorly contribute to the supposed chemical reaction, within a clinically reasonable time (Yoshida et al., 2004).

The light discrepancy between demineralization and infiltration depths recorded for Multilink Sprint may be the result of a deeper diffusion of non-cured non-neutralized acidic monomers below the smear layer. Similarly to self-etching primers, these residuals may retain their etching potential forming an unprotected dentin zone and jeopardizing adhesion (Wang and Spencer 2005) (Carvalho et al., 2005).
A bond strength study performed under same laboratory conditions (Mazzitelli et al, 2008), attained results that correlate with present findings. Calibra obtained the highest bond strength and Bis-Cem the lowest (68% of Bis-Cem specimens produced pre-testing failures); Rely-X and G-Cem recorded bond strengths somewhat higher than Multilink Sprint and Bis-Cem, but due to the high standard deviations, differences were not encountered. Long-term bond strength results remain to be ascertained.

Intimate adaptation of self-adhesive cements to dentin was observed by SEM, but no hybrid layer or resin tags formation were evidenced. Other mechanisms (as chemical interactions) had been advocated to occur at these complex interfaces (De Munck et al, 2004) (Hikita et al, 2007). It should also be noticed, that decalcification (red line) of underlying dentin was not produced by any of these cements, so ionic bonding may also be impaired. Following the adhesion/decalcification concept, demineralization is a surface-controlled phenomena involving interaction with hydroxyapatite and depends upon adsorption of the acid anions onto hydroxyapatite. Acidity of the cements/adhesives may not be as determining as previously been thought (Yoshida et al, 2001).

It is worth mentioning that a standardized cementation pressure was applied in this experiment in consideration of previous investigations (Goracci et al, 2006). The cement viscosity most likely decreased while undergoing shear, producing better adaptation and reducing cement film thickness. However, such a thixotropic behavior does not necessarily allow a deeper interaction of auto-adhesive cements with the substrate (De Munck et al, 2004).
In conclusion, self-adhesive cements were not able to completely demineralise/dissolve the smear layer and no decalcification/infiltration of dentin was observed. The presence of partially demineralized/infiltrated smear layer and/or micromechanical retention with dentin may be responsible for the previously reported adhesion values, always weaker than those of conventional resin-based cements (De Munck et al, 2004) (Mazzitelli et al, 2008).
References


Erhardt MC, Toledano M, Osorio R, Pimenta LA. Histomorphologic characterization and bond strength evaluation of caries-


Chapter 3

3.1 Vital dentin as adhesive substrate

Vital dentin is characterized by an outward fluid flow through dentinal tubules. According to the hydrodynamic theory, the rapid fluid movements would activate the pulpal nerves and cause dentine sensitivity (Pashley, 1994). In order to limit this inconvenience, the resinous material should fulfill the tubules and prevent dentine sensitivity. From the other side, there is a common opinion for which the water proceeding through the dentinal tubules may reach the bonded interface and permeate through the material before the advent of the complete polymerization (Itthagarun et al, 2004) (Carrilho et al, 2007). This phenomena is particularly evident in contemporary adhesive systems, whose formulations are based on an high amount of hydrophilic monomers (Yiu et al, 2006). Once they absorb water, they undergo a plasticization effect that affect their mechanical properties (Ito et al, 2005) (Sideridou et al, 2003) (Yiu et al, 2004). In this case, the longevity of the restoration is jeopardize, and microleakage and post-operative sensitivity is more likely to be observed.

The water transudation occurring in vivo conditions should be taken into consideration when simulating the dental biomaterials behaviour in laboratory. Prosthetic preparation does not always require previous endodontic treatments, that is vital dentin would represent the bonding substrate of interest. The convective water fluid flow can be easily reproduced in laboratory allowing for a better simulation of intra-oral conditions. In the following investigation, the effects of a pulpal pressure of 15 cm H2O on the bonding performance of different self-adhesive cements and a cement based on a total-etch system were evaluated. The
microtensile test was used to measure the bond strength in presence or not of a simulated pulpal pressure; representative fractured slices were used for the SEM evaluations.


3.2 Effect of simulated pulpal pressure on self-adhesive cements bonding to dentin.

Introduction


Adverse chemical interactions and adhesive permeability were identified as the two main factors responsible for the reduction in bond strength when auto/dual-cured slow setting resin cements were coupled to bonding on hydrated dentin (Tay et al, 2003). The trans-dentinal fluid movement under a slight positive pulpal pressure may permeate polymerized adhesive interfaces and hinder with the subsequent coupling of the cement (Sauro et al, 2007).

Conventional resin cements that rely on the application of etch-and-rinse adhesives are mostly affected by simulated pulpal pressure, due to the increase in dentin permeability after etching (Musanje et al, 2003). Large fluid shifts during bonding may permit water from dentin to mix with the hydrophilic monomers during solvent evaporation, plasticizing polymer chains and promoting hydrolysis of resin and collagen fibrillar
components (Sauro et al., 2007) (Musanje et al., 2003) (Ito et al., 2005) (Hashimoto et al., 2003) (Yiu et al., 2006) Hashimoto et al., 2004).

Self-etch luting systems do not require separate conditioning of dentin, since their adhesion mechanism is based on the partial retention of smear layer. Although this feature should render them less affected by moisture contamination, degradation of resin-dentin bonds may also be expected to occur also in self-etch systems, due to the presence of hydrophilic monomers and high solvent concentrations in the adhesive blends (Hashimoto et al., 2004) (Okuda et al., 2002).

Simplified self-adhesive cements have been marketed to simplify clinical procedures and overcome the technique sensitivity of multi-step systems. These luting agents do not require any pretreatment of the tooth surface and their application is accomplished through a single clinical step.

The adhesive mechanism is claimed to rely on the chemical reaction between phosphoric acid monomers and hydroxyapatite of dental hard tissues (Pashley et al., 1999) (De Munck et al., 2004). Their application on smear layer-covered substrates should limit post-operative sensitivity and ideally, make these materials less affected to moisture.

Alternative strategies have been recently proposed in an attempt to limit water-induced interfacial changes, such as the application of static seating pressure during luting or an additional layer of hydrophobic resin (Chieffi et al., 2007) (Goracci et al., 2006). However, the real benefit of these procedures under simulated pulpal pressure is uncertain.

Therefore, the aim of this study was to determine the bond strength of different self-adhesive cements to deep-coronal dentin with and without simulated pulpal pressure. The null hypotheses tested are that is that there
is no difference in the bond strength of different adhesive cements to dentin regardless of the presence or absence of simulated pulpal pressure.

**Materials and Methods**

*Experimental design*

Thirty human caries-free third molars stored in 0.5% Chloramine T solution at 4°C were cut above the CEJ to expose flat deep-coronal dentin surfaces. The root of each tooth was cut below the CEJ with a slow speed diamond saw (Isomet, Buehler Ltd, Lake Bluff, IL, USA) under water-cooling so as to expose the pulp chamber. The pulp tissue was completely removed with forceps, trying not to touch the pulp chamber walls.

Composite cylinders were made by layering two 2-mm thick increments of a nano-filled hybrid light cured composite (Aelite All-Purpose Body, Bisco, Schaumburg, IL, USA, shade A3, Batch no. 0500002459) in a split aluminium mold (Ø 8 mm; height: 4 mm). Each increment was light-cured for 40 s with a halogen-curing light Astralis 7 (Ivoclar Vivadent, Schaan, Liechtenstein). Light output was monitored at 600 mW/cm². Specimens were removed from the mold, and additionally light-cured from five aspects for 40 s each on the portion previously in contact with the metal surface of the mold.

Resin blocks were abraded with #600 SiC-paper under water-cooling in order to simulate the clinical condition of sandblasting. Before bonding, each cylinder was cleaned with 34% phosphoric acid (Scotchbond Etchant, 3M ESPE, Seefeld, Germany) for 30 s, rinsed with deionised water for 20 s and air-dried.
Luting procedures

Half of the specimens (n=15) were glued with cyanoacrylate (Super Attak Gel, Henkel Loctite Adesivi, s.r.l. Milan, Italy) to a Plexiglass slab (1.5 x 1.5 x 0.5 cm), taking care that the pulp chamber resulted completely glue-free. On one side of each Plexiglass plate a fissure was created with a diamond bur and a short length of 18-gauge stainless steel tube was glued parallel to the platform extending 2 cm out from the platform (Ciucchi et al, 1995). Each slab-tooth assembly was connected to a 20 mL syringe barrel through polyethylene tubing. All syringe barrels were filled with deionised water to produce a simulated hydrostatic pulpal pressure of 15 cm of H₂O at the dentin surface. Dentin was ground with #600 SiC-paper in order to create a thin smear layer. Then, the surface was etched with 34% ortho-phosphoric acid for 15 s and thoroughly rinsed with water. After assessing the presence of fluid transudation under a stereomicroscope at 40x to confirm the permeability of the dentin, before luting, the smear layer was re-created by means of a new grinding procedure (600 grit).

Five different luting materials were used: 1) Calibra (DeTrey Dentsply, Konstanz, Germany); 2) Multilink Sprint (Ivoclar Vivadent, Schaan, Liechtenstein); 3) BisCem (Bisco, Schaumburg, IL, USA); 4) G-CEM (GC corp., Tokyo, Japan); 5 RelyX Unicem (3M ESPE, Seefeld, Germany).

All the materials were handled strictly following manufacturer’s recommendations, at room temperature (RT: 23.0°C ± 1.0°C) and relative humidity (50% ± 5%). Application mode, chemical composition and pH values of the investigated materials are reported in Table 1.
When Calibra was used for luting, a silane solution (Calibra Silane Coupling Agent, DeTrey Dentsply) was applied to the composite surface to be bonded and spread with air blowing.

The luting procedure was performed under a constant pressure of 1 kg (0.098 MPa) by means of a metal tool at RT until the seating of the material was complete. The seating force was applied for the first 5 minutes leaving the material to set in the self-curing mode. Finally, after 5 min of self-curing, two additional 20 s of light irradiations (Astralis 7) were performed from each side of the specimens to ensure optimal polymerization.

Bonded specimens were stored in a laboratory incubator for one month at 37°C and 100% relative humidity until the microtensile bond strength test was performed. In the case of group 1, the simulated hydrostatic pulpal pressure was maintained until testing.

Microtensile bond strength test

Teeth were sectioned vertically into 1 mm-thick slabs with a slow-speed diamond saw (Isomet). Each slab was fixed on a glass platform with sticky wax and serially sectioned into 1 mm² sticks, according to the “non-trimming” method of the microtensile test. Each stick was measured with a digital caliper (Orteam s.r.l, Milan, Italy), glued with cyanoacrylate (Super Attak Gel) to the free-sliding doors of a Gerardeli’s jig and tested in a universal testing machine (Triax Digital 50, Controls, Milan, Italy; cross-head speed: 0.5 mm/min) until failure.

Failure modes were evaluated by a single operator under a stereomicroscope (Olympus SZ-CTV, Olympus, Tokyo, Japan) at 40x magnification and classified as cohesive (within the cement, dentin or
composite), adhesive (between composite/cement or at the cement/dentin level) or mixed.

Statistical analysis

Bond strength data were first analyzed for normality with the Kolmogorov-Smirnov test and the Levene’s test for equal variance. All the sticks that failed prematurely were included and considered in the statistical calculations as “zero bond” values. As bond strength data were not normally distributed, Kruskall-Wallis Analysis of Variance was applied to assess differences in bond strength among the experimental groups (p<0.05). Mann-Whitney tests were used for post-hoc comparisons (p <0.001).

Scanning Electron Microscopy evaluation (SEM)

Four fractured sticks from each experimental subgroup (previously classified as adhesive or mixed failures) were dehydrated with ascending ethanol solutions, mounted on metal stubs, gold-sputtered (Polaron Range SC 7620, Quorum Technology, Newhaven, UK) and evaluated under a Scanning Electron Microscope (SEM, JSM-6060LV, Jeol, Tokyo, Japan) at different magnification.
<table>
<thead>
<tr>
<th>Material</th>
<th>Composition</th>
<th>Application</th>
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</thead>
<tbody>
<tr>
<td>Calibra (Dentsply) Batch no.</td>
<td>Prime&amp;Bond NT: Acetone; di- and tri-methacrylate resins; urethane dimethacrylate; PENTA; nanofiller-amorphous silicone dioxide; photoinitiators; stabilizers; cetylamine hydrochloride. Calibra: Barium boron fluororutinio silicate glass; bis-pheno l A diglycol dimethacrylate; polymerizable dimethacrylate resin; hydrophobic amorphous fumed silica; titanium dioxide; di-camphorquinone. Catalyst: Barium boron fluorocalcium silicate glass; bis-pheno l A diglycol dimethacrylate; polymerizable dimethacrylate resin; hydrophobic amorphous fumed silica; titanium dioxide; benzoyl peroxide.</td>
<td>Silanize composite. Etch with 37% phosphoric acid for 15 s. Water rinse and air-dry. Apply adhesive in a single coat. Gently air-drying after 5 s. Light cure for 20 s. Mix base and catalyst (1:1). Apply and self-cure (5 min) Light-cure (40 s).</td>
</tr>
<tr>
<td>05110061, pH 0.4</td>
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<tr>
<td>Rely X Ultracur (3M ESPE) Batch</td>
<td>Powder: glass powder, silica; calcium hydroxide, self-curing initiators, pigments, light-curing initiators, substituted pyrimidine, peroxycarbonate, liquid: methacrylated phosphoric esters, dimethacrylates, acetate, stabilizers, self-curing initiators, light-curing initiators.</td>
<td>Mix cement. Apply, self-cure (5 min) and light-cure (40 s).</td>
</tr>
<tr>
<td>no. 233110, pH 2.1</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Multilink Sprint (Ivoclar Vivadent) Batch no.</td>
<td>Dimethacrylates; adhesive monomer; fillers; initiators/stabilizers.</td>
<td>Auto-mix cement. Apply, self-cure (5 min) and light-cure (40 s).</td>
</tr>
<tr>
<td>J22739, pH 4.2</td>
<td></td>
<td></td>
</tr>
<tr>
<td>G-Cem (GC) Batch no. 0601091, pH</td>
<td>UDMA; phosphoric acid ester monomer; 4-META; water; dimethacrylates; silica powder; initiators/stabilizers; fluoro-amino-silicate glass.</td>
<td>Mix cement. Apply, self-cure (5 min) and light-cure (40 s).</td>
</tr>
<tr>
<td>2.7</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Bis-Cem (Ivoclar) Batch no.</td>
<td>Bis (hydroxethyl) methacrylate phosphate (base); tetramethylene glycol dimethacrylate; dental glass.</td>
<td>Auto-mix cement. Apply, self-cure (5 min) and light-cure (40 s).</td>
</tr>
<tr>
<td>060001758, pH 2.1</td>
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</table>
Results

Microtensile bond strength test

Mean microtensile bond strength values recorded in the groups tested are summarized in Table 2. Bond strengths were statistically influenced by the presence of simulated pulpal pressure (p<0.001) and by the type of luting agent (p<0.05).

When no pulpal pressure was applied, the total-etch cement system Calibra exhibited the highest luting strength. RelyX Unicem and G-Cem produced significantly lower bond strengths, while Bis-Cem recorded the lowest μTBS under the same laboratory condition. In the presence of simulated pulpal pressure, bond strength values of Calibra significantly decreased. The self-adhesive cements RelyX Unicem and Bis-Cem gave significant increased in bond strength. The bonding effectiveness of the self-adhesive cements Multilink Sprint and G-Cem were not influenced by the experimental conditions, since no significant differences were found in the presence or absence of simulated pulpal pressure (p>0.05).

The percentage of failure mode distribution is summarized in Table 2. Specimens bonded with self-adhesive cements (Multilink Sprint, Rely X Unicem, G-Cem and Bis-Cem) recorded a higher percentage of cohesive failures within the cement layer under simulated pulpal pressure than when no pulpal pressure was applied. Higher percentages of adhesive failures were related to lower bond strengths. Adhesive failures occurred mainly at the cement/dentin interface. A remarkable percentage of pre-test failures (34-74%, Table 2) were recorded in all the experimental groups.
<table>
<thead>
<tr>
<th>Experimental groups</th>
<th>Failure Mode</th>
<th>Mean (SD)</th>
<th>Failure Mode</th>
<th>Mean (SD)</th>
</tr>
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<tbody>
<tr>
<td></td>
<td>PF</td>
<td>C</td>
<td>A</td>
<td>M</td>
</tr>
<tr>
<td>Multilink Sprint</td>
<td>74%</td>
<td>40%</td>
<td>40%</td>
<td>20%</td>
</tr>
<tr>
<td>Rely X Unicem</td>
<td>23%</td>
<td>37%</td>
<td>36%</td>
<td>27%</td>
</tr>
<tr>
<td>Calibra</td>
<td>41%</td>
<td>48%</td>
<td>20%</td>
<td>32%</td>
</tr>
<tr>
<td>G-Cem</td>
<td>48%</td>
<td>43%</td>
<td>40%</td>
<td>17%</td>
</tr>
<tr>
<td>Bis-Cem</td>
<td>34%</td>
<td>33%</td>
<td>50%</td>
<td>17%</td>
</tr>
</tbody>
</table>

**Table 2:** Mean bond strength (SD) values (MPa) and failure mode distribution (%) recorded in each experimental group. PF: premature failure; C: cohesive (within the cement, dentin or composite); A: adhesive (between the composite and the cement or at the cement/dentin level); M: mixed. Different letters in each column and asterisks in each row indicates significant differences (p<0.001).
Scanning electron microscopy analysis (SEM)

SEM images of debonded beams are shown in Figs. 1-2 (with and without PP, respectively).

When RelyX Unicem was used, structural defects were observed in fractured specimens tested under pulpal pressure (Fig. 1A). These defects consisted of compartmentalized honeycombs and resin globules (Fig. 2) and were absent when no pulpal pressure was applied (Fig. 1B).

Under simulated pulpal pressure, debonded specimens of Bis-Cem were characterized by the presence of dentinal tubules occluded by resin tags (Fig. 1C). The occurrence of globular interfacial agglomerates was assessed when using the cement without PP (Fig. 1D) (Fig. 3).

Multilink Sprint interacted with the underlying dentin forming short resin tags that were detected both in the presence or absence of PP (Figs 1E and F).

Under pulpal perfusion, a detachment of filler from the resinous matrix was noticed when luting with G-Cem (Fig. 1G). This feature was not evident in the absence of pulpal pressure (Fig. 1H).

Irregular adhesive interfaces were evident for the total-etch system Calibra under trans-dentinal perfusion (Fig. 1I). When luting procedures were performed in the absence of pulpal pressure, demineralised/infiltrated areas with resin tags formation were detectable (Fig. 1J).
Fig 1. SEM microphotographs of representative fractured beams bonded respectively with or without PP: A and B) RelyX Unicem; C and D) Bis-Cem; E and F) Multilink Sprint; G and H) G-Cem; I and J) Calibra. Trans-dentinal perfusion somewhat affected the extent of polymerization of RelyX Unicem and Calibra (A and I): areas of compartmentalized honeycomb structures and resin globules are detectable at the interface level. Light demineralization with the formation of resin tags in the tubule orifices is evident in the case of Bis-Cem and Multilink Sprint (C and E). The authors do not know how short or long the tags are from this projection. The application of simulated pulpal pressure impaired G-Cem cohesive strength: frank detachments between the resinous matrix and the filler are evident in the cement bulk (G). In the absence of PP, no signs of interaction with dentin are evident for RelyX Unicem and G-Cem. Multilink Sprint showed a lower demineralising ability and infiltration of dentinal tubules (B and H). Limited areas of poor polymerization are detectable in the Bis-Cem group (D). Tubules filled with adhesive/cement residues are visible when Calibra was applied (J).
**Fig. 2:** High magnification SEM image of a debonded beam luted with RelyX Unicem under PP (1.500x). The fractured surface consisted of porous agglomerate and honeycomb structures, probably filled with water coming from the perfused dentin that made the cement/dentin interface irregular.

![High magnification SEM image of a debonded beam luted with RelyX Unicem under PP (1.500x).](image)

**Fig. 3:** SEM image of a debonded beam luted with Bis-Cem when no pulpal pressure was applied (400x, bar 50 um). The fracture pattern showed a rough, irregular interface. These sites may expedite restoration debonding.

![SEM image of a debonded beam luted with Bis-Cem when no pulpal pressure was applied (400x, bar 50 um).](image)
Discussion

The hydration of dentin surfaces represents a critical variable during bonding procedures, especially when testing adhesive materials in vitro with the intent of simulating in vivo conditions (Sauro et al., 2007).

The results of this investigation require the rejection of the null hypotheses, since microtensile bond strength was affected by the presence of simulated pulpal pressure and the bonding cement.

Deep vital dentin is a highly permeable substrate in which hydrodynamic outward fluid may occur along dentinal tubules (Gale and Darvell, 1999). The presence of smear layer and smear plugs in dentinal tubules limits excessive transudation (Grégoire et al., 2003) (Őzok et al., 2004). To achieve optimal dentinal sealing, resin monomers should flow into tubule orifices, which are water-filled, diffuse into the interfibrillar collagen spaces and properly polymerize forming hybridized resin tags (Őzok et al., 2004).

Nevertheless, dentin wetness and fluid movement through bonded interfaces may hinder optimal resin seal (Tay and Pahley, 2003) (Tay et al., 2004). In the absence of pulpal pressure, the bond strength of the tested etch-and-rinse luting system (Calibra) was significantly higher to that recorded on perfused dentin. The increased trans-dentinal permeability after smear layer removal may have counteracted adhesive penetration and exerted an inhibitory effect on polymerization.

The omission of HEMA in in Prime&Bond NT has been considered advantageous in removing water, separating it from other components upon solvent evaporation (Van Meerbeek et al., 2005). The acetone contained in the adhesive blend is extremely volatile (Glaucher et al,
and its rapid evaporation facilitate the formation of a monomer-rich phase, which may promote cross-linking (Yip and McHugh, 2005). However, excessive transudation of fluids from dentinal tubules and substrate moisture may exceed its water-chasing ability: with acetone evaporation exceeding that of water, the accumulation of the aqueous fraction accumulated in the adhesive film prior to polymerization tends to impair bonding (Zhou et al., 2001).

As self-adhesive cements are applied on smear layer-covered dentin, simulated pulpal pressure should not significantly affect their behaviour. Nevertheless, the simplified luting systems tested recorded differences in their tolerance to wetness and adhesive effectiveness. Manufacturers do not provide detailed information on the chemical composition of these cements. However, the adhesion mechanism of some self-adhesive cements seems closer to the behaviour of conventional luting systems (i.e. silicate or zinc phosphate cements).

Rely X Unicem and Bis-Cem achieved higher bond strengths under simulated dentin perfusion than in the absence of PP revealing a setting acid-base reaction close to that of silicate cements. In the presence of water, silicate cement setting may occur due to the reaction between phosphoric acid and glass silicate fillers (Anusavice, 2003). Theoretically, the phosphoric acid esters of self-adhesive cements behave similarly and need water to become ionized and acid-etch and interact with dentin (Moszner et al., 2005). Since water is not mentioned in their chemical composition and may only derive from the interaction of phosphoric acid groups and alkaline fillers or tooth apatite, intrinsic dentinal wetness may have optimized these acid-base reactions allowing better setting. However, concerns remain regarding the ability of these high viscosity materials to
etch through clinically relevant smear layers into the underlying dentin (Behr et al, 2004) (Gerth et al, 2006).

Despite the improvement in bond strength, RelyX Unicem produced areas of irregular adhesive interfaces under pulpal pressure. The observation of material discontinuity (presence of globules), “honeycomb structures” (Fig. 3) on the fractured dentinal side of the specimens may represent a separation of phase of resin components and has been identified in previous studies that employed self-etch or etch-and-rinse adhesives (Mack et al, 2002) (Tay et al, 2002). In those studies, the authors suggested that the honeycomb structures were filled with water that permeated from dentinal tubules or represented incompletely polymerized regions due to water entrapment. Globules may be the result of the emulsion of resin cement hydrophobic components once in contact with water (Tay and Pashley, 2003) (Carvalho et al, 2004). When stressed to failure, these abundant defects may act as stress raisers that expedite crack propagation through the resin cement bulk (Fig. 3). The higher number of cohesive failures that occurred among the experimental groups when luted under simulated intrapulpal pressure may be related to the excessive water sorption of the material (Hosaka et al, 2007b) (Tay et al, 2005), especially when compared to the control group, in which the adhesive failures at the cement/dentin interface were prevalent.

The absence of pulpal pressure (i.e. dentin perfusion) limited the auto-adhesive cement bonding potential, especially for Bis-Cem.

The presence of water in the chemical composition of G-Cem may explain the similar behaviour exerted by the cement both on moist and perfused dentin. The bonding potential of the functional acidic monomer (4-META) and its high molecular weight, may have contributed to the
chemical reaction with dentin, in both the tested experimental conditions (Abo et al, 2006) (Yoshida et al, 2004).

The patent tubule orifices detected on dentinal substrates bonded with Multilink Sprint in the absence of pulpal pressure, may depend on the slight diffusion of low molecular weight acidic monomers from the high viscosity cement bulk through the smear layer (Fig. 1C). This appearance was more evident on perfused dentin where the cement exerted a superior etching potential most likely due to acidic monomers dilution (Fig. 2C). However, deeper interaction with dentin did not always reflect a superior bonding potential. If not properly neutralized, these monomers may retain their etching potential affecting the polymerizing reaction and jeopardizing adhesion (Carvalho et al, 2004) (Wang and Spencer, 2005).

It is worth mentioning that luting procedures have been previously performed under a sustained pressure (Goracci et al, 2006). Even if resin cements benefits from the application of seating pressure during setting, it is doubtful that this counteracted fluid transudation from the underlying dentin (Chieffi et al, 2007) (Chieffi et al, 2006). It is more likely that the thixotropic behaviour of the materials tested, that are maintained in a low viscosity condition under shear forces, lowered cement thickness allowing better substrate adaptation (De Munck et al, 2004). It may be speculated that the percentage of filler and the particle size may have influenced the results.
References


Chapter 4
4.1 The role of smear layer on the bonding quality of self-adhesive resin cements

Adhesion requires an intimate contact between the luting material and the dental substrate. During prosthetic restoration, smear layer is usually formed. The smear layer is composed of a mixture of denatured collagen and dentinal mineral constituents rearranged after cavity preparation (Pashley, 1992). This layer is a permeable and disaggregate substrate composed of penetrable subunits that can establish interconnections and permit lateral infiltration (Tay et al, 2001) (Prati et al, 1995) (Tay et al, 2000). From one side, as dentin is a permeable substrate, smear layer could limit post-operative sensitivity and excessive water trasudation (Grégoire et al, 2003). The complete removal of smear layer would result in an increased water flux through dentinal tubules (Tay et al, 2005 (Carrilho et al, 2007). The effects of an excessive trans-dentinal fluid movement were identified in an inhibition of the cement polymerization and hydrolitic degradation of resins and collagen fibrillar components, hence hindering optimal seal and bond durability (Sauro et al, 2007) (Musanje et al, 2003). On the other hand, smear layer may represent an inadequate area to be bonded (Glasspoole et al, 2002) and weakened adhesive interfaces are likely to be formed (Tay et al, 2000= (Oliveira et al, 2003).

According to the adhesive system adopted for the adhesion mechanism, smear layer can be totally removed (i.e. with phosphoric acid) or modified and partially dissolved with mild acidic solutions (i.e. polyacrylic acid or oxalate) or chelating agents (i.e. EDTA). Self-adhesive
cements incorporate the smear layer as an intermediate bonding substrate, reducing the possibility of post-op sensitivity possibly reminding the adhesion concepts of simplified adhesive systems. Ideally, this bonding process would result less sensitive to the regional variability of the substrate (i.e. deep and superficial dentin) compared to that employed by multi-step cements that utilize a separate acid etching step. Moreover, the maintenance of the dentin mineral component would be beneficial in terms of bond durability, due to the absence of unprotected collagen fibrils (Hashimoto et al, 2000) (Kato and Nakabayashi, 1998).

To date, there is no accordance regarding the effective ability of self-adhesive cements to attain a reliable bond strength to dentin. Possibly, the cements do not possess the capacity necessary to modify the smear layer, demineralize and simultaneously infiltrate the underneath dentin (Al-Assaf et al, 2007). Previous studies, demonstrated the negative effects of pre-conditioning dentin with 35% phosphoric acid (De Munck et al, 2004) (Hikita et al, 2007). Although the acid completely remove the smear layer and open up the dentinal tubules, the cements possess an high viscosity that would impede the diffusion into the conditioned substrate.

In the following investigation, the effect of two mild acidic solutions (EDTA and/or 10% polyacrylic acid) on the bonding performance of three self-adhesive cements were tested. The microtensile test was associated with a scanning electron microscopy evaluation of fractured beams. An histomorphologic characterization of the adhesive interface with or without pre-treating dentin was also executed in order to individuate the efficacy of the cement to penetrate into the partially cleaned dentin.
References


4.2 Dentin treatment effects on the bonding performance of self-adhesive resin cements.


Introduction

Self-adhesive resin cements were introduced to lute indirect restorations, such as all-ceramic crowns, composite inlays/onlays or fiber posts with a simple and standardized approach. The use of self-adhesive cements is accomplished by a single clinical step, where a simultaneous demineralization/infiltration of the substrate is expected to occur. Previous studies revealed dentin bond strength values of some self-adhesive cements comparable to those of conventional resinous luting agents (Piwowarczyk et al, 2007) (Bitter et al, 2006). However, the bonding effectiveness of these simplified cements on smear layer-covered dentin still remain a concern (Mazzitelli et al, 2008) (De Munck et al, 2004) (Goracci et al, 2006).

Recent studies reported that self-adhesive cements might superficially interact with dentin, leading to smear layer partial demineralization and short resin tags formation (Mazzitelli et al, 2008) (Monticelli et al, 2008) (Al-Assaf et al, 2007). The presence of partially demineralized/infiltrated smear layer at the adhesive interface may result in a relatively poor bonding mechanism (De Munck et al, 2004) (Glasspoole et al, 2002) and weak adhesive interfaces are likely to be formed (Tay et al, 2000) (Oliveira et al, 2003). Although self-adhesive
cements do not require pre-treatment of the dental substrate, previous removal of the smear layer with acidic solutions has been proposed in order to enhance a direct cement/dentin interaction and bond strength improvement (De Munck et al, 2004) (Hikita et al, 2007) (Behr et al, 2004). It is noteworthy that dentin conditioning with 35% phosphoric acid before luting with an auto-adhesive luting material was ineffective or detrimental (De Munck et al, 2004) (Hikita et al, 2007). The use of milder acidic agents (i.e. EDTA and polyacrilic acid) was suggested (De Munck et al, 2004) (Monticelli et al, 2008) (Behr et al, 2004). These conditioning agents could partially remove the smear layer, leaving the dentin mineral phase, ideally enhancing the chemical reaction between the cement and the substrate (Monticelli et al, 2008).

Most self-adhesive cements contain functional methacrylated phosphoric ester monomers and little is known about their chemical interaction potentials with hydroxiapatite (Mine et al, 2009). Adhesion may also be dependent on the chemical formulation and physical properties (i.e. wetting) of the cement (Sarr et al, 2009). Therefore, self-adhesive cements containing hydrophilic compounds (4-META, HEMA) or solvents (water, HEMA) were included in the study.

The purpose of this study was to compare the dentin/cement bond strengths and the interfacial characteristics of three commercial adhesive cements as a function of different pre-treatment regimens. A staining technique for optical microscopy, that specifically identifies depth of decalcification/infiltration or exposed collagen at the dentin/cement interface, was employed. SEM analysis of debonded surfaces was also performed.
The null hypothesis tested was that dentin treatments do not influence the bond strengths or the morphologic characteristics of the self-adhesive resin cement-dentin interfaces.

**Materials and Methods**

*Samples preparation*

Forty-five intact, non-curious human third molars were employed after the institutional informed consent from all donators. The protocol was approved by the Research Ethics Commission. Molars were stored in 0.5% Chloramine T solution at 4°C for less than one month. Teeth were decoronated to expose flat, enamel-free, deep-coronal dentin surfaces. The pulp chamber of each tooth was exposed after cutting the root below the CEJ with a slow speed diamond saw (Isomet, Buehler Ltd, Lake Bluff, IL, USA) under water-cooling. The pulp tissue was carefully removed with a forceps, being worry to not touch the pulp chamber walls.

Two 2-mm thick increments of a nano-filled hybrid light-cured composite (Aelite All-Purpose Body, Bisco, Schaumburg, IL, USA, shade A3, Batch n° 0500002461) were layered in a split aluminium mold (Ø 8 mm x 4 mm height) to prepare composite cylinders. Each increment was light-cured for 40 s with an halogen-curing light (Astralis 7, Ivoclar Vivadent, Schaan, Liechtenstein, 600 mW/cm²). Composite cylinders were removed, additionally light-cured from four aspects for 40 s each and on the portion previously in contact with the metallic surface of the mold.

*Bonding procedure*

Each tooth was glued with cyanoacrylate (Super Attak Gel, Henkel Loctite Adesivi, s.r.l. Milan, Italy) to a Plexiglass slab (1.5 x 1.5 x 0.5 cm).
On one side of each Plexiglass plate a fissure was created with a diamond bur and a short length of 18-gauge stainless steel tube was glued parallel to the platform extending 2 cm out from it (Ciucchi et al., 1995). A polyethylene tubing joined each slab-tooth assemblage to a 20 ml syringe. All syringes were filled with deionised water to produce a simulated hydrostatic pulpal pressure of 15 cm of H$_2$O at the dentin surface. A thin smear layer was produced on dentin with #180 SiC-paper.

Three experimental groups (n=15) were prepared according to the dentin pre-treatment: 1) No dentin pre-treatment; 2) 0.1 M EDTA (pH 7.4) was scrubbed onto the dentin surfaces with a micro-brush for 60 s and then rinsed with deionised water for 10 s; 3) 10% polyacrylic acid (Voco, Cuxhaven, Germany, Lot: 691545) was applied with a micro-brush for 30 s, water rinsed for 30 s.

Resin composite cylinders were luted to the prepared dentin surface (n=5 each group) with: 1) RelyX Unicem (3M ESPE, Seefeld, Germany) a hydrophobic and solvent-free self-adhesive cement; 2) Bis-Cem (Bisco, Schaumburg, IL, USA), an HEMA-based self-adhesive cement; 3) G-Cem (GC corp., Tokyo, Japan) a 4-META-based and water containing self-adhesive cement. Materials were handled according manufacturers’ recommendations at room temperature (23.0°C ± 1.0°C) and relative humidity (50% ± 5%). pH of each material was measured. After mixing, the material was dispensed on pH indicator strips with narrow ranges (0.0-1.8; 1.8-3.8; 3.8-5.5; Panreac Química, Barcelona, Spain). Application mode, chemical composition and pH values of the tested materials are reported in Table 1.

A constant standardized pressure of 40 g/mm$^2$ by means of a metallic tool was applied (Goracci et al., 2006); the seating force was maintained
for the first 5 minutes leaving the material to set in the self-curing modality. Additional 20 s of light irradiations (Astralis 7, 600 mmW/cm²; the tip was maintained at a distance of 5 mm) from each side of the specimen and at the top of the restoration previously in contact with the metal were performed in order to ensure an optimal polymerization (Vrochari et al., 2009).

Three bonded specimens per group were stored in a laboratory oven (37°C and 100% relative humidity) maintaining the simulated hydrostatic pulpal pressure for one month until the microtensile bond strength test.

**Microtensile bond strength test**

Specimens were detached from the Plexiglass slab with a scaffold, taking care to not touch and stress the bonded interfaces. Each tooth was sectioned vertically into 1 mm-thick slabs with a slow-speed cut off diamond wheel (Isomet) under copious water cooling. Each slab was then serially sectioned into 0.9 x 0.9 mm sticks, according to the “non-trimming” technique of the microtensile test. Each stick was measured with a digital caliper (Orteam s.r.l, Milan, Italy), glued with cyanoacrylate (Super Attack Gel) to the free-sliding doors of a jig and stressed to failure in tension by means of a universal testing machine (Triax Digital 50, Controls, Milan, Italy; cross-head speed: 0.5 mm/min).

Prematurely fractured sticks were included in the statistical analysis and considered as “zero bond” values. The normal and equal distribution of the bond strength data was first checked by Kolmogorov-Smirnoff and Levene’s tests, respectively. As bond strength values were not normally distributed, Kruskall-Wallis Analysis of Variance was used to analyze the differences in bond strengths among the experimental groups (p<0.05).
with the bond strength as the dependent variable, surface conditionings and luting cements as factors. A series of Mann-Whitney tests (p<0.001) were used for post-hoc comparisons. Calculations were handled using the SPSS 14.0 software (SPSS Inc.; Chicago, IL, USA).

Failure modes were examined by a single operator under a stereomicroscope (Olympus SZ-CTV, Olympus, Tokyo, Japan) at 40x magnification and classified as cohesive (within the cement, dentin or composite), adhesive (between the restoration and the cement or at the luting agent/dentin level) or mixed (adhesive and cohesive fractures occurred simultaneously).

**Scanning electron microscope evaluation (SEM)**

Three fractured sticks were selected from each experimental group and dehydrated with ascending ethanol solutions and air-dried, mounted on metallic stubs, gold-sputtered (Polaron Range SC 7620, Quorum Technology, Newhaven, UK) and evaluated under a Scanning Electron Microscope (SEM, JSM-6060LV, Jeol, Tokyo, Japan) at different magnifications.

**Light microscopy – Masson’s trichrome staining technique**

After 24-h, two remaining bonded teeth per group were sectioned perpendicularly to the bonded interface into 1-mm thick slabs using a low-speed diamond wheel under water cooling (Isomet 1000). A total of eight sections were analyzed for each experimental group. The medial aspect of each resin-dentin bonded slab was glued on methacrylate supports with a photo-curing adhesive (Technovit 7200 VLC, Kulzer, Norderstedt, Germany) and grinded with an Exakt polishing machine (EXAKT
Technologies Inc., Oklahoma City, OK, USA) using SiC abrasive wet paper of increasing fine grits (# 800; 1200; 2500; 4000) until getting a thickness of 5-6 µm. Sections were treated with Masson’s trichrome acid staining technique (Monticelli et al, 2008) (Erhardt et al, 2008). Each slide was then cover-slipped and ready to be examined using a light microscope (BH2, Olympus, Tokyo, Japan).

**Table 1.** Chemical composition, pH values and application modality of the tested self-adhesive resin cements.

<table>
<thead>
<tr>
<th>Material</th>
<th>Composition</th>
<th>Application</th>
</tr>
</thead>
<tbody>
<tr>
<td>RelyX Unicem</td>
<td><strong>Powder:</strong> glass fillers, silica, calcium hydroxide, self-curing initiators, pigments, light-curing initiators, substituted pyrimidine, peroxo compound. <strong>Liquid:</strong> methacrylated phosphoric esters, dimethacrylates, acetate, stabilizers, self-curing initiators, light-curing initiators</td>
<td>Mix cement. Apply, self-cure (5 min) and light-cure (40s).</td>
</tr>
<tr>
<td>(3M ESPE)</td>
<td><strong>Batch n°:</strong> 270644 pH: 2.1</td>
<td></td>
</tr>
<tr>
<td>Bis Cem</td>
<td>Bis (Hydroxyethyl methacrylate) phosphate (Base); Tetraethylene glycol dimethacrylate; dental glass.</td>
<td>Auto-Mix cement. Apply. self-cure (5 min) and light-cure (40s).</td>
</tr>
<tr>
<td>(Bisco)</td>
<td><strong>Batch n°:</strong> 0600010898 pH: 2.1</td>
<td></td>
</tr>
<tr>
<td>G-Cem</td>
<td>UDMA; phosphoric acid ester monomer; 4-META; water; dimethacrylates; silica powder; initiators/stabilizers; fluoro-amino-silicate glass.</td>
<td>Mix cement. Dispense, self-cure (5 min) and light-cure (40s).</td>
</tr>
<tr>
<td>(GC Corp.)</td>
<td><strong>Batch n°:</strong> 0611091 pH: 2.7</td>
<td></td>
</tr>
</tbody>
</table>
Results

**Microtensile bond strength test**

Mean microtensile bond strengths and standard deviations (SD) recorded in the experimental groups are displayed in Table 2. Bond strengths were influenced by dentin pre-treatment and luting cement (p<0.05).

No differences were evidenced for the hydrophobic and solvent-free cement (RelyX Unicem) after the different tested conditioning modes. The HEMA-based self-adhesive cement (Bis-Cem) recorded higher bond strengths when luted to non treated dentin; bond strength values dropped after EDTA or after polyacrylic acid treatments, without differences between these two groups. Bond strengths of the 4-META-based and water containing cement (G-Cem) significantly increased after 10% polyacrylic acid etching.

The percentage of pre-testing failures and fracture classification recorded in the experimental groups are presented in Table 3. The most frequently observed fracture modes were adhesive between cement and dentin surface or cohesive within the cement. The number of cohesive failures increased after treating dentin with EDTA or polyacrylic acid. Mixed failures were also assessed for all the cements on non treated dentin.
Table 2. Mean microtensile bond strength (SD) values (MPa) and post-hoc comparisons results obtained for the experimental groups (n=3). Different letters in each column and asterisks in each row indicate significant differences (p<0.005).

<table>
<thead>
<tr>
<th>Experimental Groups</th>
<th>No-Treated dentin</th>
<th>EDTA</th>
<th>PAA</th>
</tr>
</thead>
<tbody>
<tr>
<td>RelyX Unicem</td>
<td>16.73(12.5) B</td>
<td>12.4(9.9) B</td>
<td>14.87(15.5) B</td>
</tr>
<tr>
<td>Bis-Cem</td>
<td>12.29(11.1) AB*</td>
<td>7.47(8.4) A</td>
<td>6.36(9.4) A</td>
</tr>
<tr>
<td>G-Cem</td>
<td>8.03(8.2) A</td>
<td>10.3(8.4) AB</td>
<td>13.47(11.5) B*</td>
</tr>
</tbody>
</table>

Table 3. Percentage of failures recorded in each experimental group. PF: premature failures; A: adhesive (at the cement/composite or cement/dentin interfaces); C: cohesive (within the cement); M: mixed (a combination of A and C).

<table>
<thead>
<tr>
<th>Exp. Groups</th>
<th>No-Treated dentin</th>
<th>EDTA</th>
<th>PAA</th>
</tr>
</thead>
<tbody>
<tr>
<td>Failure mode</td>
<td>PF</td>
<td>A</td>
<td>C</td>
</tr>
<tr>
<td>RelyX Unicem</td>
<td>23%</td>
<td>36%</td>
<td>37%</td>
</tr>
<tr>
<td>Bis-Cem</td>
<td>34%</td>
<td>50%</td>
<td>33%</td>
</tr>
<tr>
<td>G-Cem</td>
<td>48%</td>
<td>40%</td>
<td>43%</td>
</tr>
</tbody>
</table>
Scanning electron microscopy (SEM) analysis

Debonded dentin surfaces are shown in Fig. 1. When the hydrophobic cement (RelyX Unicem) was used on non treated dentin, voids and bubbles within the cement were observed. Structural defects consisting on compartimentized honeycombs and resin globules were detectable (Fig. 1A). When EDTA-treated surfaces were observed, an alternation of filamentous debris and voids within the cement layer were found (Fig. 1B). At the polyacrylic acid etched group an area of cohesive dentin fracture was detected, smear layer removal and opened dentinal tubules were noticed and some protruding resin tags were evidenced (Fig. 1C). When using the HEMA-based luting agent (Bis-Cem), partial removal of smear layer and presence of a compact cement layer still adhered to the dental substrate were seen (Fig. 1D). In the EDTA-treated group, a more porous cement layer was evident (Fig. 1E). When the cement was bonded to polyacrilic acid etched dentin, dentinal tubules were visibly opened with limited resin tag formation (Fig. 1F). The 4-META-based self-adhesive cement (G-Cem) completely covered the underneath non-treated dentin (Fig. 1G). Tubules partially opened, with some resin tags and cement remnants adhered to the underlying substrate were assessed when the material was luted on EDTA treated dentin (Fig. 1H). After polyacrylic acid etching, dentinal tubules were opened and cement infiltrations encountered at the intertubular dentin (Fig. 1I).
Fig. 1: SEM images of debonded sticks (dentin side) after microtensile bond strength test. Each debonded stick (dentin side) is shown at 85x, while zones shown at higher magnifications are marked with white asterisks. A) RelyX Unicem on non treated dentin; B) RelyX Unicem and EDTA treated dentin; C) RelyX Unicem and polyacrylic acid etched dentin; D) Bis-Cem luted on non treated dentin; E) Bis-Cem and EDTA demineralized dentin; F) Bis-Cem on polyacrylic acid dentin; G) G-Cem on non treated dentin; H-I) G-Cem on EDTA and polyacrylic acid treated dentin, respectively.
Light microscopy – Masson’s trichrome staining technique

Light microscopy images of cement/dentin interfaces after the Masson’s trichrome staining are presented in Fig. 2, where the dentin minerals stain green, the unprotected proteins stain red and the cement appears beige.

No demineralization was seen for the HEMA-based and 4-META-based self-adhesive cements when luted to non-treated dentin (Figs. 2D and G). Slight purple spots and a narrow light red layer, representing mild collagen demineralization, were observed at the bottom of the adhesive interface when the hydrophobic and solvent-free cement was used on non treated or polyacrylic acid etched dentin respectively, but no clear dentin infiltration could be assessed (Figs. 2A and C). When the same self-adhesive cement was luted on EDTA decalcified dentin, no signs of demineralization/infiltration were observed (Fig. 2B). Dentin was infiltrated when the HEMA-based cement (Bis-Cem) was applied onto EDTA or polyacrylic acid treated dentin (Figs. 2E and F). The cement containing 4-META and water (G-Cem) seemed to infiltrate the EDTA and/or polyacrylic acid treated dentin (Figs. 2H and I). Short and sparsely distributed resin tags formations were noticed at the cement/dentin interfacial levels, although areas of partially exposed collagen fibrils (purple spots) not enveloped by resin were present beneath the cement (Fig. 2I).
Fig. 2: Light microscopy images of cement/dentin interfaces stained with Masson’s trichrome on non treated, EDTA and polyacrylic acid etched dentin: mineralized dentin (green), resin cement (beige), exposed collagen (red). A) RelyX Unicem on non treated dentin: slight purple intermittent spots immediately beyond the cement were evidenced, but no resin penetration could be assessed; B) RelyX Unicem/EDTA; C) RelyX Unicem/polyacrylic acid; D-E-F) Bis-Cem bonded non non treated, EDTA and polyacrylic acid treated dentin, respectively; G) G-Cem and non treated dentin; H-I) G-Cem on EDTA and polyacrylic acid treated dentin, respectively.
Discussion

The null hypothesis must be rejected since both dentin pre-treatment and cement type influenced the microtensile bond strengths of self-adhesive cements to dentin. The interfacial characteristics of the self-adhesive cements-dentin interfaces varied according to the performed surface treatment. Reproducing a positive intra-pulpal pressure during luting procedures, resulted in different bonding performances when self-adhesive cements where used for the cementation of composite overlays (Mazzitelli et al, 2008). The maintenance of the hydration state of dentin was suppose to optimize the chemical reaction of selected self-adhesive cements. For this reason, a simulated pulpal pressure was applied during cementation and preserved until testing in the present study.

No differences in bond strengths were observed when the hydrophobic and solvent-free self-adhesive cement (RelyX Unicem) was tested in the three experimental conditions (Table 2). The calcium chelating ability of EDTA produced slight smear layer and smear plug removal and, although no increase in surface roughness is expected (Osorio et al, 2007), the cement appeared partially adherent to the bonded substrate (Fig. 1B). After polyacrylic acid etching dentinal tubules were visibly opened, inter-tubular dentin was exposed and limited resin infiltration could be observed (Fig. 1C). Exposed and un-enveloped collagen network at the bottom of the adhesive interface were also noticed by light microscopy (purple spots) (Fig.2C). The maintenance of dentinal inter-fibrillar mineral components (after tested dentin treatments) has been suggested to facilitate the chemical bonds with the cements and ideally

Pre-treatment of dentin caused a significant drop of the HEMA-based cement (Bis-Cem) bond strengths (Table 2). Demineralised and non-infiltrated dentin was not found at the light microscopy images of these bonded interfaces, when dentin was pre-treated with EDTA or polyacrylic acid (Figs. 2E and F). The cement is composed by a mixture of Bis-HEMA and, in a lower percentage, of TEGDMA as a diluent agent (Table 1). HEMA is a water-soluble molecule that promote resin infiltration into demineralised dentin (Nakabayashi and Takarada, 1992), but it can also attracts water from the underneath perfused dental substrate, leading to poor polymerization (Nunes et al, 2005), and mechanical properties decrease, jeopardizing bonding performances (Carvalho et al, 2004) (Van Landuyt et al, 2008). Water exposure would also exert a detrimental plasticization effect (Nunes et al, 2005) (Carvalho et al, 2004). The water content of the dentin has differential effects on the bond strengths of self-adhesive cements (Mazzitelli et al, 2008). After dentin conditionings (in the presence of pulpal pressure) the gradient of water fluid flow will
increase at the bonded interfaces causing water entrapment within this HEMA-rich material (Carvalho et al, 2004) (Van Landuyt et al, 2007). The accumulation of water within the cement will give the material a porous appearance when bonded on perfused, treated dentin (Fig. 1E). Filamentous formations onto the treated dentin surfaces and non-resin filled opened dentinal tubules were assessed after treating dentin with polyacrylic acid (Fig. 1F). The high number of cohesive failures registered for the Bis-HEMA-based cement after pre-treating dentin with EDTA and/or polyacrylic acid (Table 3) suggests that the bond strength decrease will be related to the inherent weakness of the material in presence of abundant water.

Polyacrylic acid etching of dentin caused a significant increase in bond strength for the 4-META-based cement (G-Cem) (Table 2). According to the manufacturer, this cement exhibits a glass-ionomer like technology. It contains water, fluoro-alumino-silicate glass and phosphoric acid esters (Table 1), and therefore a setting reaction similar to that of silicate cements may occur (Anusavice, 2003). A chemical interaction between the cement and the dentinal mineral content may also be expected after polyacrylic acid application (Wilson et al, 1983). Treating dentin with this acidic solution may result beneficial in activating ions (i.e. P and Ca\(^+\)) from the dental substrate that can be incorporated into the cement mass and enhance a chemical reaction between the cement and the dentin (Tay et al, 2007) (Yoshida et al, 2001). Polyacrylic acid application will also increase dentin surface roughness facilitating micromechanical retentions being a clear benefit for the bonding process. The interfacial evaluation revealed a slight discrepancy between etching depth and cement penetration (a slight narrow purple line at the bottom of the resin
layer) (Fig. 2I). The limited diffusion of this viscous filled cement may produce an area of exposed and non-resin impregnated collagen layer at the adhesive joint, that is prone to premature degradation (Wang and Spencer, 2005) (Spencer et al, 2004) (Hashimoto et al, 2003). EDTA treatment of dentin did not improve the bond strengths of this cement based upon a glass-ionomer technology. EDTA exerts just a slight smear layer removal and does not increase surface roughness (Osorio et al, 2007) (Osorio et al, 2005), hence mechanical interlocking within the intertubular dentin will not be facilitated (Coli et al, 2003). However, EDTA treatment caused opening of dentinal tubules that resulted in some resin tags formations (Fig. 2H). It remains to be proved if a previous application of adhesives as intermediate layers would be beneficial in promoting cement penetration (Hikita et al, 2007) (Behr et al, 2004) (Lürhs et al, 2009). Self-adhesive cements are a heterogeneous subgroup of resin cements and there are substantial differences between them in terms of setting reaction, chemical composition and pH. It is clear that a more specific classification of these new-marketed materials is needed. More information from manufacturers regarding their exact chemical compositions and further research will be highly desirable.

Within the limits of this study, it may be concluded that self-adhesive cements were not able to completely demineralise/dissolve the smear layer and no decalcification/infiltration of dentin was observed. Dentin conditioning facilitates smear layer removal, but the viscosity of the materials hampers their penetration into dentin. Opening of dentinal tubules permits resin tags formations, but also produces a water flow that may affect bond strengths. The exact bonding mechanism of these simplified materials remains to be ascertained.
References


Osorio R, Aguilera FS, Osorio E, Cabrerizo-Vilchez MA, Toledano M. Changes in surface roughness properties of dentin after different
conditioning treatment. Ab. #1515; IADR, 21-24 March 2007; New Orleans, LA, USA.


Chapter 5

5.1 Self-adhesive cements and fiber posts

Fiber post are clinically used to restore endodontically treated teeth with massive coronal distruction and they are necessary to increase the retention of the coronal restoration. Their clinical behaviour and mechanical properties have been widely investigated (Ferrari et al, 2003) (Grandini et al, 2005) (Malferrari et al, 2003) (Cagidiaco et al, 2007) (Ferrari et al, 2007). Factors that may affect the bonding of fiber posts to root canal dentin are considered the lack of direct vision due to the restricted access (D’Arcangelo et al, 2008a), the difficult control of moisture when using multi step systems (Toba et al, 2003), the type of luting agents (Akgunkor et al, 2006) (Bitter et al, 2006) and the cementing techniques (D’Arcangelo et al, 2008b). Although a vertical root fracture represents the most severe cause of irreparable failure, all these factors may contribute to a premature loss of retention at both dentin/cement or cement/post interfaces.

In a context in which the concept of a post/cement/root dentin monoblock unit results quite unpredictable, the type of luting agent, intended as the physical join between post and dentin, become the instrument for obtaining a reliable restoration. Several luting material have been proposed over the year for the cementation of fiber posts, although resin cements showed good mechanical properties and were related to higher dilocaunt resistance when compared to conventional cements. Information regarding the ability of self-adhesive cements to lute fiber posts is relatively present in literature and contrasting results in the immediate and long-term data could be noticed. To date, different self-adhesive cements are available in the market. Differences in their chemical
composition would influence their bonding mechanism, that is each material should be considered independent of the class it belong to.

In this chapter, the bonding potential of different cements was evaluated with the push-out test. Thermal ageing was performed in the second study in order to foresee whether simplified composite cements would resist to the physiologic changes that occur in the oral cavity.
References


5.2 Evaluation of the push-out bond strength of self-adhesive resin cements to fiber posts

Introduction

Fiber posts are increasingly used for the restoration of endodontically treated teeth with massive coronal distructions. Their mechanical properties and clinical behaviour have widely been investigated (Ferrari *et al*, 2003) (Grandini *et al*, 2005) (Cagidiaco *et al*, 2007) (Ferrari *et al*, 2007) The dislocation resistance of fiber posts into root canal is significantly influenced by the luting agent and the cementation procedures (D’Arcangelo *et al*, 2008a). Resin cements allow superior post retention and increase the fracture resistance of the post-restored tooth when compared to conventional cements (Bitter *et al*, 2006) (D’Arcangelo *et al*, 2008b) (Qualthrough and Mannocci, 2003)

Several factors contribute to render post luting procedures difficult: the lack of direct vision and the limited access to the bonding substrate make cementation procedures very technically-related (D’Arcangelo *et al*, 2008a). Moisture control within root canals represents an additional limitation during the management of multi-step resin cements (Chersoni *et al*, 2005).

Self-adhesive resin cements simplified luting procedures of indirect restorations and have been designed to be less technique-sensitive than their multi-step counterparts. Laboratory investigations found comparable bond strength values between RelyX Unicem and resin cements that
utilized two or three-steps adhesives (Radovic et al, 2008) (Bitter et al, 2006) RelyX Unicem is undoubtedly the most investigated self-adhesive material, notwithstanding the variety of products that have been launched by different manufacturers. Self-adhesive cements possess different chemical compositions and dispensing modalities that could influence their mechanical properties and bonding performances.

Push-out test is an appropriate method to measure the bond strength inside the root canals. The shear stresses are created parallel to the cement/dentin and cement/post interfaces, resulting in a better simulation of the stress occurring in clinical conditions (Goracci et al, 2007) (Goracci et al, 2004).

The purpose of the present study was to assess the push-out bond strength of three self-adhesive resin cements used for the cementation of epoxy resin-based fiber posts. The null hypothesis tested was that no differences in bond strength are present among different self-adhesive resin cements independently from their chemical compositions and application mode.

**Materials and Methods**

*Specimen preparation*

Thirty extracted, single-rooted, carie-free human premolars stored in 0.5% Chloramine T solution at 4° C for preventing bacterial growth were selected for the study after informed consent of the donors was obtained. The crown of each tooth was removed 1 mm above the CEJ by means of a slow speed diamond saw (Isomet, Buehler, Lake Bluff, IL, USA) under copious water cooling. Working length was established at 1 mm from the root apex. Cleaning and shaping of the root canal were performed with
Protaper Ni-Ti rotatory instruments (size S1, S2, S3; Dentsply Maillefer, Ballagues, Switzerland) following the crown-down technique. Irrigations with 5% sodium hypochlorite were performed between the instrumentations. Gutta-percha cones (Coltène/Whaledent, Langenau, Germany) were used for filling the root canal and cemented with a resin sealer (AH Plus Jet, Dentsply DeTrey, Konstanz, Germany) following the lateral condensation technique. Roots were then coronally sealed with a temporary restorative material (Fuji VII, GC Corporation, Tokyo, Japan; batch n° 0410221) and stored in a laboratory oven at 37° C and 100% relative humidity. After 24 hours, the temporary seal was abraded by means of #240 SiC paper under water cooling, and the coronal gutta-percha was removed with a pre-shaping drill (Dentsply DeTrey, Konstanz, Germany), leaving a 5 mm-long apical seal. A 7 mm-deep post space was prepared with a universal drill (3M ESPE, Seefeld, Germany) to match the size of the co-respective RelyX Fiber Post (#1; 3M ESPE, Seefeld, Germany; LOT: 02363200603). The drilled canal was gently air-blowed in order to eliminate any residual gutta-percha. Three self-adhesive composite cements were used for fiber post cementation (n=10): 1. Rely X Unicem (3M ESPE, Seefeld, Germany); 2. Multilink Sprint (Ivoclar-Vivadent, Schaan, Liechtenstein); 3. Max-Cem (Kerr Corp, Orange, CA, USA).

Materials were handled according to manufacturer’s instructions. Application modes, chemical compositions and batch numbers of the investigated materials are listed in Table 1.

The cements were used in the dual-cured modality. After the first 5 min of auto-cure in which the post was loaded under finger pressure, additional 40 s of light polymerization through the translucent fiber post
were performed (Astralis 7, Ivoclar-Vivadent, Schaan, Liechtenstein; output: 500 mW/cm²). The cement in excess was carefully removed with a spatula. A core build-up was performed with Fuji VII (GC corp; LOT: 0703071). Specimens were maintained for 1 month in a flower sponge slightly wetted with demonized water and stored in a laboratory incubator (37±1 °C).

**Push-out bond strength test**

The portion of the root containing the fiber post was subsequently sectioned into four to six 1 mm-thick slices with a diamond saw (Isomet) under water cooling. The cylindrical plunger of the testing machine (Triax 50, Controls S.P.A, Milan, Italy) was forced to dislodge, via an apical-coronal direction, each inverted, truncated fiber post from the root dentin. A load (0.5 mm/min until failure) was then applied to the post surface that resulted in shear stresses along the cement/dentin – cement/post interfaces. The retentive strength of the post fragment (MPa) was calculated by dividing the load at failure (Newton) by the interfacial area of the post segment ($S_L$). The formula used for measuring the tronco-conical area was so expressed:

$$S_L = \pi (R+r) \left( h^2 + (R-r)^2 \right)^{0.5}$$

In which $\pi$ was equal to 3.14, $R$ and $r$ were the coronal and the apical post radius respectively, and $h$ the root slice thickness. The diameters of the post and the thickness of the slice were individually measured using a digital caliper with 0.01 mm accuracy.

Failure modes were evaluated by a single operator under a stereomicroscope (Olympus SZ-CTV, Olympus, Tokyo, Japan) at 40x magnification and classified as cohesive (within the post), adhesive
(between the post and the cement or at the cement/intra-radicular dentin level) or mixed (adhesive and cohesive fractures occurred simultaneously).

One stressed-to-failure slice per group was used for scanning electron microscopy (SEM) evaluation. Specimens were rinsed with ascending ethanol solutions, mounted on metallic stubs, gold-sputtered (Polaron Range SC 7620, Quorum Technology, Newhaven, UK) and observed under a scanning electron microscope (JSM-6060LV, Jeol, Tokyo, Japan).

**Statistical analysis**

The normal and equal distributions of the push-out bond strength data were first checked and verified by the Kolmogorov-Smirnov and Levene’s test respectively. A one-way ANOVA was performed to verify the differences in push-out bond strengths between the tested luting cements (p<0.05). A Tukey test was then executed for post-hoc comparisons (p<0.001). Calculations were handled by the SPSS 15.0 software (SPSS Inc.; Chicago, IL, USA).
Table 1. Manufacturers, chemical compositions and application modes of the materials tested in the study.

<table>
<thead>
<tr>
<th>Material</th>
<th>Composition</th>
<th>Delivery system</th>
<th>Instructions for use</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Rely X Unicem</strong></td>
<td><strong>Powder:</strong> glass fillers, silica, calcium hydroxide, self-curing initiators, pigments, light-curing initiators, substituted pyrimidine, peroxo compound. <strong>Liquid:</strong> methacrylated phosphoric esters, dimethacrylates, acetate, stabilizers, self-curing initiators</td>
<td>Capsule and Aplicap Elongation Tip</td>
<td>Mix cement. Apply, self-cure (5 min) and light-cure (40s)</td>
</tr>
<tr>
<td>(3M ESPE,)</td>
<td>Batch n°:270644 pH= 2.1</td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Multilink Sprint</strong></td>
<td><strong>Dymethacrylates, adhesive monomers, fillers, initiators/stabilizers</strong></td>
<td>Paste/paste dual syringe with a mixing tip</td>
<td>Automix cement. Apply, self-cure (5 min) and light-cure (40s)</td>
</tr>
<tr>
<td>(Ivoclar-Vivadent)</td>
<td>Batch n°:j22739 pH: 4.2</td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Max-Cem</strong></td>
<td><strong>Base:</strong> Uretanedymethacrylate, Camphoroquinone, Fluoroaluminosilicate, others. <strong>Catalyst:</strong> Bis-GMA, Triethyleneglycoldimethacrylates, Glycerophosphatedimethacrylates, Bariumaluminopolosilicate glass, Others</td>
<td>Paste/paste dual syringe with a mixing tip</td>
<td>Automix cement. Apply, self-cure (5 min) and light-cure (40s)</td>
</tr>
<tr>
<td>(Kerr Dental)</td>
<td>Batch n°:</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>pH: 2.2</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Results

Mean push-out bond strengths (SD) and failure modes of the tested cements are displayed in Table 2. Differences in bond strengths exist among the self-adhesive cements used for luting fiber posts (p<0.05).

RelyX Unicem exhibited significantly higher bond strengths than the other tested cements. Push-out values of Multilink Sprint were lower than those of RelyX Unicem but higher than Max-Cem that recorded the worst push-out bond strength values.

The failure modes recorded were mostly adhesives in nature both between dentin and cement and at the post/cement interface. Cohesive failures were only observed for RelyX Unicem. Mixed failures also occurred in the three self-adhesive cements investigated. No cohesive failure within the fiber post were observed in the present study.

Table 2. Push-out strengths and the percentage of slices with their respective failure modes. Numbers are means (MPa), values in brackets are standard deviations. Different letters show statistically significant differences (p<0.05). AD: adhesive failures between dentin and luting agent; AP: adhesive failures between post and cement; C: cohesive failures within the post; M: mixed failures.

<table>
<thead>
<tr>
<th>Experimental groups</th>
<th>Sample size</th>
<th>Failure mode</th>
<th>Mean (SD)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>AD</td>
<td>AP</td>
</tr>
<tr>
<td>Rely X Unicem</td>
<td>32</td>
<td>69%</td>
<td>19%</td>
</tr>
<tr>
<td>Multilink Sprint</td>
<td>31</td>
<td>42%</td>
<td>29%</td>
</tr>
<tr>
<td>Max-Cem</td>
<td>30</td>
<td>33%</td>
<td>57%</td>
</tr>
</tbody>
</table>
Discussion

The results of this study require the rejection of the null hypothesis since differences in push-out bond strength exist between the tested self-adhesive cements.

The bonding mechanism of self-adhesive cements rely on chemical interactions and micromechanical retentions with the bonding substrate, but concerns still exist on the effective adhesive potential of these simplified cement.

In the present study, RelyX Unicem attained the higher bond strength values when compared to the other materials. In line with previous studies, these values may be comparable to those of multi-step luting agents (Radovic et al, 2008) (Bitter et al, 2006). When compared to another self-adhesive materials, RelyX Unicem registered the higher bond strength (Zicari et al, 2008). The use of the cement/post combination as recommended by the manufacturers, may have counted for the results obtained (Radovic et al, 2008) (Kececi et al, 2008). In the present study, only one type of fiber post was used (RelyX Fiber Post, 3M ESPE) as the main purpose was to estimate the bond strengths of self-adhesive cements. Differences in the application mode may have similarly influenced the results. RelyX Unicem utilized an elongation tip during its insertion into root canal resulting in inferior chance of bubbles formation and air-entrapment that would lead to an improvement in the marginal adaptation of the material both to the dental substrate and to the fiber post (Watzke et al, 2008). Simonetti and colleagues, addressed the higher sealing ability of RelyX Unicem dispensed with the elongation tip in comparisons with
Max-Cem and others multi-step cements that were inserted into root canal with a lentulo spiral or directly applied on the post surface (Simonetti et al, 2008). The two self-adhesive cements employed in the present study have a paste-to-paste composition. Base and catalyst are mixed together through an auto-mixing tip on a glass pad and the material was then inserted into the root canals with lentulo spirals. This method appeared less feasible as could increase the risk of air entrapment causing the formation of voids and interfacial defects that expedite premature failure in presence of cyclic stresses.

Max-Cem attained the worse push-out values (Table 2). The cement layer appeared inhomogeneous with frank voids and bubbles incorporated into the bulk (Fig. 1). Max-Cem is considered extremely technical-sensitive and any errors occurring during the mixing process can be determinant for its physical and mechanical properties (Behr et al, 2008). A prevalence of adhesive decementation at the cement/post side were recorded. Max-Cem possess an acidic pH that is maintained high even after 48 hours from its application. This may exert a detrimental effect on its physical properties, diminishing the possibility to establish effective micro-mechanical retentions. Moreover, the presence of an acidic layer on the post surface may jeopardize the formation of hydrogen bonds between the cement and fiber post, and the bonding potential of the cement itself result limited (Wrbas et al, 2007).

Multilink Sprint bond strengths were inferior than those of RelyX Unicem, but higher than those of Max-Cem. The material was able to partially demineralise the dental substrate, although discrepancies between the degree of demineralization and depth of resin penetration was assessed by light microscopy (Monticelli et al, 2008). At the dentin site bond, the
material appeared porous, probably due to an incomplete polymerization reaction (Fig. 2). The presence of residual acidic monomers at the bottom of the adhesive interface may represent weak areas as they can retain their etching potential jeopardizing adhesion (Mazzitelli et al, 2008) (Spencer and Swafford, 1999). These areas and the presence of collagen fibrils at the adhesive joint would undergo to premature degradation hence limiting the bonding potential of the material and reducing the service life of the restoration (Spencer and Swafford, 1999) (Wang and Spencer, 2005). Further studies should be performed to assess the longevity of these self-adhesive cements.

Nowadays, many studies are performed using ageing tests to assess the longevity of bonded interfaces. Several authors found that thermocycling may increase the retentive strength especially for RelyX Unicem. The thermal changes were supposed to promote a complete chemical polymerization enhancing its bonding potential (Bitter et al, 2006) (Reich et al, 2005). Self-adhesive cements work as dual-cure materials, where the chemical polymerization can be completed by light irradiation (Radovic et al, 2008b). Anyway, doubts exist on the degree of monomer conversion of the simplified cements. Some authors attained inferior bond strengths and decreased mechanical properties when RelyX Unicem was only auto-cured (Vrochari et al, 2009) (Piwowarczyk et al, 2003) (Kumbuloglu et al, 2004). No differences in the degree of monomer conversion were found between RelyX Unicem and Multilink Sprint, whereas Max-Cem attained the lower values (Vrochari et al, 2009). Several factors may count for the differences recorded, first of all the diverse chemical formulations. However, few information are furnished by
manufacturers and more specific details are highly desirable in order to define their characteristics and bonding behaviour.

**Fig. 1.** Representative SEM image of Max-Cem after the push-out test (45x). Voids are detected within the cement bulk, possibly due to air-entrapment during the mixing and insertion procedures.

**Fig. 2.** SEM microphotographs of Multilink Sprint (20 bar, original magnification: 350X). A detachment of the fillers from the resinous matrix showing a porous appearance was assessed when the material was submitted to shear forces.
References


caused by mixing errors: the therapeutic range of different cement types. Dent Mater 2008


5.3 Effect of thermocycling on the bond strength of self-adhesive cements to fiber posts

Introduction

Fiber posts are routinely used in dental practice for anchoring and reinforce the prosthodontic restorations. Due to their passive retention into root canal, the dislocation resistance of fiber posts is mainly ascribed to the luting agents and cementation techniques (Bitter et al, 2006) (D’Arcangelo et al, 2008). Resin-based luting materials are preferred for fiber post cementation, as increase in post retention and higher fracture resistance would be expected when compared to conventional cements (Dietchsi et al, 2008) (Rosenstiel et al, 1998).

Self-adhesive cements represent a subgroup of resin cements and are characterized by one-step, simple and standardized adhesive procedures. These one-step cements do not require pre-conditioning of the post-space walls. Due to the variability of the substrate, bonding to intra-radicular dentin has been considered a challenge. Simplifying luting procedures would be helpful in overcoming some technical problems observed with multi-step cements systems (Radovic et al, 2008), such as the difficult control of moisture and/or the chemical incompatibility between simplified adhesives and dual-cured methacrylate-based resin cements (Carilho et al, 2004) (Chersoni et al, 2005) (Pfeifer et al, 2003) (Tay et al, 2003).

Although high bond strength values are immediately desirable, the cement should possess long-term satisfactory performances in order to be
clinically recommendable. The mouth is a complex and heterogeneous environment, where a multitude of factors may simultaneously participate to stress the adhesive interfaces and interfere with the longevity of the restoration (Li et al, 2002). Oral conditions can be simulated in laboratory enabling for a better understanding of the dental biomaterials properties and possibly allow to predict the durability of the restorations (Breschi et al, 2008) (Goracci et al, 2007). Thermocycling test is conventionally used to simulate the thermal changes occurring in the oral cavity during eating, drinking or breathing that may concur in stressing the adhesive interfaces (Breschi et al, 2008) (Gale and Darvell, 1999) (Titley et al, 2003).

The bond strength of dental materials may be better assessed with the push-out test. Push-out test has been considered appropriate for measuring the bond strength inside the root canal, as the shear movements are created parallel to the cement/dentin and cement/post interfaces and seem to procure similar stresses to those occurring in clinical conditions (Goracci et al, 2004) (Soares et al, 2008).

The present laboratory study was conducted to evaluate the effect of thermocycling on the bond strength of translucent glass fiber posts luted with different self-adhesive resin cements. The null hypothesis tested was that thermocycling does not affect the bond strength of the three self-adhesive resin cements when luting fiber posts into root canals.

**Materials and Methods**

*Specimen preparation*

Thirty-six extracted, single-rooted human premolars stored in 0.5% Chloramine T solution at 4°C were collected after the informed consent of
the donors was obtained. Exclusion criteria were presence of caries, cracks or resorptions on the root. The crown of each tooth was removed 1 mm above the CEJ by means of a slow-speed diamond saw (Isomet, Buehler, Lake Bluff, IL, USA) under copious water cooling. Working length was established at 1 mm from the root apex. Cleaning and shaping of the root canal were performed with Protaper Ni-Ti rotatory instruments (size S1, S2, S3; Dentsply Maillefer, Ballagues, Switzerland) following the crown-down technique. Irrigations with 5% sodium hypochlorite were performed between the instrumentations. Root canal was filled with gutta-percha cones (Coltène/Whaledent, Langenau, Germany) cemented with a resin sealer (AH Plus Jet, Dentsply DeTrey, Konstanz, Germany) referring to the lateral condensation technique. The canal access was sealed with a temporary restorative material (Fuji VII, GC Corporation, Tokyo, Japan; batch n° 0410221) and stored in a laboratory incubator (100% relative humidity; 37° C). After 24 hours, the coronal seal was abraded by means of #240 SiC paper under water cooling, and the gutta-percha was removed with a pre-shaping drill (Dentsply DeTrey), leaving a 5 mm-long apical seal. A 7 mm-deep post-space was prepared with a universal drill (3M ESPE, Seefeld, Germany) to match the size of the co-respective epoxy resin-based RelyX Fiber Post (#1; 3M ESPE; Lot: 02363200603). The post was cut with a bur mounted on a water-coolant handpiece to the selected coronal length. Three self-adhesive materials were employed for fiber post cementation (n=12): 1) RelyX Unicem (3M ESPE); 2) G-Cem (GC Corp, Tokyo, Japan); 3) Breeze (Pentron Clinical Technologies, Wallingford, CT, USA).
Each material was handled according to manufacturer’s instructions. Application modes, chemical composition and batch numbers of the materials investigated are presented in Table 1.

Self-adhesive cements polymerized in a dual-cure mode. Immediately, 2 s of light curing allowed to remove the extruding cement with a spatula. After the first 5 min of auto-cure, during which the post was seated to full depth in the prepared spaces using finger pressure, additional 40 s of light polymerization through the top of the translucent fiber post were performed (Astralis 7, Ivoclar-Vivadent, Schaan, Liechtenstein; output: 500 mW/cm²). A core build-up was performed with a glass-ionomer cement (Fuji IX; GC corp; Batch n°: 0703071). All bonded specimens were stored for 1 month in a flower sponge slightly wetted with deionised water in a laboratory stove at 37°C and relative humidity.

Prior to push-out test, half of the specimens (n=18) were additionally thermocycled for 5,000 cycles in deionized water from 5 to 50 °C. The dwell time at each temperature was 30s in each bath; the transport time between the water baths was 2s.
Table 1. Chemical composition, application modality and manufacturers instruction of the tested materials.

<table>
<thead>
<tr>
<th>Material</th>
<th>Composition</th>
<th>Delivery system</th>
<th>Instruction for use</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rely X Unicem</td>
<td>Powder: glass fillers, silica, calcium hydroxide, self-curing initiators, pigments, light-curing initiators, substituted pyrimidine, peroxyl compound. Liquid: methacrylated phosphoric esters, dimethacrylates, acetate, stabilizers, self-curing initiators, light-curing initiators</td>
<td>Capsule and Aplicap Elongation Tip</td>
<td>Mix cement. Apply, self-cure (5 min) and light-cure (40s)</td>
</tr>
<tr>
<td>(3M ESPE,)</td>
<td>Batch n°:270644</td>
<td></td>
<td></td>
</tr>
<tr>
<td>G-Cem</td>
<td>UDMA; phosphoric acid ester monomer; 4-META; water; dimethacrylates; silica powder; initiators/stabilizers; fluoro-amino-silicate glass</td>
<td>Capsule</td>
<td>Mix cement. Apply, self-cure (5 min) and light-cure (40s)</td>
</tr>
<tr>
<td>(GC corp.)</td>
<td>Batch n°:0707051</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Breeze</td>
<td>Mixture of BisGMA, UDMA, TEGDMA, HEMA, 4-META resins, silane-treated bariumborosilicate glasses, silica with initiators, stabilizers and UV absorber, organic and/or inorganic pigments, opacifiers.</td>
<td>Paste/paste dual syringe with a mixing tip</td>
<td>Automix cement. Apply, self-cure (5 min) and light-cure (40s)</td>
</tr>
<tr>
<td>(Pentron Clinical Technologies)</td>
<td>Batch n°:161936</td>
<td></td>
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</tr>
</tbody>
</table>

**Push-out bond strength test**

The portion of each root containing the fiber post was sectioned into four to six 1 mm-thick slices with a diamond saw (Isomet; thickness: 0.1 mm) under water cooling. The cylindrical plunger of the testing machine (Triax 50, Controls S.P.A, Milan, Italy) was forced to dislodge, via an
apical-coronal direction, each inverted, truncated fiber post from the root dentin. A load (cross-head speed: 0.5 mm/min until failure) was then applied to the post surface that resulted in shear stresses along the cement/dentin – cement/post interfaces. The retentive strength of the post fragment (MPa) was calculated by dividing the load at failure (Newton) by the interfacial area of the post segment ($S_L$). The formula used for measuring the tronco-conical area as follows:

$$S_L = \pi (R+r) \left\{ h^2 + (R-r)^2 \right\}^{0.5}$$

In which $\pi$ was equal to 3.14, $R$ and $r$ were the coronal and the apical post radius respectively, and $h$ the root slice thickness. The diameters of the post and the thickness of the slice were individually measured using a digital caliper with 0.01 mm accuracy.

Failure modes were evaluated by a single operator under a stereomicroscope (Olympus SZ-CTV, Olympus, Tokyo, Japan) at 40x magnification (Fig. 1) and classified as cohesive (within the cement, C), adhesive (between the post and the cement, AP, or at the cement/root dentin level, AD) or mixed (adhesive and cohesive fractures occurred simultaneously, M) (Fig. 1).

**Statistical analysis**

The normal and equal distributions of the push-out bond strength data were first checked and verified by the Kolmogorov-Smirnov and Levene’s test respectively.

A 2-way ANOVA was executed to determine the effect of the type of cement, thermocycling and interactions (p<0.05). Mean bond strengths of the three cements were analyzed with the Tukey test for post-hoc multiple comparisons (p<0.05). Calculations were handled by the SPSS 15.0 software (SPSS Inc.; Chicago, IL, USA).
Results

Bond strength was significantly influenced by the luting material (F=14.640; p=0.00) and thermocycling (F=18.205; p=0.00), interactions were also significant (F=3.836; p=0.01). Mean push-out bond strengths (SD) of the tested cements (MPa) prior and after thermal cycling are shown in Table 2.

Initially, push-out bond strengths of RelyX Unicem and Breeze were statistically comparable and higher than those exhibited by G-Cem. Thermocycling did not affect the bond strengths of RelyX Unicem and Breeze. After the thermal challenge, increased push-out values were registered for G-Cem when compared to the initial group and no differences were then found among the tested groups.

The most frequently recorded modes of failure were adhesives at the cement/dentin interfaces (Table 3). Debonded specimens between cement and fiber posts were also recorded both in the initial and in the thermocycled groups (Fig. 1). Cohesive failures within the cement were only registered for RelyX Unicem prior to thermocycling. Mixed failures were observed for RelyX Unicem and G-Cem before being thermally challenged.
Table 2. Mean bond strength (SD) values (MPa) and post-hoc comparisons results.

<table>
<thead>
<tr>
<th>Material</th>
<th>Initial</th>
<th>TC</th>
</tr>
</thead>
<tbody>
<tr>
<td>RelyX Unicem</td>
<td>10.27(2.18) A</td>
<td>13.66(8.23) A</td>
</tr>
<tr>
<td>G-Cem</td>
<td>6.78(2.57) B</td>
<td>10.0(4.34) A*</td>
</tr>
<tr>
<td>Breeze</td>
<td>10.31(4.60) A</td>
<td>9.92(6.33) A</td>
</tr>
</tbody>
</table>

Different capital letters in columns and asterisks in rows indicate differences (p<0.05). TC: thermocycling.

Table 3. Percentage of failure registered in each experimental group.

<table>
<thead>
<tr>
<th>Experimental groups</th>
<th>Initial</th>
<th>TC</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>AD</td>
<td>AP</td>
</tr>
<tr>
<td>RelyX Unicem</td>
<td>69%</td>
<td>19%</td>
</tr>
<tr>
<td>G-Cem</td>
<td>61%</td>
<td>15%</td>
</tr>
<tr>
<td>Breeze</td>
<td>54%</td>
<td>46%</td>
</tr>
</tbody>
</table>

AD: adhesive failure at the dentin interface; AP: adhesive failure between cement and post; C: cohesive failure within the cement; M: mixed failure (a combination of the above mentioned modes). TC: thermocycled group.
Discussion

The null hypothesis has to be rejected as bond strengths were different for the tested cements and thermocycling did affect these values.

Self-adhesive cements were introduced to simplify the luting procedures of indirect restorations and shorten chair time. One clinical study, reported that RelyX Unicem worked as efficiently as a zinc phosphate cement during a period of 138 days (Behr et al, 2008). However, only few investigations dealt with the resistance of simplified cements to thermal stresses, and contrasting results were noticed.

It was speculated that the thermal stress occurring during the laboratory test would enhance the chemical polymerization of the materials, promoting their complete setting reactions (Bitter et al, 2006) (Piwowarczyk et al, 2006) (Reich et al, 2005). In the present study, no differences in push-out values were encountered for RelyX Unicem and Breeze before or after being thermally challenged. An increase in bond strength was previously reported for RelyX Unicem when used for luting fiber posts or ceramic restorations (Piwowarczyk et al, 2006) (Reich et al, 2005). Contrary to these investigations, in the present study the roots were not embedded in acrylic resins and no isolation was created around the bonded interfaces. The direct exposure of the root to the different temperatures may have promoted weak adhesive-dentin interfaces counting for the differences registered between the investigations. Some questions arise on the degree of monomer conversion of self-adhesive cements when used in an auto-cure mode, an incomplete polymerization is expected and it will jeopardize bond strength (Kumbuloglu et al, 2004) (Vrochari et al, 2009). If so, dual-curing self-adhesive cements have
become imperative for achieving reliable bonding. Future studies are warranted to deeply investigate on this topic.

Scarce information are present in literature regarding the bonding ability of Breeze as fiber post cementing agent. When compared to another self-adhesive cement and a glass-ionomer cement Breeze showed the highest dislodgement force after storage in water for 30 days (Elsayed et al, 2009). Breeze is composed by a mixture of hydrophilic components (i.e. BisGMA and HEMA) and hydrophobic monomers (i.e. TEGDMA). The latter molecule would furnish the material hydrophobic characteristics necessary to withstand to the moisture condition of dentin and prevent an excessive water sorption that would jeopardize the polymerization reaction of the material. However, further studies are required to well define the bonding performance of Breeze.

G-Cem have previously showed the worse push-out values when used for luting fiber posts in comparison to RelyX Unicem and multi-step resin cements (Zicari et al, 2008). Although the auto-adhesion mechanism of G-Cem follows a glass-ionomer technology (G-Cem technical information, GC corp.), its high viscosity has been responsible of the scarce interaction with the adhesive substrates (Zicari et al, 2008). However, bond strength of G-Cem increased after thermal challenging. Thermal changes will cause expansion/contraction stresses within the material (El Araby and Talic, 2007) (Titley et al, 2003), what may affect the adhesive stability; but the cement expansion will also create frictions along the root canals that are thought to improve its mechanical retention (Cury et al, 2006). After setting, G-Cem showed a porous appearance at the adhesive interface (Cantoro et al, 2009). The pores and bubbles would function as stresses-absorbers that would prevent the premature
degradation of the adhesive interfaces (Monticelli et al, 2007). It remains to be proved if prolonged expansion/contraction phenomena would result in crack formations within the cement bulk hence diminishing the mechanical properties of the material and if the pores present into the material would resist to prolonged cycles and mechanical occlusal loading and their resistance to degradation when submitted to chemical solutions (i.e. sodium hypochlorite).

Although in vivo studies are the ultimate testing stages, laboratory tests and aging simulations are important sources for reproducing intra-oral conditions (Alani and Toh, 1997) (Amaral et al, 2007) (Goracci et al, 2004). Thermocycling test is conventionally used to simulate the thermal changes and water exposure that may occur in the oral cavity during eating, drinking or even breathing (Gale and Darvell, 1999). The ISO TR 11450 (ISO reports) reports that 500 thermocycles in water (5 and 55°C), is an appropriate method to test thermal stability of a dental material. To date, concerns still arise on the ability of the test to simulate intra-oral ageing. In the present study, specimens were thermocycled for 5,000 times, although increasing the number of cycles would be desirable to evaluate eventually pronounced differences in a longer period of time.

The importance of using the cement/post combination recommended by each manufacturer has been previously highlighted (Kececi et al, 2008) (Radovic et al, 2008). Glass-, quartz-, zirconium- and/or titanium- posts differ in terms of structure, composition and mechanical properties that can influence their bonding mechanism as well as affect their responses to chemo/mechanical treatments. In the present investigation, only one type of post was used, that it is made of glass fibers (60-70 vol%) embedded in an epoxy resin (RelyX Fiber Post technical pamphlet, 3M ESPE).
Although no differences were found between cements after thermocycling, the bonding performance of self-adhesive cements to others fiber posts differently branded should also be evaluated.

The most frequently registered mode of failures were adhesives between cement and dentin, followed by adhesive at the cement/post interface both prior and after thermocycling (Table 4). The self-adhesive cement-dentin joint represents the weaker point of the one-step cements. Although self-adhesive cements do not require any pre-treatment of the bonding substrate, their retention was increased once the post-space cavity was roughened, resulting in micro-mechanical grooves formations were the cement could flow and establish an improved bonding (Balbosh et al, 2005). Doubts also exist on the efficacy of pre-treating fiber posts in the attempt of increase the retention of the auto-adhesive cements/fiber post bonds, such as with silane agents (Bitter et al, 2007). Due to the limited chemical interaction established between self-adhesive cements and fiber posts, chemo-mechanical post surface treatments could be proposed to increase the surface area available for bonding and enhance micro-retentions (Wrbas et al, 2007).

In general, bond strength of dual-cure self-adhesive luting agents is not compromised by physiologic thermal stresses. A combination of chemical adhesion and mechanical retention seemed to characterize the adhesion mechanism of these simplified cements although further studies are desirable to define the overall bonding process of the different self-adhesive luting agents.
References


Chapter 6

6.1 Post surface treatments for improving the adhesive bonds

Although the adhesion into root canal may represent the weakest point of the restoration (Ferrari et al, 2000a) (Ferrari et al, 2002), decementation at the cement/post level may compromise the final outcome (Ferrari et al, 200b) (Monticelli et al, 2003). The occurrence of post debondings does not have to be considered an irreversible failure, since an advantage of using fiber post is that they can easily be replaced. However, this event would be time-consuming and not always accepted by the patients.

The search for improved adhesions of resin cements to fiber post has ever forced investigators to experiment new methodologies that can enhance the resin cement/fiber post interfacial strength. These consist in different chemical and mechanical treatments (Goracci et al, 2005) (Monticelli et al, 2006a) (Monticelli et al, 2006b) (Sahafi et al, 2004) intended to modify the post matrix composition and morphology as well as to increase the surface energy of dental posts (Asmussen et al, 2005) (Le Bell et al, 2004). Although silane application has been recommended for enhancing adhesion (Aksornmuang et al, 2004) (Perdigao et al, 2006) thank to the chemical bridges it can establish, others chemo/mechanical post surface treatments have been proposed in order to strengthen the bonds. These conditioning approaches, can be performed solely (i.e. silane application) or in combinations (i.e. sandblasting plus silane application) (Magni et al, 2007). These treatments are efficient for improving the union between methacrylate resin based cements and epoxy-resin based fiber posts that are chemically incompatible. The rationale rely on the removal/dissolution of the superficial and inter-fiber epoxy-matrix, on the
exposure of the underlying glass fibers that can be then activated with silane or adhesive solutions. However, attention should be paid to the methodology adopted for conditioning fiber post: an excessive diameter of air-bone particle, a limited distance between the sandblasting device and the post the high pressure are all possible factors that may damage the fiber posts (Monticelli et al, 2008) (Sahafi et al, 2004). Methods already adopted for treating ceramic crowns were applied to fiber post as well, such as hydrofluoric acid. However, this treatment was too aggressive for the post fibers thus affecting the post’s integrity (Vano et al, 2006). Others laboratory and industrial techniques have been proposed over the years to improve the bonding potential of fiber posts (Monticelli et al, 2006a) (Monticelli et al, 2006b).

In the following trials it was of interest to examine the effects that different post conditioning approach could have on its topographic appearance as well as to distinguish the treatments that could be proposed in order to obtain reliable and safer bonds. It was also taken into consideration whether and to what extent the bonding potential of self-adhesive cements would be reinforced after the post surface treatments. Different in vitro studies were presented: in the first, two combined microscopic methodologies (confocal microscopy and atomic force microscopy) were used to analyze and determine the post surface roughness after the different conditioning procedures and whether these treatments were able to dissolve the incompatible epoxy-matrix without damaging the glass fibers. In the second study, a push-out test and a scanning electron microscopy evaluation were performed to assess the retentive strength of self-adhesive resin cements to pre-treated epoxy-resin based fiber posts.
References


6.2 Surface roughness analysis of fiber post conditioning processes.

Introduction

Considerable attention has been paid to the clinical application of fiber posts. Reliable bonding can be achieved when post, luting material and dentin achieve good adhesion, thus forming a “mono-block” unit (Schwartz and Robbins, 2004). Most of the studies of fiber posts bonded to radicular dentin have stated that the majority of failures occurred between the post and the cement (Baldissara et al, 2006) (Perdigao et al, 2006). Recent investigations have been focused on improving this adhesive interface in attempt to enhance the durability of final restorations (Valandro et al, 2006).

The benefit of applying silane-coupling agents as adhesion promoters has been reported (Aksornmuang et al, 2004) (Aksornmuang et al, 2006) (Goracci et al, 2006). However, the post/composite joint still remains relatively weak. Coupling of conventional epoxy-resin-based fiber posts to dental composites is hampered by the absence of chemical union between the epoxy resinous matrix and methacrylate-based resins (Monticelli et al, 2006a).

Different post surface treatments may improve adhesion of posts to composite resins (Balbosh and Kern, 2006) (Monticelli et al, 2006a). These are chemical and mechanical treatments intended to roughen the post surface, generating mechanical interlocks between the post and resin
cements. They may include the use of etching solutions as well as physical roughen procedures, such as sandblasting (Balbosh and Kern, 2006) (Monticelli et al, 2006a) (Monticelli et al, 2006b).

Previously, conditioned surfaces have been analyzed by scanning electron microscope (SEM) (Monticelli et al, 2006b) (Vano et al, 2006). However, atomic force microscopy (AFM) may represent an alternative methodology with some additional advantages. It can works in air, requires little or no sample preparation, and provide high-resolution imaging of 3-D surface topography (Marshall et al, 1995). A confocal imaging profiler is routinely applied to analyze surface texture, measuring the actual profile and standard numerical roughness parameters that can be calculated from the profile itself. It has been widely used for assessing the surface topography of dental implants (Hallgren et al, 2001).

Therefore, the aim of this study was to evaluate the surface topography and changes in average roughness (Ra) provided by different fiber post surface treatments through the combined use of atomic force (AFM) and confocal microscopy. The null hypothesis tested was that different surface treatments would neither modify the post’s surface morphology nor affect its individual components (fiber/matrix).

Materials and Methods

Forty translucent quartz fiber posts #3 with a maximum diameter of 2.14 mm (DT Light Post, batch #120US0401A; RTD, St Egrève, France) were used for the study. The posts are made of unidirectional pre-tensed quartz fibers (60% vol) bound in an epoxy resin matrix (40% vol). Posts were divided into 8 groups (n=5) according to the surface pre-treatment performed: Group 1, no treatment; Group 2, 10% hydrogen peroxide
(H₂O₂) for 20 min; Group 3, 30% hydrogen peroxide (H₂O₂) for 10 min; Group 4, 21% sodium chloride (NaOCH₂CH₂) for 20 min; Group 5, etching with potassium permanganate (KM₂O₄); Group 6, etching with 4% hydrofluoric acid for 1 min; Group 7, sandblasting; Group 8, silicate/silane coating (DT Light SL post, batch #05/65; VDW GmbH, Munich, Germany).

Posts from Groups 2 and 3 were immersed in hydrogen peroxide solutions (for 20 and 10 min, respectively) (Panreac Quimica SR, Barcelona, Spain) at room temperature (RT) and rinsed with de-ionized water (3 min). Fiber posts in Group 4 were etched with 21 wt% sodium ethoxide solution (Sigma-Aldrich Chem., GmbH, Steinheim, Germany) in ethanol (20 min) at RT, rinsed with pure ethanol and 50% ethanol in de-ionized water, and finally in de-ionized water to reach a stable pH of 7 (5 min for each cleaning bath).

The etching for Group 5 was performed in three consecutive steps: 1) immersion in a conditioning solution (60 vol% of methyl-pyrrolidone in de-ionized water) for 3 min at 50-60°C (E-K Hole Cleaner, Elkem, Torino, Italy); 2) etching in an alkaline potassium permanganate solution (20 vol% in de-ionized water, pH 12-13) (E-K Hole Oxidizer, Elkem) for 10 min at 70-80°C; and 3) immersion in a neutralizing bath containing dilute sulphuric acid (10 vol% in de-ionized water) (E-K Hole Reducer, Elkem) for 5 min at 40-50°C to reduce and neutralize the excess permanganate and clean the post surface (Monticelli et al, 2006b).

Posts in Groups 6 were immersed in 4% hydrofluoric acid solution (Panreac Quimica SR) for 1 min at RT and then extensively rinsed with de-ionized water. Samples of Group 7 were sandblasted (Rocatec Pre, 3M ESPE, Seefeld, Germany) for 5 s at 2.8 bar. The tip of the sandblasting
device was held perpendicularly to the post at a distance of 1 cm. During the procedure, the post was rotated so that the aluminium oxide particles (110 um) would be blasted on the entire surface. Posts in Group 8 had already been coated with silicate and silane by the manufacturer; a patented protective layer ensured that the superficial coating was not contaminated or deactivated.

Each fiber post was then cut longitudinally with a slow-speed diamond saw under water cooling (Isomet 4000; Buheler, Lake Bluff, IL, USA), so that the post was divided into two equal halves. Posts of Groups 1 and 7 were ultrasonically cleaned in de-ionized water (10 min), rinsed in 96% ethanol, and dried with an oil-free air stream. Posts from Group 8 were gently air-dried, to avoid any possible alteration of the coating.

**Confocal microscopy**

One half of each treated fiber post was evaluated under a confocal imaging profiler (Eclipse L150 Sensofar, Nikon, Tokyo, Japan), with a X50/0.80 numerical aperture and an extra-long working distance dry objective (Nikon), for the collection of reference images of the post surface. Images were captured by a CCD camera (Nikon) and reconstructed with a computer software program (plµ Confocal Imaging Profiler). Average surface roughness (Ra) recorded for each treated post (10 measurements each experimental group) was quantitatively expressed as a numerical value (in microns) and a graph of the profile.

**Atomic Force Microscope (AFM) evaluation**

The second half of each sample was evaluate by atomic force microscopy (AFM, Multimode Nanoscope IIIa, Digital Instruments,
Veeco Metrology group, Santa Barbara, CA, USA). Images were taken in air. The tapping mode was performed with a 1-10 Ohm-Cm phosphorous-doped (n) Si tip (at 50µ). Changes in vertical position provided the height of the images, registered as bright and dark regions. The tip sample “tap” was maintained stable through constant oscillation amplitude (set-point amplitude). Fields of view at 50X50 µm scan size were considered for each post at a data scale of 1504 µ and recorded with a slow scan rate (0.1 Hz). A single operator analyzed the average surface roughness (Ra) of the matrix/quartz fiber post after different surface treatments, expressing it as a numeric value (in nanometers) with specific software (Nanoscope V530R35R). Five measurements were performed for each pre-treated post, on both the epoxy matrix and quartz fibers, with a standardized rectangular spot (1.56X1.37 µm). Regarding the DT Light SL post, it was not possible to measure fiber roughness, since they were completely covered by the superficial coating.

**Statistical analysis**

Surface roughness data and matrix/fiber average roughness values were statistically analyzed with one-way Analysis of Variance. The Student-Newman-Keuls test was used for post hoc comparisons. The level of significance was set at p<0.05.

**Results**

Post surface average roughness (Ra) resulting from digital images recorded by confocal microscopy (Figs. 1A-1H) revealed that chemomechanical conditioning treatments significantly modified surface roughness (p<0.001) (Table 1). Etching with HF, sandblasting and
potassium permanganate and sodium ethoxide treatments resulted in a significant improvement in (Ra). 3-D cnfocal profiler image revealed a variation in post surface topography with micro retentive space formation and fiber exposure.

Changes in average roughness (Ra) of the epoxy resin matrix were recorded by AFM analysis after all post surface pre-treatments (Table 2). HF attained the highest Ra value (Fig. 2A). After treatment with potassium permanganate, the resulting matrix was smoother (Fig. 2B). In the DT Light SL Post, quartz fibers were enshrouded by the silicate/silane coating (Fig. 2C). A significant increase in roughness of the superficial quartz fiber after treatment with hydrofluoric acid was detected (Fig. 2A). Fibers appeared to be fracture-free, with no evident signs of degradation in the other experimental groups.

Table 1. Mean (SD) of surface roughness values recorded with a confocal image profiler after different post surface pre-treatments. Same alphabetical letters indicate groups that are statistically similar (p<0.05).

<table>
<thead>
<tr>
<th>Superficial pre-treatment</th>
<th>Mean Roughness (SD, µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrofluoric acid (4%)</td>
<td>3.929(1.14) A</td>
</tr>
<tr>
<td>Sandblasting (Rocatec Pre)</td>
<td>4.042(1.04) A</td>
</tr>
<tr>
<td>Potassium permanganate</td>
<td>3.210(0.73) AC</td>
</tr>
<tr>
<td>Sodium ethoxide</td>
<td>3.149(0.81) AC</td>
</tr>
<tr>
<td>Hydrogen peroxide (30%)</td>
<td>2.538(0.36) BC</td>
</tr>
<tr>
<td>Hydrogen peroxide (10%)</td>
<td>2.824(0.58) BC</td>
</tr>
<tr>
<td>Silicate/silane coating (DT Light SL Post)</td>
<td>2.539(0.43) BC</td>
</tr>
<tr>
<td>Control (no treatment)</td>
<td>2.207(0.56) BC</td>
</tr>
</tbody>
</table>
Fig 1. Confocal profiler 3D images of the post surface after different chemo-mechanical pre-treatments. A) Control (DT Light Post). B) 30% hydrogen peroxide. C) 10% hydrogen peroxide. D) Silicate/silane coating (DT Light SL Post). E) Hydrofluoric acid. F) Potassium permanganate. G) Sandblasting (Rocatec Pre). H) Sodium ethoxide. The differences in colors between yellow and red represent the “peak” and the “valley” of the surface. A partial removal of the external layer of epoxy resin after conditioning treatments determined the partial exposition of the fibers. The oxidative etching procedure exerted their function mainly via a dissolution process of the resin matrix, leaving the quartz fiber undamaged. A more aggressive approach was determined by the application of HF. DT Light SL Posts were completely smooth, due to the superficial coating responsible for their chemical adherence to resin composites.
Table 2. Mean (SD) average surface roughness values (Ra) of the resinous matrix and the fibers recorded under AFM after different post-surface pre-treatments. Same alphabetical letters indicate groups that are statistically similar (p<0.05).

<table>
<thead>
<tr>
<th>Superficial pre-treatment</th>
<th>Matrix average roughness (SD, nm)</th>
<th>Fiber average roughness (SD, nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrofluoric acid (4%)</td>
<td>75.15(14.3) A</td>
<td>58.15(11.5) A</td>
</tr>
<tr>
<td>Sandblasting (Rocatec Pre)</td>
<td>36.03(14.81) B</td>
<td>16.68(12.9) B</td>
</tr>
<tr>
<td>Hydrogen peroxide (30%)</td>
<td>30.66(7.96) B</td>
<td>9.09(1.7) B</td>
</tr>
<tr>
<td>Sodium ethoxide</td>
<td>37.32(15.3) B</td>
<td>12.09(2.8) B</td>
</tr>
<tr>
<td>Hydrogen peroxide (10%)</td>
<td>28.23(1.6) B</td>
<td>8.31(5.2) B</td>
</tr>
<tr>
<td>Control (no treatment)</td>
<td>23.3(14.3) BC</td>
<td>4.86(1.9) B</td>
</tr>
<tr>
<td>Potassium permanganate</td>
<td>13.64(7.9) CD</td>
<td>6.3(3.7) B</td>
</tr>
<tr>
<td>Silicate/silane coating (DT Light SL Post)</td>
<td>6.61(2.3) D</td>
<td>---</td>
</tr>
</tbody>
</table>
Fig. 2. AFM images of treated quartz fiber post (50 x 50 µ). The squares (1.56 x 1.37 µm) show the area used for roughness measurements at the resin matrix (grey) and the fiber (white). (A) Hydrofluoric acid. The etching procedure was able to dissolve the resin matrix, but the effects were too corrosive, with exposed quartz fibers resulting in superficial blister formations; (B) Potassium permanganate. An intact fiber with no sign of damage is evident, and the matrix appeared smoother than with other treatments, most likely as a consequence of the resin-swelling step that preceded the etching procedure. (C) Silicate/silane industrial coating (DT Light SL Post). No quartz fibers are exposed on the post surface.
Discussion

The results of this study revealed an increase in fiber post surface roughness after chemo-mechanical conditioning. The etching procedures reacted mainly with the epoxy-resin matrix, most likely by dissolution. Thus, the null hypothesis was rejected.

Most of the study on fiber post morphology have been performed with scanning electron microscope (SEM), providing only qualitatively information (Monticelli et al, 2006b) (Vano et al, 2006).

AFM has been widely used to investigate the structural changes determined by etching procedures on enamel and dentin (Marshall et al, 1997) (Hegedüs et al, 1999) (Saeki et al, 2001) (Lippert et al, 2004), or for evaluating different biomaterials (Cross et al, 2005). Together with the use of a confocal imaging profiler, this represents an effective methodology for analyzing not only a curved surface, like that of fiber posts, but also its modification after conditioning with chemical of physical agents. The average surface roughness can be qualitatively determined and converted into a numerical reading of the surface topography (Marshall et al, 1997) (Hallgren et al, 2001). Moreover, AFM allowed for the quantification of treatment effectiveness on the post’s individual components (matrix and fibers), expressing it as a nanometric increase in roughness.

The concept of conditioning artificial substrates to enhance bonding has precedents in dentistry, e.g. the etching of Maryland bridges (Thompson et al, 1983) (Thompson et al, 1984) or feldspathic porcelain restorations (Horn, 1983).

The rationale for conditioning the fiber posts relies on the purpose of removing a surface layer of epoxy resin, rendering more quartz fibers
available for silanization, and improving the fiber post surface bonding area. The etching potential of the alkaline chemicals used depends on its capacity to partially dissolve the epoxy resin matrix through a mechanism of substrate oxidation (Baskin et al, 1979) (Kirman et al, 1998) (Brorson, 2001). The spaces between the fibers provide site for micro-mechanical retention of resin composites. Results of the present investigation confirmed the benefit of some chemo-mechanical treatments, also considering the previously reported bond strength results obtained at the fiber/composite interface (Monticelli et al, 2006b).

Sandblasting is commonly used for treating ceramic and composites, or as a part of the tribochemical silica-coating process (Saunders, 1990) (Borges et al, 2003). The efficacy of blasting zirconia and fiber posts with silica oxide (CoJet System, Praxair, Inc., Danbury, CT, USA) has been tested (Sahafi et al, 2003) (Sahafi et al, 2004a) (Sahafi et al, 2004b). Despite the satisfactory bond strengths achieved, the treatment was considered too aggressive for fiber posts, because of the risk of significantly modifying their shape and fit within the root canals (Sahafi et al, 2004a). In this study, a significant increase in post surface roughness has been recorded after sandblasting (Rocatec Pre, 3M ESPE). The treatment was effective on the resinous matrix. However, no apparent signs of deterioration of the post were detectable. Dimensions of the aluminum oxide particles, as well as the time of application and distance, may have influenced these results.

Hydrofluoric acid has recently been proposed for etching glass fiber posts (Vano et al, 2006). The acid is able to “activate” the post surface, allowing for the formation of micro-retentive spaces. However, the texture of the exposed quartz fibers was more irregular than with other treatments,
with blister formation. As a consequence of the extremely corrosive effect of hydrofluoric acid on the glass phase (Addison and Fleming, 2004) (Vano et al, 2006), the technique may produce substantial damages to fiber post substructure, especially when used with extended application time. Thus, its application is discouraged.

Currently, the dental market offers posts that have already been pre-coated with combined silicate/silane layers. No fibers are exposed on the surface of SL post, which appears relatively smooth. The adhesion mechanism is essentially based on the chemical interaction of the coating with the resin composite/luting cement. The industrial coating appears promising for simplifying the clinical procedures during post placement. Further investigations are needed to assess if post surface pre-treatments with chemical and/or mechanical agents can withstand longevity testing and influence the long-term clinical effectiveness.

In conclusion, atomic and confocal microscopy represents an effective method for evaluating fiber posts surface topography. Etching with potassium permanganate or sodium ethoxide increases surface roughness through partial removal of the epoxy resin matrix and improve the surface area available for adhesion by creating micro-retentive spaces. The choice of aggressive conditioning chemicals, such as hydrofluoric acid, should be avoided to prevent any damage to quartz fibers.
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Brorson SH. Deplasticizing or etching of epoxy sections with different concentration of sodium ethoxide to enhance the immunogold labeling. Micron 2001; 32: 101-105.


6.3 Effects of post surface treatments on bond strength of self-adhesive cements
Claudia Mazzitelli, Federica Papacchini, Francesca Monticelli, Manuel Toledano, Marco Ferrari. American Journal of Dentistry

Introduction

Fiber posts gained popularity over the years for the restoration of endodontically treated teeth with massive coronal destruction (Cagidiaco et al, 2007) (Ferrari et al, 2007). In the attempt to predict the clinical success of the post-restored teeth, great attention has been focused on the uniformity of the root dentin/cement/post joints that should form a “monoblock” unit (Schwartz and Robbins, 2004) (Monticelli et al, 2008a). However, decementations at the cement/post site bonds have frequently been observed, possibly compromising the longevity of the final restorations (Ferrari et al, 2007b) (Baldissarra et al, 2006). The variability of the dental substrate and the different chemical composition between most of the cement/post systems, make hard to establish a unique bonding mechanism into root canals (Perdigao et al, 2006) (Monticelli et al, 2006) (Aksornmuang et al, 2004) (Tay and Pashley, 2007).

Methacrylate resin-based luting agents showed good mechanical properties and are increasingly used for the cementation of fiber posts. Frequently, posts are made of fibers (i.e. glass, quartz) embedded in an epoxy resin matrix that is chemically incompatible with the methacrylate resin-based cements (Monticelli et al, 2008a). Chemo/mechanical post surface treatments have been proposed to enhance the adhesion with the resin cements through the removal/dissolution of the incompatible epoxy resin matrix and the exposure of the underneath fibers, that can be then
activated by silanization establishing more suitable bonds with the luting agent (Monticelli et al, 2008b) (Vano et al, 2006). An increase in the post surface roughness has recently been assessed after selected surface treatments; the micro-spaces created along with the post surface are intended to increase the surface are available for bonding and to facilitate the overall retentive strength of resin cements (Mazzitelli et al, 2008). Among the luting materials available for the cementation of fiber posts, self-adhesive resin composite cements are the least introduced category designed for ensuring simpler and standardized bonding procedures (Toman et al, 2009) (Bateman et al, 2005). Although their applications rely on a single clinical step, the chemo/mechanical post surface treatments on self-adhesive cements bonding performances have been investigated would ideally improve the self-adhesive cements bonding performances, as the establishment of stringer mechanical retentions would be desirable to reinforce the self-adhesive cement/fiber post joints (Hayashi et al, 2008) (Wrbas et al, 2007) (Bitter et al, 2007).

The purpose of this study was to determine the push-out bond strength of two self-adhesive resin cements to epoxy resin-based quartz fiber posts after different fiber conditioning treatments. The null hypothesis tested was that post surface treatments do not influence the bond strengths of self-adhesive resin cements.

**Materials and Methods**

Fifty single-rooted human premolars, extracted for periodontal or orthodontic reasons, were used for the study after receiving the informed consent of the donors. Exclusion criteria were presence of caries, cracks or root resorptions.
The crown of each tooth was removed 1 mm above the CEJ by means of a slow-speed diamond wheel\textsuperscript{a} under copious water cooling. Working length was established at 1 mm from the root apex. Cleaning and shaping of the root canal were performed with Protaper Ni-Ti rotator instruments (size S1, S2, S3)\textsuperscript{b} following the crown-down technique. Irrigations with 5% sodium hypochlorite were performed between instrumentations. Gutta-percha cones\textsuperscript{c} were used for filling the root canal and cemented with an epoxy-resin based sealer\textsuperscript{d} according to the lateral condensation technique. Roots were coronally sealed with a glass-ionomer cement\textsuperscript{e} (LOT: 0410221) and stored in a laboratory incubator at 37°C and 100% relative humidity. After 24 hours, the temporary seal was abraded by means of #240 SiC paper under water cooling, and the coronal gutta-percha was removed with a pre-shaping drill\textsuperscript{f}, leaving a 5 mm-long apical seal. A 7 mm-deep post space was prepared with a universal drill to match the size of the co-respective DT Light Post #3\textsuperscript{g} (maximum diameter: 2.14 mm). The posts are made of unidirectional pre-tensed quartz fibers bound in an epoxy resin matrix. Fiber posts were divided in 5 groups (n=10) according to the surface pre-treatment performed: Group 1. Silanization: a single-component, pre-hydrolized silanizing agent\textsuperscript{h} (Batch n°: H34023) was applied with a microbrush on the entire post surface for 60 s and then gently air-dried. Group 2. Etching: posts were immersed in 10% hydrogen peroxide solution\textsuperscript{i} for 20 min at room temperature and then rinsed with deionized water for 3 min. Group 3. Sandblasting: the entire post surface was blasted with aluminum oxide particles (110 µm)\textsuperscript{j} for 5 sec at 2.8 bar. The tip of the sandblasting device was held perpendicularly to the post surface at a distance of 1 cm. Group 4. Industrially silicate/silane coating\textsuperscript{k}. 
A patented protective layer ensured that the superficial coating was not contaminated or deactivated during its handling. Group 5. No treatment.

Prior to the luting procedures, each post was cleaned in an ultrasonic device for 10 min, immersed in 95% ethanol solution and air-dried.

Two self-adhesive composite cements were used for luting fiber posts into root canals: 1) RelyX Unicem unidose\textsuperscript{1}; 2) Max-Cem\textsuperscript{m}. The materials were handled following manufacturer’s instructions. Application modes, chemical compositions and batch numbers of the investigated materials are presented in Table 1. After capsule activation, RelyX Unicem was injected into the post space by means of a specific elongating tip directly connected to the capsule. Base and catalyst (1:1) of Max-Cem were dispensed onto a glass pad and carefully mixed until obtaining an homogeneous paste. The material was inserted with a lentulo spiral into the post-space and was applied on the entire post surface. Each post was inserted into the canal and maintained under pressure until the complete setting of the materials. Two seconds of light irradiation allowed to easily remove the cement excesses. Self-adhesive cements were polymerized in a dual-curing mode. After the first 5 min of auto-cure, additional 40 s of light polymerization through the translucent fiber post were performed (Astralis 7, output: 600 mW/cm\textsuperscript{2})\textsuperscript{n}. A core build-up was created with CoreX resin composite\textsuperscript{f} (batch n\textsuperscript{o}: 0703000080) around the post. Specimens were then stored in a laboratory incubator for 1 month at 37°C and relative humidity in order to simulate a clinical hydration state.

**Push-out bond strength test**

The portion of the root containing the fiber post was subsequently sectioned into four-to-six 1 mm-thick slices with a diamond saw (Isomet)
under water cooling. The cylindrical plunger of the testing machine (Triax 50, Controls S.P.A, Milan, Italy) was forced to dislodge, via an apical-coronal direction, each inverted, truncated fiber post from the root dentin. A load (cross-head speed: 0.5 mm/min until failure) was then applied to the post surface that resulted in shear stresses along the cement/dentin – cement/post interfaces. The retentive strength of the post fragment (MPa) was calculated by dividing the load at failure (Newton) by the interfacial area of the post segment (S_L). The formula used for measuring the tronco-conical area was so expressed: 

\[ S_L = \pi (R+r) \left( h^2 + (R-r)^2 \right)^{0.5} \]

in which \( \pi \) was equal to 3.14, \( R \) and \( r \) were the coronal and the apical post radius respectively, and \( h \) the root slice thickness. The diameters of the post and the thickness of the slice were individually measured using a digital caliper with 0.01 mm accuracy.

Failure modes were evaluated by a single operator under an optical microscope (Olympus SZ-CTV, Olympus, Tokyo, Japan) at 40x magnification and classified as follows: cohesive within the cement (C), adhesive between the post and the cement (AP), adhesive at the cement/radicular dentin level (AD), mixed, adhesive and cohesive decementations occurred simultaneously (M).

**Scanning electron microscopy evaluation (SEM)**

Four stressed-to-failure beams from each group were used for scanning electron microscopy (SEM) evaluation. Each beam was conditioned with ascending ethanol solution (from 50% up to 90%), mounted on a metal stub, gold-sputtered (Polaron Range SC 7620, Quorum Technology, Newhaven, UK) and evaluated under an SEM (JSM-6060LV, Jeol, Tokyo, Japan) at different magnifications.
<table>
<thead>
<tr>
<th>Materials</th>
<th>Compositions</th>
<th>Cement Application</th>
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| **RelyX Unicem**  
 (*3M ESPE*)  
 **Batch n°: 270644** | **Powder:** glass fillers, silica, calcium hydroxide, self-curing initiators, pigments, light-curing initiators, substituted pyrimidine, peroxide compound. **Liquid:** methacrylated phosphoric esters, dimethacrylates, acetate, stabilizers, self-curing initiators, light-curing initiators. | Mix capsule for 2-4 s and the insert it into the Aplicap Applier. Attach the elongation tip to the applicator. Apply cement in the root canal from bottom to top. Self-cure for 5 min and light-cure for 40s. |
| **MaxCem**  
 (*Kerr Corp.*)  
 **Batch n°: 432187** | **Base:** Uretanemethacrylate, Camphoroquinone, Fluoroaluminosilicate, others.  
 **Catalyst:** Bis-GMA, Triethyleneglycoldimethacrylates, Glycerophosphatedimethacrylates, Bariumaluminopolosilicate glass, Others. | Automix cement. Apply, self-cure for 5 min and light-cure for 40 s. |
| **DT Light Post**  
 (*Vereinigte Dentalwerke*)  
 **Batch n°: 120US0401A** | **Unidirectional pre-tensed quartz fibers:** 60% vol. Epoxy resin: 40% vol. Fiber density: 32/mm². Translucent, double flared. Apical diameter: 1.2 mm. Apical taper: 0.02. Coronal diameter: 2.2 mm. Coronal taper: 0.10. | - |
| **DT Light SL Post**  
 (*Vereinigte Dentalwerke*)  
 **Batch n°: 05/65** | **Unidirectional pre-tensed quartz fibers coated with silicate/silane and a protective layer made of MMA:** 60% vol. Epoxy resin: 40% vol. Fiber density: 32/mm². Translucent, double flared. Apical diameter: 1.2 mm. Apical taper: 0.02. Coronal diameter: 2.2 mm. Coronal taper: 0.10. | - |

**Results**

**Push-out bond strength test**

Mean (SD) push-out bond strengths are displayed in Table 2. Bond strengths of RelyX Unicem were statistically higher than those recorded for Max-Cem (p<0.05). Post treatment did not influence bond strength of RelyX Unicem (p>0.05). Max-Cem attained superior bond strength when

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Table 1. Chemical composition, manufacturer, batch numbers and application mode of the materials tested in the study.
bonded to silanated posts. No significant differences were found among the other tested treatments.

Failure mode distribution of the experimental groups are presented in Table 3. A predominance of adhesive failures at the cement/dentin interfaces were recorded for RelyX Unicem, independently from the post conditioning treatment. Mixed (2-5%) and adhesive failures (38-44%) between Max-Cem and the hydrogen peroxide etched- and sandblasted-posts was recorded, while cement/dentin debondings were prevalent when Max-Cem luted the non treated, silanated and/or industrially coated posts. Cohesive failures (from 2% up to 34%) within the cement were also registered for Max-Cem in all the experimental groups. No cohesive failures of the posts were observed.

Table 2: Mean (SD) bond strengths (MPa) recorded for the self-adhesive cements used for luting conditioned fiber posts. Different letters in each column and asterics in each row indicate statistically significant differences among the experimental groups (p<0.05).

<table>
<thead>
<tr>
<th>Experimental groups</th>
<th>Unicem</th>
<th>Maxcem</th>
</tr>
</thead>
<tbody>
<tr>
<td>Silane (DT Light Post)</td>
<td>7.24 (2.45) A</td>
<td>4.79 (2.06) B*</td>
</tr>
<tr>
<td>Hydrogen peroxide (10%)</td>
<td>7.25 (2.42) A</td>
<td>1.63 (1.23) A*</td>
</tr>
<tr>
<td>Sandblasting (Rocatec Pre)</td>
<td>7.02 (3.14) A</td>
<td>1.15 (1.09) A*</td>
</tr>
<tr>
<td>Silicate/silane coating (DT Light SL post)</td>
<td>6.25 (2.46) A</td>
<td>1.83 (1.94) A*</td>
</tr>
<tr>
<td>DT Light Post</td>
<td>9.3(2.6)</td>
<td>3.86(2.94) A*</td>
</tr>
</tbody>
</table>
Table 3. Percentage of failure recorded in each experimental group. AD: adhesive failures occurred between the cement and the dentin; AP: Adhesive failures between cement and post; C: cohesive failures observed within the cement; M: Mixed failures, a combination of AD and AP.

<table>
<thead>
<tr>
<th>Experimental groups</th>
<th>Unicem</th>
<th>Maxcem</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>AD</td>
<td>AP</td>
</tr>
<tr>
<td>Silane (DT Light Post)</td>
<td>100%</td>
<td>-</td>
</tr>
<tr>
<td>Hydrogen peroxide (10%)</td>
<td>100%</td>
<td>-</td>
</tr>
<tr>
<td>Sandblasting (Rocatec Pre)</td>
<td>100%</td>
<td>-</td>
</tr>
<tr>
<td>Silicate/silane coating (DT Light SL post)</td>
<td>100%</td>
<td>-</td>
</tr>
<tr>
<td>DT Light Post</td>
<td>69%</td>
<td>19%</td>
</tr>
</tbody>
</table>

Scanning electron microscopy (SEM)

Representative SEM images of debonded slices are presented in Figs. 1 and 2. RelyX Unicem resulted in a continuous bonded interface when luting silanated posts (Fig.1A). Micro-spaces were created on the post surface after 10% hydrogen peroxide etching and mechanical blasting. The cement was able to flow into the micro-porosities forming tight bonded interfaces (Fig. 1B and C). The fibers of the industrially silicate/silane coated post surface appeared wrapped in the coating blend (Fig. 1 D). The cement structure was compact and neither voids nor bubbles were highlighted (Fig. 1E).

Max-Cem was able to properly bond to the post after silane application; nevertheless, voids were observed within the cement bulk and more irregular interfaces were assessed at the dentin side (Fig. 2A).
cement appeared unable to penetrate the microretentive spaces formed on the post surface after 10% hydrogen peroxide etching and sandblasting (Figs. 2B and 2C). Tight adhesive interfaces were formed when Max-Cem was luted to the industrially coated DT Light SL Post (Fig. 2D), although porosities were observed within the cement thickness (Fig. 2E).

**Fig. 1:** Representative SEM images of fiber posts cemented with RelyX Unicem after different chemo/mechanical surface pre-treatments (1,000x, 10 bar). **A)** Silanized post (DT Light Post); **B)** After etching with 10% hydrogen peroxide; **C)** After sandblasting (Rocatec Pre); **D)** Silicate/silane coating (DT Light SL Post, VDW); **E)** No treatment (DT Light Post). Adhesive failures at the cement/dentin interface were predominant in all the experimental groups. The cement appeared well adherent to the post substrate, independently of the post surface pre-treatment performed. In the DT Light SL Post image (D) the glass fibers were completely wrapped into the resin.
Discussion

The null hypothesis has to be rejected as post surface treatments influenced the bond strengths of the tested self-adhesive cements. Differences exist between the attained bond strengths of RelyX Unicem and Max-Cem.

Fig. 2: SEM photographs of fiber posts cemented with Maxcem after different post surface pre-treatments (1,000x, 10 bar). A) Silanated (DT light Post); B) After etching with 10% hydrogen peroxide; C) After sandblasting (Rocatec Pre); D) Silicate/silane coating (DT Light SL Post); E) No treatment (DT Light Post). Maxcem was adherent to silanated post, even though the cement appeared viscous and inhomogeneities were observed at the interfaces. No tight bonded interfaces at the posts side were evident in the others groups. Although rough post surfaces were created after the chemomechanical treatment, the material is not able to penetrate the spaces created.
A reliable bonding between post/cement and cement/dentin is guaranteed when strong joints are simultaneously created at the two adhesive interfaces. Although bonding to radicular dentin may be considered as the weakest point of a fiber post-restored tooth, debondings at the fiber post/resin cement interfaces may also compromise the clinical outcome of the restoration (Cagidiaco et al, 2007) (Ferrari et al, 2007b) (Hagge et al, 2002) (Ferrari et al, 2000). Bonding of resin cements to fiber posts is based on a chemical interaction. However, the combination of chemical interaction and mechanical retentions may be of help for increasing their retentive strength (Monticelli et al, 2008). In general, this is possible by treating the fiber post surface with chemo-mechanical procedures which are intended to eliminate/dissolve the epoxy matrix of the fiber posts, considered unsuitable to establish chemical bonds with methacrylate based materials, and to expose the underneath fibers that can be then activated and readily react with the resinous cement (Monticelli et al, 2006) (Monticelli et al, 2008b) (Vano et al, 2006).

The application of a silane coupling agent on fiber post surface resulted in different bonding behaviours of the tested self-adhesive cements: no increase in the bond strength values were assessed for RelyX Unicem, whereas Max-Cem attained superior retentive strength when compared to the other experimental groups, notwithstanding the push-out bond strength values of Max-Cem were inferior to those recorded with RelyX Unicem (Table 2). Previous findings are controversial regarding the real benefits of fiber post silanization for improving the adhesion to composite materials. The alkoxy groups of organosilanes establish chemical bridges with OH-covered substrates, such as glass fibers, and increase the surface wettability. The application of a silane coupling agent
before luting with the self-adhesive cement was previously questioned (Hayashi et al, 2008) (Bitter et al, 2007). In the present study, the cement/post interfaces were homogeneous for the tested cements (Figs. 1A and 2A) and no adhesive failures at the fiber post side were found (Table 3). However, the failure analysis revealed a predominance of adhesive debondings at the cement/dentin side (Table 3), still highlighting concerns regarding the effective sealing ability of these simplified resin cements (Watzke et al, 2008).

Hydrogen peroxide etching was proposed as an alternative fiber post surface treatment (Monticelli et al, 2006) (Vano et al, 2006). The chemical etching procedure increased the fiber post surface roughness, the epoxy-resin matrix was partially dissolved and the fibers were exposed and undamaged. The microretentive spaces created on fiber post surface represented sites where the material could flow and establish stronger mechanical retentions (Monticelli et al, 2006) (Monticelli et al, 2008b). However, according to the obtained results, etching the fiber post surface with 10% hydrogen peroxide for 20 min did not improve the bond strength values of the tested cements (Table 2). The SEM images revealed an intimate contact between RelyX Unicem and the etched fiber post, as the material homogeneously flowed into the retentive site bonds formed (Fig. 1B). On the contrary, discontinuous interfaces were evidenced between Max-Cem and the hydrogen peroxide treated fiber post (Fig. 2B). The inherent viscosity of the material may hamper its penetration into the created micro-porosities (Monticelli et al, 2005). The presence of gaps at the bonded interfaces would negatively affect the ultimate adhesion mechanism, representing sites from which stresses may be expedited (Mazzitelli et al, 2007). The presence of voids and bubbles into the cement
bulk may also have accounted for the high number of mixed and cohesive failure registered for Max-Cem (Table 3). This phenomenon can be attributed to the different dispensing modalities. RelyX Unicem was directly injected into the root canal by means of a specific elongating tip, whereas Max-Cem was manually mixed and then inserted into the canal with a lentulo spiral and applied directly on the post. The use of a flexible root-canal shaped elongating tip is advisable in order to reach the deeper apical third of the root canal and avoid undesirable air-bubble entrapments and imperfections within the cement bulk (Watzke et al, 2008) (Simonetti et al, 2008).

Sandblasting is a mechanical procedure intended to provide a plastic deformation and to improve the surface roughness of a fiber post, resulting in an increased surface areas for bonding (Kern et al, 1994). This procedure determined higher bond strengths of fiber posts into root canals and no damages of the glass fibers were manifested (Sahafi et al, 2003) (Mazzitelli et al, 2008). The time and the distance of application and the size of aluminium-oxide particles were all considered factors determining the effect of the blasting procedure. In the present study, sandblasting of fiber post was performed using 110 µm aluminium-oxide particles blasted for 5 s on the post surface at a distance of 1 cm. This resulted in mild conditioning effect which was considered advantageous for treating fiber posts when compared to other aggressive procedures, such as hydrofluoric acid (Vano et al, 2006) (Mazzitelli et al, 2008). Based on the SEM images, the mechanical blasting created roughened fiber post surface with the formation of micro-spaces. Although RelyX Unicem was able to flow along with these retentive spaces (Fig. 1C), it did not represent an additional advantage that could improve the bond strengths (Table 2).
Max-Cem produced irregular cement/fiber post interfaces, being difficult to diffuse into the created grooves (Fig. 2C) and no differences in its retentive strength were found when compared to the hydrogen peroxide etched, silicate/silane coated and non treated groups (Table 2).

The industrially treated fiber posts have been recently introduced in the market in the attempt of simplifying the clinical procedures while ensuring reliable bonding. The post inner structure is wrapped in the pre-coating blend, and nor the epoxy resin nor the fibers are exposed. Accordingly, the bonding mechanism is based on the chemical interaction between the silicate/silane coating and the luting cement with no direct involvement of the epoxy-rein matrix (Mazzitelli et al, 2008). So, more stable bonds are supposed to be formed. However, no differences in bond strengths were observed for RelyX Unicem in comparisons with non treated posts. Max-Cem recorded inferior bond strengths when luted to DT Light SL Post and compared to the silanated post (Table 2). Based on the findings of a previous report, the industrially coated fiber post is superficially smooth leaving no chance to develop micromechanical interlocking (Mazzitelli et al, 2008). Additionally, due to the high acidity of the cement, the formation of a layer of acidic monomers on the post surface would hamper the chemical reaction with the post and this, plus the lack of mechanical retentions, may have accounted for the high percentage of debondings at the cement/fiber post side (Table 3) (Wrbas et al, 2007).

The concept of an “ideal monoblock” inside the root canal should further be investigated. According to the circumferential interfaces created for the cementation of fiber posts into radicular dentin, three type of monoclock units can be individuated (Tay et al, 2007). Self-adhesive
cements seemed to represent the secondary monoblock, as they can form two bonding interfaces: the cement/dentin and the cement/fiber posts. In order to be successfully and mechanically validated as effective bonding agents, they should possess two main prerequisites: 1) the cement has to strongly bond to the radicular dentin and to the fiber post; 2) the cement has to show a moduli of elasticity similar to the bonding substrates. The fiber posts, the cement and the dental substrate have to show similar moduli of elasticity in order to better distribute the stresses along the root. The involvement of a third interface would make the idea of monoblock more complex. When using the DT Light SL Post, another interface is introduced between the bonding substrates: the external silicate/silane coating transform the bond into a tertiary monoblock unit (Tay et al, 2007). In general, although the effectiveness of self-adhesive cements has previously been emphasized, further studies are highly warranted to improve the bonding mechanism of these simplified cements used for the cementation of fiber posts into root canal. In addition, more information regarding their mechanical properties (i.e. moduli of elasticity, polymerization shrinkage) is needed.

Within the limits of the present investigation, it can be concluded that the effectiveness of post surface treatments on the bond strength of self-adhesive cements is material-related. No increasing in bond strength would be expected for RelyX Unicem, whereas it seemed necessary to pre-treat the fiber post surface with silane solutions when using Max-Cem for luting fiber posts.
References


Wrbas KT, Altenburger MJ, Schirrmeister JF, Bitter K, Ktelbassa AM. Effect of adhesive resin cements and post surface silanization on the


Chapter 7

7.1 Summary, Conclusions and Future Directions

The selected luting material and the cementation procedures may influence the retention and durability of prosthetic restorations. Luting materials and techniques have been widely investigated both in the clinical practice and research attempting to simplify them while ensuring a certain longevity of the prosthetic restoration. The issues related to this topic include the evaluation of the bond strength, the effectiveness of the cements as sealing agents, the interaction with the dental substrates and the restorations, the dispensing modality and mechanical properties. A simplification of the luting procedures was made possible with the introduction of self-adhesive cements. Self-adhesive resin cements have been defined as “universal” as they can lute different types of indirect restorations, such as fiber posts, zirconia/ceramic crown and/or bridges, composite inlays/onlays, and screws. According to manufacturers claims, only veneer cementation should be avoided.

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 luting procedures nowadays available for luting indirect restorations, the introduction went deeply to analyze the cement systems characteristics, focusing on those of self-adhesive resin cements. In particular, the data available into the literature regarding the bonding effectiveness of self-adhesive resin cements used to lute composite onlays, fiber posts or ceramic crowns were analyzed. The scope of the auto-adhesive technology was to simplify luting procedures and overcome the difficulties related to
the multi-step cement systems, such as the high-influence of the operator variability and the chemical incompatibility that can occur when using dual-curing luting agents with their simplified adhesive systems. However, differences in the chemical composition, dispensing modality, pH and mechanical properties influence the bonding mechanism of the simplified cements. In particular, the interaction between these cements and the dentin substrate is still a matter of study, and research is now focused to improve this site bond. The ability of self-adhesive cements to lute fiber posts was also considered with and without thermal ageing test, and the possibility to combine pre-treated posts with the simplified cements was analyzed.

In the second part of this thesis (Chapter 2), the study was centered to deeply analyze the interaction between simplified self-adhesive cements and the dentin substrate. An observational study based on light and scanning electron microscopy allowed to compare the interfacial characteristics (in terms of dentin demineralization/resin penetration) of self-adhesive cements and resin cements that use a total-etch and a self-etch adhesive. Differences between the material were encountered, showing that a total-etch cement system was able to deeply demineralize the dentin substrate. Self-adhesive cements showed only limited capacity to interact with dentin.

In Chapter 3, bond strength and scanning electron microscopy evaluations of different self-adhesive cements to perfused dentin was presented. Again, a total-etch resin cements was used for comparisons. The hydration state of dentin differently influence the tested cements. While having a detrimental effects on the multi-step resin cement, the presence of pulpal pressure may be beneficial during the cementation of
selected self-adhesive cements. A more specific classification of the simplified resin cements was made, as some of them showed some silicate-like cement characteristics. In presence of vital dentin, certain cements could be advisable for the cementation procedures, while attention should be paid when using multi-step resin cements, since water can permeate through dentinal tubules, reaching the adhesive interfaces and hamper a proper setting reaction of the cement itself. In this terms, the simulation of an hydrostatic intra-pulpal pressure should be taken into consideration when performing luting procedures \textit{in vitro}.

Although self-adhesive cements do not need pre-treatments of dentin, in Chapter 4 the attention has been focused to the possibility of conditioning the dental substrate with mild acidic solutions (0.1 M EDTA and 10% polyacrylic acid) before cement application in order to improve the cement/dentin interactions. According to the microtensile bond strength values recorded, differences in the bonding performance of self-adhesive cements were found. An improved adhesion was recorded when a self-adhesive cement, that showed glass-ionomer-like characteristics, was luted on 10% polyacrylic acid etched dentin. The Masson’s staining technique for optical microscopy allowed to individuate the interfacial characteristics of the three self-adhesive cements tested under the experimental conditions. However, scarce cement/dentin interaction was observed, and, in particular, exposed collagen fibers were found at the bottom of the adhesive interfaces renewing concerns regarding the effective sealing ability of the simplified resin cements.

In Chapter 5, two studies based on the evaluation of the bonding performance of self-adhesive cements to fiber posts were performed. In Paragraph 5.1 the push-out bond strengths of three self-adhesive cements
used to lute epoxy-resin based fiber posts into radicular dentin were evaluated. The results of the study indicate that differences can be found between the bonding mechanism of the self-adhesive resin cements mainly due to their different chemical compositions, that make necessary a deep classification of this new class of resin cements. The cement application mode into the post space was also considered an important factor influencing the results obtained. The quality of the adhesive interface can be affected by the operative procedure, so the use of an elongating tip is advisable for placing the cement into the post space. The various indications coming from the results of the study described before found a confirmation in Paragraph 5.2, in which a durability test was performed. Three differently marketed self-adhesive resin cements were used to lute fiber posts and their bond strengths were examined prior and after being submitted to 5,000 cycles of thermal ageing. This study revealed that the thermal stresses did not affect the push-out bond strength values of RelyX Unicem and Breeze, whereas increased those of G-Cem. A combination of chemical interactions and micro-mechanical retentions seemed to characterize the bonding mechanism of self-adhesive cements to fiber posts. Although the incidence of adhesive failure at the cement/dentin side was relevant, decementations at the cement/fiber post interfaces occurred in all the experimental groups.

More precisely, fiber reinforced material technology gave suggestion to test different fiber post superficial treatments with the aim of obtaining an effective adhesion. A series of preliminary observations were conducted in Chapter 6. A research was carried out and described in Paragraph 6.1: confocal microscopy and atomic force microscopy evaluations were combined to evaluate the effects of different
chemo/mechanical fiber post surface treatments on their surface topographies and average surface roughness. This approach successfully validated the use of potassium permanganate, sodium ethoxide and sandblasting to treat the post surface among the tested conditioning procedures: increased surface roughness through the partial removal/dissolution of the epoxy-resin matrix would improve the surface area available for adhesion by creating micro-retentive spaces. Conversely, hydrofluoric acid was considered an aggressive conditioning method, as it caused excessive damages of the quartz fibers. In Paragraph 6.2, the influence of fiber post superficial treatments on the retentive strength of self-adhesive resin cements used to lute epoxy-resin based fiber posts into radicular dentin was considered. Non destructive chemo/mechanical conditioning approaches were adopted for treating fiber posts and two self-adhesive cements were used. Post surface conditionings did not improved the retention of RelyX Unicem to fiber posts, whereas Max-Cem additionally benefit of the application of a silane coupling agent to optimize the bond strength. However, the viscosity of the materials seemed to hamper the complete penetration of the cements into the micro-spaces created by the conditioning approaches. Differences were also found between the luting agents. An inferior percentage of defects was detected when an elongating tip was used to place the cement into the dowel space. The use of application aids is highly advisable to limit the occurrence of defects and air-entrapment into the cement bulk.
Conclusions

The following conclusions can be drawn from the laboratory researches based on the evaluation of the bonding potential of different self-adhesive resin cements employed for the cementation of coronal composite restorations and/or fiber posts:

1) A limited interaction with dentin characterize the bonding behavior of self-adhesive resin cements when compared to a total-etch or a self-etch adhesive systems. In particular, the chemical composition of each product influences their bonding mechanism.

2) The hydration state of dentin should be taken into consideration when restoring a vital tooth. The continuous water fluid flow through dentinal tubules can influence the bonding effectiveness of luting cements; in particular it can be detrimental for cements that utilize multi-step adhesive systems. Conversely, self-adhesive resin cements, take benefits from the water transudation as a setting reaction similar to that of silicate cements can be postulated.

3) Considerations should be made when pre-treating dentin with mild acidic solutions before luting indirect restorations with self-adhesive cements. Dentin etching with 10% polyacrylic acid could be proposed before using selected self-adhesive cements.

4) Differences exist between the bonding potential of different self-adhesive resin cements used to lute fiber posts into radicular dentin. The dispensing modality affect their bonding mechanism and the use of an elongating tip become necessary to avoid any defect within the cement bulk.

5) Thermal ageing does not affect the bonding potential of the tested self-adhesive cements. An improved setting reaction is speculated
in presence of high temperature. A combination of chemical reactions and micro-mechanical retentions characterize the self-adhesive cements bonding to fiber posts.

6) Surface conditioning procedures that selectively react with the epoxy-resin matrix of the fiber post enhance roughness and improve the surface area available for adhesion by creating micro-retentive spaces without affecting the post’s inner structure. Hydrofluoric acid affects the superficial texture of quartz fibers.

7) Fiber posts surface treatments do not improve the retention of self-adhesive resin cements. The viscosity of the materials hampers their penetration into the micro-spaces created on the post surface after the conditioning modalities. The adhesion to radicular dentin remains an “hot topic” which need to be optimized.

Future directions

Dental research targeted at the development of ideal materials has been ongoing for many years. Since its introduction in the late 50s, adhesion has undergone considerable maturation, increasing the role exerted in daily practice and dental investigation. The desire to obtain a long lasting prosthetic restoration with simple and less time consuming cementation procedures is a driving force behind the continuous quest of clinicians and dental manufacturers and leads researchers to continuously develop their thoughts. The self-adhesive technology is undoubtedly innovative and open the door to a simplified cementation procedure. However, many limitations may moderate their clinical use. Innovations should be performed on the adhesion mechanism to dentin (coronal and radicular).
Some characteristics of self-adhesive cements, in terms of viscosity or contraction stress, should be improved and tested. Techniques intended to reduced the high viscosity of the materials are necessary in order to enhance a deeper penetration of the resin into the demineralized dental tissue or into the conditioned restoration. Future studies should also evaluate the shrinkage percentage of the simplified resin cements in presence of an high C-factor (i.e. into root canal).

There are no doubts that clinical studies are highly warranted in order to validate the results presented in the present doctoral thesis and to make these cements totally clinically recommendable.
7.2 Riassunto, Conclusioni e direzioni future

L’esito di un restauro protesico è direttamente relazionato al tipo di materiale e alla tecnica di cementazione utilizzati. La ricerca dei materiali è costantemente concentrata a individuare e migliorare i materiali e le tecniche da cementazione in maniera da favorire una certa longevità del restauro. In generale, i temi relazionati allo studio dei materiali da cementazione si riferiscono principalmente alla valutazione della forza di adesione, alla capacità sigillante dei suddetti prodotti, l’interazione tra il cemento e i substrati dentali e i differenti materiali da restauri, le modalità di applicazione e le proprietà meccaniche. L’introduzione dei cementi resinosi auto-adesivi ha permesso di semplificare le tecniche di cementazione. Questi cementi sono stati definiti “universalì”, in quanto capaci di legarsi a diversi tipi di restauri, come ai perni in fibra, corone o ponti in zirconia/ceramica, inlays/onlays di composito e screws. Secondo quanto consigliato dai produttori, è sconsigliata la cementazione di faccette con i materiali semplificati. Nella parte iniziale di questo progetto (Capitolo 1), è stato presentato l’obiettivo prefissato nello studio. Dopo una breve descrizione dei materiali e delle tecniche da cementazione attualmente disponibili, l’introduzione si è concentrata a descrivere le caratteristiche dei vari cementi, focalizzando l’attenzione sui cementi resinosi auto-adesivi. In particolare, sono stati analizzati i dati presenti nella letteratura dentale riguardo l’utilizzo dei cementi auto-adesivi per la cementazione di onlays in composito o perni in fibra. Lo scopo principale dei cementi auto-adesivi è stato quello di semplificare le tecniche di cementazione limitando le difficoltà riscontrate con l’utilizzo di cementi che utilizzano sistemi adesivi a tre o due passaggi, come per esempio la
grande influenza dell’operatore, e l’incompatibilità chimica che si può verificare tra i cementi duali e i sistemi adesivi semplificati. Il meccanismo di adesione dei cementi one-step può essere influenzato dalla loro composizione chimica, pH, modalità di applicazione e proprietà meccaniche. In particolare, il conoscimento del tipo di interazione tra i cementi resinosi semplificati e la dentina è continuamente oggetto di studio, e attualmente la ricerca si propone di migliorare il tipo di adesione.

L’abilità dei cementi auto-adesivi utilizzati per la cementazione dei perni in fibra è stata inoltre presa in considerazione, in presenza o meno di stress termici, così come è stata anche analizzata la possibilità di combinare la semplicità di utilizzo dei suddetti cementi con perni in fibra pre-trattati in superficie con metodiche chimico/meccaniche.

Nella seconda parte di questa tesi (Capitolo 2), l’attenzione è stata focalizzata nell’analizzare più approfonditamente l’interazione tra i cementi auto-adesivi e il substrato dentinario. Le osservazioni sono state condotte utilizzando la microscopia elettronica a scansione (SEM) e la tecnica tricromia di Masson per la microscopia ottica, che, unitamente, hanno permesso di comparare le caratteristiche dell’interfaccia (in termini di demineralizzazione del substrato dentale/penetrazione del materiale resinoso) tra i cementi auto-adesivi e cementi che utilizzano sistemi adesivi a tre o due passaggi. I risultati dello studio hanno rivelato che esistono differenze tra i materiali testati, mostrando una maggiore abilità per i cementi resinosi che utilizzano sistemi adesivi a demineralizzare la dentina. Al contrario, i cementi semplificati hanno dimostrato una minore capacità di interazione con il substrato dentale.

Nel Capitolo 3, è stato presentato uno studio basato su misurazioni della forza di adesione e valutazioni con microscopia elettronica a
scansione dei cementi adesivi utilizzati su dentina in presenza di pressione pulpare. Nuovamente, un cemento total-etch è stato utilizzato per la comparazione. I dati ottenuti hanno dimostrato che in presenza di pressione pulpare, i cementi testati si comportano diversamente. Da un lato, i cementi resinosi total-etch non traggono beneficio dalla presenza di perfusione dentina ria, mentre i cementi auto-adesivi si comportano in maniera differente. E’ stato quindi possibile fare una classificazione più specifica dei cementi semplificati, dal momento che alcuni dei materiali testati hanno dimostrato possedere caratteristiche simili a quelle dei cementi silicati. Per questo che, quando si restauro un dente vitale, alcuni cementi auto-adesivi possono essere consigliabili nelle fasi di cementazione, mentre altri materiali dovrebbero essere utilizzati con cautela, dal momento che l’acqua procedente attraverso i tubuli dentinari può raggiungere l’interfaccia adesiva, mescolarsi al materiale non ancora indurito e interferire con l’ultima fase della polimerizzazione. Inoltre, avendo osservato diversi comportamenti tra i materiali, la simulazione di pressione idrostatica dovrebbe essere preso in considerazione quando si adoperano procedure di cementazione in vitro.

Nonostante i cementi auto-adesivi non richiedono alcun previo trattamento della dentina e dei restauri, l’obiettivo dello studio del Capitolo 4 è stato prendere in considerazione la possibilità di trattare la dentina con soluzione acide deboli (0.1 EDTA e acido poliacrilico al 10%) prima di applicare i cementi auto-adesivi con l’intenzione di migliorare l’interazione tra i due substrati adesivi. I dati della forza di adesione microtensile hanno evidenziato differenze nella performance adesiva dei materiali testati. L’interazione adesiva è stata migliorata solo per un cemento che dimostra una tecnologia simile a cementi vetro-ionomerici e
utilizzato su dentina mordenzata con il 10% di acido poliacrilico. La colorazione tricromia di Masson ha permesso, inoltre, di individuare le caratteristiche dell’interfaccia tra i tre materiali utilizzati e la dentina. In generale, una scarsa interazione cemento/dentina è stata osservata per tutti e tre i cementi testati, evidenziando, in particolare, la presenza di fibre collagene non encapsulate alla base dell’interfaccia adesivi, rinnovando nuovamente dei dubbi riguardo alla effettiva capacità sigillante fornita dai cementi resinosi semplificati.

Nel Capitolo 5, sono stati presentati due studi basati sulla valutazione della capacità adesiva dei cementi auto-adesivi per la cementazione di perni in fibra. Nel Paragrafo 5.1, è stata valutata la forza di adesione push-out di tre cementi auto-adesivi utilizzati per la cementazione di perni in fibra con una matrice a base di resina epossidica all’interno del canale radicolare. Le principali differenze annoverate tra le diverse capacità adesive dei materiali testati sembra più essere legata alle differenti composizioni chimiche. Di contro, una classificazione più dettagliata dei nuovi cementi auto-adesivi dovrebbe essere presa in considerazione. Inoltre, il modo di applicazione del cemento all’interno dello spazio endodontico può influenzare i risultati, per questo che l’utilizzo di un puntale elastico dovrebbe essere consigliato per permettere l’inserimento del cemento. Le indicazioni riscontrate nel precedente studio, hanno trovato conferma nel Paragrafo 5.2, dove è stato eseguito un test di durabilità. Tre cementi auto-adesivi sono stati impiegati per cementare i perni in fibra e la loro forza di adesione è stata determinata prima e dopo essere stati sottoposti a 5.000 cicli di stress termici. I risultati ottenuti hanno dimostrato che il termociclaggio non ha influenza sulle forza di adesione di RelyX Unicem e Breeze, mentre hanno migliorato
quella di G-Cem. Un insieme di interazione chimica e ritenzione meccanica sembra caratterizzare il meccanismo di adesione dei cementi semplificati ai perni in fibra nel canale radicolare. Nonostante il numero di fallimenti adesivi tra cemento e dentina, è stato anche registrato un alto numero di decemntazioni cemento/perno in fibra in tutti i gruppi sperimentali.

Max-Cem ha avuto beneficio dalla previa applicazione di una gente silano sulla superficie del perno. La viscosità del cemento può, comunque, ostacolare la completa penetrazione della resina negli spazi ritentiv creati dopo i trattamenti chimico/meccanici. Differenze sono state inoltre riscontrate per i due cementi testati, e un minor numero di difetti è stato visto quando il materiale è stato introdotto nello spazio endodontico utilizzando il puntale elastico. Per questo che l’utilizzo del suddetto puntale è clinicamente raccomandabile per evitare l’instaurarsi di difetti interni e inglobamento di aria nella struttura del cemento.

**Conclusioni**

Le seguenti conclusioni si possono dedurre dagli studi *in vitro* condotti sulla valutazione della capacità adesiva dei cementi resinosi semplificati utilizzati per la cementazione di overaly in composito su dentina coronale e/o di perni in fibra:

1) L’interazione cemento auto-adesivo/dentina è limitata quando comparata a un cemento total-etch. In particolare, le diverse composizioni chimiche possono influenzare il loro meccanismo di adesione;

2) Lo stato idratato della dentina deve essere preso in considerazione quando si tratta un dente vitale. Il continuo flusso di acqua attraverso i tubuli dentinari influenza il comportamento adesivo dei cementi; in particolare, può avere effetti detrimenti sulla forza di adesione dei cementi che utilizzano sistemi adesivi a tre pasaggi, mentre alcuni cementi auto-adesivi traggono beneficio dalla
presenza di acqua, postulando una reazione simile a quella dei cementi silicati;

3) Alcune considerazioni devono essere fatte quando si desidera pretrattare la dentina con soluzioni acide deboli prima dell’applicazione dei cementi auto-adesivi. La mordenzatura della dentina con una soluzione di acido poliacrilico al 10% può essere raccomandabile solo per quei cementi che si basano su una tecnologia simile ai cemento vetro-ionomerici;

4) Differenze nelle forze di adesione esistono tra i vari materiali autoadesivi utilizzati per la cementazione dei perni in fibra. La modalità di applicazione del cemento nello spazio endodontico influenza il loro meccanismo di adesione, e l’utilizzo di un puntale elastico è necessario per limitare i difetti e gli inglobamenti di aria nella struttura del cemento;

5) Gli stress meccanici non influenzano la forza di adesione dei cementi auto-adesivi testati. Una combinazione di interazione chimica e ritenzione micromecanica caratterizza il meccanismo di adesione dei cementi semplificati ai perni in fibra nel canale radicolare;

6) I trattamenti di superficie dei perni in fibra, che reagiscono selettivamente con la resina epoxidica della matrice, aumentano la loro rugosità e migliorano l’area disponibile per l’adesione tramite la creazione di spazi micro-ritentivi e senza danneggiare le fibre. L’utilizzo dell’acido fluoridrico non è consigliabile in quanto affetta la tessitura superficiale delle fibre del perno;

7) Alcuni trattamenti di superficie dei perni in fibra possono influenzare la forza di adesione di determinati cementi auto-
adesivi. Comunque, la viscosità del materiale può ostacolare la completa penetrazione del cemento negli spazi micro-ritentivi creati dai trattamenti condizionanti del perno in fibra. L’adesione dei cementi semplificati alla dentina rimane un “tema caldo” che necessita maggiori chiarimenti.

Direzioni future

La ricerca dentale che si è prefissata di sviluppare materiali con migliori caratteristiche va avanti da diversi anni. Fin dalla sua prima introduzione negli anni ’50, il tema dell’adesione ha preso sempre più piede nella pratica clinica e nella ricerca dentale. Il desiderio da parte dei dentisti di ottenere restauri protesici duraturi con l’utilizzo di materiali semplici e che permettano un risparmio di tempo ha incentivato i ricercatori a sviluppare nuovi pensamenti. La tecnologia auto-adesiva è, senza dubbio, innovativa e apre le porte a un nuovo metodo, semplice e veloce, di cementazione. Comunque, alcune limitazione dimostrate nella letteratura possono limitare il loro utilizzo clinico. Per questo che nuove innovazioni e miglioramenti dovrebbero essere ottenuti nel loro meccanismo di adesione alla dentina (sia radicolare che coronale).

Alcune caratteristiche dei cementi auto-adesivi, come la viscosità o la contrazione da polimerizzazione, dovrebbero essere testate e migliorate. Tecniche rivolte a diminuire la viscosità dei cementi semplificati sono necessario, in maniera da favorire una più completa penetrazione della resina nel tessuto dentale demineralizzato o negli spazi ritentivi dei restauri pre-trattati. Inoltre, studi futuri dovrebbero focalizzarsi sulla
valutazione dello stress da contrazione dei cementi auto-adesivi in presenza di un alto fattore C (come per esempio, nel canale radicolare).

Sicuramente, studi clinici sono altamente desiderabili per potere validare i risultati ottenuti nella ricerca e potere così rendere i cementi auto-adesivi completamente raccomandabili da un punto di vista clinico.
7.3 Resumen, Conclusiones y direcciones futuras

El tipo de material y técnica de cementado puede influenciar la retención y la durabilidad de una restauración indirecta. Estudios clínicos y de laboratorio se han propuesto de simplificar los materiales y las técnicas de cementado con el objetivo de preservar la longevidad de la restauración. Los temas relacionados con este asunto se basan sobre evaluaciones de la fuerza de adhesión, la habilidad de los materiales de sellar las interfases adhesivas, la interacción con el sustrato dental y las superficies de las restauraciones, la modalidad de aplicación y las propiedades mecánicas. Una simplificación de las técnicas de cementado se ha hecho posible a través de la introducción en el mercado dental de los cementos resinosos auto-adhesivos. Estos cementos resinosos han sido nombrados como “universales” gracias a la capacidad que han demostrado de ligarse a varios tipos de restauración, como por ejemplo los postes de fibra, coronas y/o puentes en zirconia/ceramica, inlay/onlay de composite, y tornillos. Según las sugerencias de los productores, estos materiales sólo no son recomendable para el cementado de carillas.

En la parte inicial de esta thesis doctoral (Capítulo 1), se realizó una introducción al principal argumento en estudio. Tras una breve descripción de las técnicas actualmente disponibles para el cementado de las restauraciones indirectas, se comentaron las características de los diferentes cementos utilizados para el cementado, encentrándose en aquellas de los cementos auto-adhesivos. En particular se analizó el potencial adhesivo de los cementos resinosos simplificados utilizados para el cementado de onlays de composite, postes de fibra o coronas de ceramica. La finalidad de la tecnología auto-adhesiva trata de simplificar...
las técnicas de cementado y sobrepasar las desventajas de los cementos resinosos que utilizan los sistemas adhesivos de paso múltiple, como por ejemplo la influencia del operador y la incompatibilidad química que se verifica entre los cementos duales y los sistemas adhesivos simplificados. Las diferentes composiciones químicas, la modalidad de dispensación, el pH, y las propiedades mecánicas, diferencian el mecanismo de adhesión de los varios cementos simplificados. La interacción entre estos cementos y el sustrato dental se queda una de las mayores dudas relacionadas con la técnica simplificada de cementación, y por eso que la investigación se concentra en mejorar esta interfase adhesiva. Igual se analizó la capacidad de unión de los cementos auto-adhesivos utilizados para el cementado de los postes de fibra antes y tras ser sometido a ciclos de termociclado, así como la posibilidad de combinar los mismos cementos con postes de fibra pre-condicionados.

En la segunda parte de esta thesis doctoral (Capítulo 2), se evaluó detalladamente el tipo de relación entre los cementos auto-adhesivos y el sustrato dental. El estudio se propuso de observar las características de las interfases adhesivas (en particular el grado de desmineralización de la dentina y la penetración de la resina en la dentina acondicionada) utilizando diferentes cementos auto-adhesivos y cementos resinosos que utilizan un sistema adhesivo de grabado total y un otro que utiliza un sistema adhesivo de grabado y lavado y para la cementación de overlays de resina compuesta. Las observaciones indicaron diferentes características de las interfases entre los cementos testados, enseñando como el cemento que se basa en un sistema adhesivo de grabado total, permite lograr un grado de desmineralización de la dentina mayor con respecto a los demás cementos utilizados en el estudio. Contrariamente, los
cementos auto-adhesivos interactuaron con la dentina solamente de manera limitada.

En el Capítulo 3, se presentó un enfoque basado en medida de fuerza de adhesión y evaluaciones de microscopía electrónica de barrido de la unión entre los cementos resinosos auto-adhesivos y la dentina hidratada. Otra vez, se utilizó como grupo control el cemento resinoso de grabado total, cuyas características se estudiaron de manera profunda. El estudio reveló que la presencia de una presión pulpar puede influenciar de manera diferente los cementos testados. Si de un lado se notó un efecto destructivo en el cemento de paso múltiple, la aplicación de una presión pulpar durante la fase de cementación de las muestras aportó beneficios a algunos cementos testados. Fue posible aclarar la específica clasificación de estos cementos, que hasta ahora se clasificaron como una clase de los cementos resinosos. En presencia de una presión pulpar a nivel de la superficie dentinal, algunos cementos pueden ser consejados para la cementación, mientras que algunos de estos enseñaron un comportamiento negativo: el agua, procedente de los tubos dentinales, llega a nivel de la interfase adhesiva y se mezcla con los monómeros resinosos todavía no completamente polimerizados. Esta reacción puede obstaculizar la fase de polimerización del material y consecuentemente influir negativamente en el proceso final de adhesión. Por todo eso, durante las investigaciones en laboratorio, una presión pulpar debería ser tomada en consideración.

A pesar de que los cementos adhesivos no requieren algún pre-tratamiento de la dentina, en el Capítulo 4 la atención se focalizó en la posibilidad de poder acondicionar la superficie dental con soluciones ácidas ligeras (0.1 M EDTA y 10% ácido poliacrílico) precedente la fase de cementación, con el objetivo de mejorar la interacción cemento/dentina.
Así como revelado de los valores de fuerza de adhesión de microtensión, los cementos auto-adhesivos testados enseñaron poseer comportamientos adhesivos diferentes. Una mejoría de la fuerza de adhesión se vio sólo cuando, un cemento que demuestra una tecnología parecida a aquella de los cementos ionomero de vidrio, fue aplicado a la dentina pre-tratada con 10% de ácido poliacrílico. La coloración tricromica de Masson para la microscopía optica, trató de individuar las características de la interfase de los tres cementos resinosos simplificados testados bajo las condiciones experimentales. Se hizo encapié de que la interacción cemento/dentina fue bastante escasa, con presencia de fibras de colageno no encapsulada al pie de la capa híbrida, aportando nuevas dudas acerca de la efectiva capacidad de sellado que poseen estos cementos simplificados.

En el Capítulo 5 se presentaron dos estudios basados en la evaluación de la capacidad adhesiva de los cementos auto-adhesivos utilizados para la cementación de postes de fibra. En el Parrafo 5.1 el enfoque se centró en medir la fuerza de adhesión de push-out de tres cementos auto-adhesivos para el cementado de postes de fibra en el interior del canal radicular. Los resultados de este estudio indicaron que hay diferencias entre el potencial adhesivo de los cementos, principalmente debido a sus diferentes composiciones químicas, que presupone una reclasificación de sus clase de pertenencia que respecte sus comportamientos adhesivos. Un factor que influenció de manera predominante la fuerza de adhesión se individuó también en la modalidad de aplicación del cemento en el espacio endodóntico. En efecto, la calidad de las interfases adhesivas puede estar negativamente afectada por el procedimiento operativo, por eso que el uso de un puntal elástico para la inserción del cemento es altamente recomendable. Los distintos resultados
de los trabajos de investigación *in vitro* fueron confirmados en el **Parrafo 5.2** en un estudio que trató de la durabilidad de la unión cemento/poste de fibra. Por ello, se emplearon 3 cementos auto-adhesivos de diferentes productores, y se examinaron las fuerzas de adhesión antes y después ser submitido a 5.000 ciclos de termociclado. Los estreses termicos no resultaron influir negativamente en la fuerza de adhesión de RelyX Unicem y Breeze, mientras que incluso aumentaron aquellos de G-Cem. Una interacción química y una retención micromecanica conjuntamente caracterizaron el mecanismo de adhesión de los cementos simplificados a los postes de fibra. A pesar de que los fallos adhesivos a nivel de la interfase cemento/dentina manifestaron una gran incidencia, se registró también un alto numero de decementaciones entre cemento/poste de fibra en todos los grupos experimentales.

En particular, la tecnología de los materiales reforzados en fibra fue de sugerencia para el análisis de distintos metodos de grabado superficial de la superficie de los postes con el objetivo de mejorar la fuerza de adhesión. Observaciones preliminares se desarrollaron en el **Capítulo 6**. Un proyecto de investigación se condujo en el **Parrafo 6.1**: la microscopía confocal y la microscopía a fuerza atomica se emplearon para la evaluación de los efectos de distintos tratamientos quimio/mecanico de la superficie de los postes de fibra en la topografía superficial y para medir la rugosidad de superficie. Este enfoque validó con suceso el uso del potasio permanganato, el etoxido de sodio y el arenado como metodos viables para el tratamiento de superficie de los postes de fibra. Sin embargo, el ácido hidrofluorico se reveló excesivamente agresivo, y se rescontaron daños de las fibras de los postes. En el **Parrafo 6.2** se evaluó la influencia de los tratamientos quimio/mecanicos de la superficie de los postes de
fibra sobre la fuerza de adhesión de los cementos auto-adhesivos. Para ello, se consideraron los tratamientos de superficie que no se demostraron agresivos para las fibras y los postes se cementaron utilizando dos materiales de cementación auto-adhesivos. El estudio no desveló una mejoría de la fuerza de adhesión de RelyX Unicem a pesar del tipo de tratamiento de superficies adoptado, mientras que Max-Cem trajo beneficio de una previa aplicación de un agente silano en la superficie del poste demostrando un aumento de la fuerza de adhesión. De todas formas, la intrínseca viscosidad del material fue un factor que influenció la completa penetración del material en los espacios creados por los tratamientos superficiales así como se mostraron diferencias en las fuerzas de adhesión entre los materiales de cementación utilizados. Se observó un porcentaje inferior de defectos en la capa de aquel cemento que fue aplicado en el canal radicular a través de un puntal elastico. Por eso, que el utilizzo de dicho puntale es recommendable clínicamente para la introducción del material en el espesor del poste para evitar la ocurrencia de defectos y entrapamiento de aire en el espesor del cemento.

Conclusiones

Las conclusiones y recomendaciones siguientes se pueden deducir de las evaluaciones básicas en la evaluación de la capacidad de unión de los cementos resinosos auto-adhesivos utilizados para el cementado de restauraciones coronales en composites o postes de fibra:

1. Una limitada interacción caracteriza las interfases cementos auto-adhesivos/dentina coronal en comparación a un cemento que utiliza un sistema adhesivo de paso multiple. En particular, la
composición química de cada producto parece influenciar el comportamiento adhesivo de los cementos testados.

2. El estado de hidratación de la dentina tiene que ser tomado en consideración cuando se desea replicar en el laboratorio la situación clínica de un diente vital. La presencia de un presión pulpar a través de los tubulos dentinarios parece influenciar negativamente el comportamiento adhesivo de cementos que utilizan adhesivos de paso múltiple. Contrariamente, algunos cementos auto-adhesivos traen beneficio de la presencia de una presión pulpar demostrando una reacción de fraguado similar a la de los cementos silicato.

3. Algunas consideraciones tienen que ser hechas cuando se desea pre-tratar la dentina con soluciones ácidas débiles antes la aplicación de los cementos auto-adhesivos. El tratamiento previo de la dentina con una solución al 10% de ácido poliacrílico puede ser conveniente antes la aplicación de G-Cem.

4. Los cementos auto-adhesivos presentan un comportamiento distinto cuando utilizados para el cementado de los postes de fibra en el canal radicular. La modalidad de dispensación puede influir en sus mecanismos de adhesión, y por ello que el uso de un puntal elastico es altamente recomendable para la introducción del cemento en el espacio del poste.

5. La simulación en laboratorio de los estreses termicos no afecta la fuerza de adhesión de los cementos auto-adhesivos. La alta temperatura parece favorecer la reacción de polimerización de los cementos resinosos simplificados. Además, el mecanismo básico
de estos cementos, parece ser caracterizado por una interacción química junto a una retención micromecánica.

6. Los tratamientos superficiales que reaccionan con la matriz de resina epoxica de los postes de fibra aumentan sus rugosidad de superficie y mejoran el área disponible para la adhesión a través la creación de espacios microretentivos en la superficie de los mismos. Entre las soluciones utilizadas, el ácido hidrofluorico ha demostrado de dañar excesivamente las fibras de los postes.

7. Los tratamiento de superficie de los postes de fibra no tienen influencia en la capacidad de unión de RelyX Unicem. El tratamiento del postes con una solución silano es recomendable antes el utilizar de Max-Cem. De todas formas, la viscosidad del material parece disminuir la capacidad de penetración del cemento en las rugosidades creadas tras los tratamientos de superficie. La adhesión a la dentina radicular se queda la mayor incertidumbre que tiene que ser optimizada.

**Direcciones futuras**

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 50, 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 con técnicas de cementado sencillas y rápidas se considera el motivo clave quel leva los investigadores a mejorar siempre sus conocimiento detrás también de la continua demanda de los odontólogos clínicos y
de la industria. La tecnología de los materiales auto-adhesiva es, sin duda, innovador y permite una simplificación de las técnica de cementación de las restauraciones indirectas. Sin embargo, algunas limitaciones pueden confinar el uso clínico de estos cementos simplificados. Mejoras deberían ser aportadas al mecanismo de unión a la dentina (coronal y radicular).

Algunas características de los cementos auto-adhesivos deberían ser testadas y mejoradas, como por ejemplo la viscosidad y la contracción de polimerización. La viscosidad de estos materiales tiene que ser reducida para alcanzar una penetración más profunda de la resina en la dentina desmineralizada o en las restauraciones acondicionadas. Estudios futuros deberían también evaluar el grado de contracción de los cementos auto-adhesivos en presencia de un alto factor C (i.e. en el conducto radicular).

Seguramente, estudios clínicos son altamente deseables para poder validar los resultados obtenidos en esta tesis doctoral y poder totalmente recomendar su uso in vivo.
7.4 Zusammenfassung, Schlussfolgerungen, Zukünftige Richtungen


In dem ersten Teil dieses Projekts (Kapitel 1) wurde eine Einleitung über das Hauptthema der Studie dargestellt. Nach einer kurzen Beschreibung der Zementierung-Techniken, die heutzutage für die indirekte Restaurationen verwendet werden, wurden die Eigenschaften der verschiedenen Zementsystemen analysiert und die selbst-adhäsiven Zemente wurden besonders berücksichtigt. Insbesondere wurden die Daten der Literatur über die Zementierung von Komposit Onlays, Faserstiften bzw. Keramik Kronen mit selbst-adhäsiven Zementen berücksichtigt. Das Ziel der selbst-adhäsiven Technologie war es, die Zementierung zu simplifizieren und die Schwierigkeiten, die mit den


Im Kapitel 3 wurde die Verbundfestigkeit zwischen verschiedenen selbst-adhäsiven Zementen und durchgeschwemmtem Dentin erforscht und REM Beobachtungen wurden auch ausgeführt. Ein Total-etch Zement

Interaktion zwischen Zement und Dentin beobachtet und insbesondere wurden ungedeckten Kollagenfasern auf dem Boden der adhäsiven Schnittstellen gefunden. Daher macht man sich Sorgen um die echte Versiegelungsfähigkeit der simplifizierten Zemente.


_Schlussfolgerungen_

Die folgende Schlussfolgerungen können aus den Laborstudien, die die Adhäsion verschiedener selbst-adhäsiven Zemente für die Zementierung von Komposit-Restaurationen bzw. Faserstiften bewertet haben, gezogen werden:


2) Die Hydratation des Dentins sollte für die Restauration eines vitalen Zahnes berücksichtigt werden. Der ständige Strom des Wassers durch die Dentinkanälen kann die Adhäsion der Zemente beeinflussen; insbesondere kann er für
Zemente, die Multi-Step Adhäsiven benötigen, schädlich sein. Dagegen ist der Strom des Wassers günstig für die selbst-adhäsiven Zemente, weil sie eine ähnliche Härtung wie die Silicat-Zemente haben.


6) Behandlungen der Oberfläche, die mit der Epoxydharzmatrix des Faserstiftes selektiv reagieren, erhöhen den Rillenabstand und sie verbessern die adhäsive Oberfläche durch Mikroretentionen ohne der Struktur der Quarzfasern zu schaden.

Zukünftige Richtungen


Manche Eigenschaften der selbst-adhäsiven Zemente bezüglich der Viskosität und des Schrumpfung-Stress sollten verbessert und getestet werden. Techniken, die auf die Senkung der großen Viskosität der Materialien zielen, sind notwendig, um ein tieferes Durchschlagen des
Resins in die demineralisierte Zahnartsubstanz bzw. in die behandelte Restauration zu ermöglichen. Zukünftige Studien sollten den Prozentsatz der Schrumpfung der simplifizierten Zemente bei einem großen C-Faktor (z. B. in dem Wurzelkanal) bewerten.

Es gibt keine Zweifel daran, dass klinische Untersuchungen stark nötig sind, um die Ergebnisse der Laborstudien zu bestätigen und um die klinische Anwendung dieser Zemente zu empfehlen.
7.5 Résumé, conclusions et Directions futures

Le ciment et la procédure de scellement choisis peuvent influencer la rétention et la pérennité des restaurations prothétiques. Les matériaux et techniques de scellement ont été largement étudiés que ce soit sur le plan clinique ou expérimental dans le but de les simplifier tout en maintenant la longévité de la prothèse. Les problèmes liés à ce sujet comprennent l’évaluation de la force d’adhésion, l’efficacité des ciments en tant qu’agents de scellement, l’interaction avec les substrats dentaires et les restaurations, et les modalités de mise en place et les propriétés mécaniques. Une simplification des procédures de scellement est intervenue avec l’introduction des ciments autoadhésifs. Les ciments autoadhésifs ont été définis comme “universels” puisqu’ils sont capables de coller différents types de restaurations indirectes comme les tenons en fibres, les couronnes ou bridges en zircone ou céramique, les inlays/onlays en composite, et les tenons vissés. Selon les fabricants, seul le scellement des facettes est à proscrire.

Le chapitre 1 constitue une introduction du sujet principal. Après une courte description des procédures contemporaines de scellement des restaurations indirectes, ce chapitre analyse en détail les caractéristiques des ciments en se penchant plus précisément sur ceux des systèmes autoadhésifs. En particulier, les données disponibles dans la littérature concernant l’efficacité du collage des onlays composites, des tenons en fibres, et des couronnes céramiques par des résines autoadhésives est analysée. L’objectif des technologies autoadhésives est de simplifier les procédures de collage et de surmonter les difficultés liées au systèmes comprenant plusieurs étapes, tels que la haute influence de la variabilité
liée à l’opérateur ou l’incompatibilité chimique qui intervient lors de l’utilisation de ciments auto/photo polymérisant avec leur systèmes adhésifs simplifies. Toutefois, les différences dans la composition chimique, les modalités de mise en place, le pH, et les propriétés mécaniques influent sur le mécanisme de collage des ciments simplifies. En particulier, l’interaction entre ces ciments et le substrat dentinaire et toujours à l’étude, et la recherche se focalise à présent sur l’amélioration du collage à ce niveau. La capacité des ciments autoadhésifs à sceller les tenons en fibres a également été étudiée avec et sans thermocyclage et la possibilité de combiner un prétraitement de ces tenons avec des ciments simplifies a également été analysée.

Dans la seconde partie de ce travail (Chapitre 2), l’étude a plus particulièrement analysé l’interaction entre les ciments autoadhésifs simplifies et le substrat dentinaire. Une étude d’observation basée sur la microscopie optique et électronique à balayage a permis de comparer les caractéristiques des interfaces (en termes de déminéralisation/pénétration de la dentine) des ciments autoadhésifs et des ciments résines qui utilisent des adhésifs automordançants, ou multi systèmes (également appelés total-etch). Des différences entre les matériaux ont été identifiées démontrant que les systèmes dits « total-etch » étaient capables de déminéraliser le substrat dentinaire en profondeur. Les ciments autoadhésifs ne se sont révélés capables que d’une interaction limitée avec la dentine.

Dans le Chapitre 3, l’évaluation de la force d’adhésion et une étude par microscopie électronique à balayage de différents ciments autoadhésifs à de la dentine perfusée a été réalisée. De même, un ciment résine « total-etch » a été utilisé comme référence. Le niveau d’hydratation de la dentine influence de façon différente les ciments testés. Bien qu’ayant un effet
préjudiciable sur le ciment « total-etch », la présence de pression pulpaire peut être bénéfique lors du collage à l’aide de certains ciments autoadhésifs. Une classification plus spécifique des ciments résines simplifiés a été établie parce que certains d’entre eux ont démontrés des caractéristiques similaires aux ciments simili-silicates. En présence de dentine vitale, certains ciments pourraient être conseillés pour les procédures de collage, alors que des précautions doivent être prises lors de l’utilisation de ciments « total-etch » en raison de la percolation d’eau à travers les tubuli dentinaires qui peut atteindre l’interface d’adhésion et compromettre une polymérisation adéquate du ciment proprement dit. Il semble. En ce sens, il est évident que la simulation de la pression hydrostatique intra-pulpaire est requise lors des tests in vitro des procédures de collage.

Bien que les ciments autoadhésifs de nécessitent pas de prétraitement de la dentine, le Chapitre 4 porte l’attention sur la possibilité du conditionnement du substrat dentinaire à l’aide de solutions acides faibles (0.1M EDTA and 10% polyacrylic acid) avant l’application du ciment dans le but d’améliorer l’interaction ciment/dentine. Selon les forces microtensiles enregistrées, des différences dans les capacités de collage des ciments autoadhésifs ont été détectées. Une amélioration de l’adhésion a été enregistrée lorsqu’un ciment autoadhésif, qui présentent des similarités aux ciments verres-ionomères, a été utilisé pour sceller à de la dentine mordançée à l’aide d’acide polyacrylique a 10%. La technique coloration de Masson en microscopie optique a permis d’individualiser les caractéristiques interfaciales des 3 ciments autoadhésifs testés expérimentalement. Toutefois, de rares interactions dentine/ciment ont été observées, et en particulier des fibres de collagène exposées on été
trouvées au fond des interfaces adhésives, ce qui a renouvelé les craintes concernant la capacité proprement dite de scellement des ciments résine simplifiés.

Dans le Chapitre 5, deux études basées sur l’évaluation des performances de collage de ciments autoadhésifs ont été réalisées. Dans le Paragraphe 5.1, les forces de push-out de 3 ciments autoadhésifs utilisés pour sceller les tenons en fibres et matrice en résine époxy à la dentine radiculaire ont été évaluées. Les résultats des études indiquent que des différences peuvent être détectées entre les mécanismes de collage des ciments résines autoadhésifs particulièrement en raison de leurs compositions chimiques différentes, ce qui rend nécessaire une classification en profondeur de cette nouvelle classe de ciments résine. Le mode d’application du ciment à l’intérieur de l’espace canalaire a également été considéré comme un facteur important conditionnant les résultats obtenus. La qualité de l’interface adhésive peut être affectée par la procédure opératoire, ainsi l’utilisation d’un embout d’élongation est conseillée pour mettre le ciment dans le logement du tenon. Les différentes indications dérivant des résultats ci dessus ont trouvé confirmation dans le Paragraphe 5.2, dans lequel un test de pérennité a été réalisé. Trois ciments résines autoadhésifs commerciaux ont été utilisés pour sceller des tenons en fibres et leurs forces d’adhésion ont été mesurées avant et après avoir été soumis à 5000 cycles de vieillissement thermique. Cette étude a révélé que les stress thermiques n’ont pas affecté les valeurs de « push-out » de RelyC Unicem ou de Breeze, alors que celles de G-Cem ont été augmentées. Une combinaison d’interactions chimiques et de rétentions micromécaniques a semblé caractériser le mécanisme d’adhésion des ciments adhésifs aux tenons en fibres. Bien que
l’incidence des fractures adhésives à l’interface ciment/dentine ait été significative, un décollement à l’interface ciment/tenon a été observé dans tous les groupes expérimentaux.

Plus précisément, la technologie des matériaux renforcés par fibres a suggéré de tester différents traitements de surface des tenons dans le but d’améliorer la rétention. Une série d’observations préliminaires a été effectuée dans le Chapitre 6. Une recherche a été menée et décrite dans le Paragraphe 6.1: des évaluations par microscopie confocale et à force atomique ont été combinées afin d’évaluer les effets des différents traitements de surface chemomécaniques des tenons en fibres sur la topographie et la rugosité de leur surface. Cette approche a validé l’utilisation de permanganate de potassium, d’éthoxyde de sodium, et du sablage parmi les différentes méthodes de traitement de la surface des tenons : l’augmentation de la rugosité de surface à travers la dissolution/retrait de la matrice en résine époxy augmenterait la surface disponible à l’adhésion par la création d’espaces microrétentifs. Inversement, l’acide hydrofluorhydrique a été considéré comme une méthode de conditionnement agressive causant des dommages sévères aux fibres de quartz. Dans le Paragraphe 6.2, l’influence des traitements superficiels des tenons en fibres sur la force d’adhésion des résines autoadhésives utilisées pour le scellement les tenons en fibres à matrice époxy dans l’espace canalaire a été étudié. Des méthodes de conditionnement chemomécaniques non destructives ont été adoptées pour traiter les tenons en fibres et 2 ciments autoadhésifs ont été utilisés. Le conditionnement de surface des tenons n’a pas amélioré la rétention de RelyX Unicem aux tenons en fibres alors qu’il s’est avéré être un bénéfice supplémentaire pour Max-Cem après application d’un agent de couple
type silane afin d’optimiser la force d’adhésion. Toutefois, la viscosité des matériaux utilisés a semblé limiter la pénétration des ciments dans les micro espaces générés par les méthodes de conditionnement. Des différences ont également été trouvées entre les ciments de scellement. Un moindre pourcentage de défauts a été observé lorsqu’un embout d’élongation était utilisé pour mettre le ciment en place à l’intérieur du logement du tenon. L’utilisation d’aides au placement est hautement conseillée afin de limiter les possibilités de défauts ou d’emprisonnement d’air au sein de la masse de ciment.

Conclusions

Les conclusions suivantes peuvent être tirées à partir des études expérimentales basées sur l’évaluation du potentiel d’adhésion de différents ciments résines autoadhésifs utilisés dans le collage de restaurations coronaires composites et/ou de tenons en fibres :


2) Le niveau d’hydratation de la dentine devrait être pris en considération lors de la restauration de dents vitales. Le flot continu d’eau à travers les tubuli dentinaires peut influencer l’efficacité du collage des ciments, en particulier il peut être néfaste à ceux utilisant des systèmes multi étapes. A l’inverse, il peut être postulé que les ciments autoadhésifs mettent à profit la
transsudation hydrique dans une réaction de prise similaire à celles des ciments silicates.

3) Certaines considérations devraient être prises en compte lors du traitement de la dentine par des solutions acides faibles avant le scellement de restaurations indirectes à l’aide de ciments autoadhésifs. Le mordançage de la dentine par de l’acide polyacrylique à 10% pourrait être proposé avant l’utilisation de certains ciments autoadhésifs.


5) Le vieillissement thermique n’affecte pas le potentiel d’adhésion des ciments autoadhésifs testés. Une réaction de prise améliorée est postulée en présence de hautes températures. Une combinaison de réactions chimiques et de retentions micromécaniques caractérise l’adhésion des ciments autoadhésifs au tenons en fibres.

6) Les procédures de conditionnement de surface qui réagissent sélectivement avec la matrice époxy du tenon en fibre améliorent la rugosité et augmentent la surface disponible pour l’adhésion en créant des espaces de micro rétention sans affecter la structure interne du tenon. L’acide fluorhydrique affecte la texture superficielle des fibres de quartz.

7) Les traitements de surface des tenons en fibres n’améliorent pas la rétention des ciments résines autoadhésifs. La viscosité des
matériaux empêche leur pénétration dans les micro espaces créés par les modalités de prétraitement. L’adhésion à la dentine demeure un sujet « chaud » qui nécessite une optimisation.

**Directions futures**

La recherche dentaire qui vise à développer des matériaux idéaux dure depuis des années. Depuis son introduction dans la fin des années 50, l’adhésion a subi une maturation considérable, augmentant son rôle dans la pratique quotidienne et la recherche dentaire. Le désir d’obtenir des restaurations prothétiques à longue pérennité en utilisant des procédures de collages simples et rapides représente la force motrice derrière la quête continue des cliniciens et des fabricants dentaires et pousse les chercheurs à développer de nouveaux concepts continuellement. La technologie autoadhésive est sans doute innovatrice et ouvre la voie à une procédure de collage simplifiée. Toutefois, de nombreuses limitations viennent modérer leurs applications cliniques. Des innovations devraient viser le mécanisme d’adhésion à la dentine coronaire et radiculaire.

Certaines caractéristiques des ciments autoadhésifs, en termes de viscosité ou de stress de contraction, devraient être améliorées et testées. Des techniques conçues pour réduire la viscosité élevée des matériaux sont nécessaire afin d’améliorer leur pénétration dans la dentine déminéralisée ou au niveau des surface restauratrices prétraitées. De nouvelles études devraient également évaluer le pourcentage de contraction des résines simplifiées en présence d’un facteur de contraction élevé (i.e. dans le canal radiculaire).
Il ne fait aucun doute que des études cliniques sont hautement requises pour valider les résultats obtenus expérimentalement et permettre de recommander ces produits en clinique.
7.6 Resumo, Conclusões, Futuras perspectivas

O material de cimentação selecionado e os procedimentos de cimentação podem influenciar a manutenção e durabilidade das restaurações protéticas. Materiais de cimentação e técnicas têm sido amplamente investigados tanto na prática clínica e na pesquisa de tentar simplificá-los, assegurando certa longevidade da restauração protética. As questões relacionadas com este tema incluem a avaliação da resistência de união, a eficácia dos cimentos como agentes de vedação, a interação com o substrato dental e as restaurações, a modalidade de injeção e propriedades mecânicas. A simplificação dos procedimentos de cimentação foi possível com a introdução de cimentos auto-adesivos. Cimentos resinosos auto-adesivos foram definidos como "universal", como eles podem cimentar diferentes tipos de restaurações indiretas, tais como pinos de fibra, zircônia / coroa de cerâmica e / ou pontes, inlays composto / onlays e parafusos. Segundo alegações dos fabricantes, somente facetas devem ser evitada a cimentação.

Na parte inicial deste projeto (Capítulo 1), uma introdução do tema principal do estudo foi apresentada. Após uma breve descrição dos procedimentos de cimentação hoje disponíveis para a cimentação de restaurações indiretas, a introdução foi profundamente para analisar as características de sistemas de cimento, com incidência nos de auto-cimentação de resina adesiva. Em particular, os dados disponíveis na literatura sobre a eficácia da ligação de cimentos auto-adesivos usada para onlays, pinos de fibra ou coroas de cerâmica foram analisados. O objetivo de cimentos auto-adesivos foi o de simplificar os procedimentos de
cimentação e superar as dificuldades relacionadas com o cimento de sistemas multi-passo, como a influência de alta variabilidade do operador e da incompatibilidade química que pode ocorrer quando utilizar agentes cimentantes de presa dupla com os seus sistemas adesivos simplificados. No entanto, as diferenças na composição química, modalidade de inserção, pH e propriedades mecânicas influenciam o mecanismo de união dos cimentos simplificado. Em particular, a interação entre esses e os cimentos para a dentina ainda é uma questão de estudo e investigação estando agora centrada em melhorar esta ligação local. A capacidade de auto-cimentos adesivos para cimentação pinos de fibra também foi considerado com e sem teste de envelhecimento térmico, bem como a possibilidade de combinar previamente tratada pinos com os cimentos simplificado foi analisada.

Na segunda parte desta tese (Capítulo 2), o estudo foi concentrado para analisar profundamente a interação entre os cimentos adesivos auto simplificado e substrato dentina. Um estudo de microscopia óptica e microscopia eletrônica de varredura permitiu a comparação das características interfaciais (em termos de desmineralização de dentina / penetração de resina) de cimentos auto-adesivos e cimentos resinosos que utilizam um total-etch e um auto-adesivo. As diferenças foram encontradas entre os materiais, mostrando que um cimento de condicionamento total foi capaz de desmineralizar profundamente o substrato dentina. Cimentos auto-adesivos mostraram uma capacidade apenas limitada para interagir com a dentina. No capítulo 3, resistência de união e microscopia eletrônica de varredura avaliações de diferentes cimentos auto-adesivos à dentina foram avaliados. Novamente, um cimento de condicionamento total foi utilizado para comparações. O
diferente estado de hidratação da dentina influenciou os cimentos testados. Embora tenha um efeito negativo sobre os cimentos de multi-passos, a presença de pressão pulpar pode ser benéfico durante a cimentação de cimentos auto-adesivos. A classificação mais específica dos cimentos simplificados foi feita, como alguns deles apresentam características semelhantes de presença de silicato. Na presença de dentina vital, cimentos, poderiam ser aconselhável para os procedimentos de cimentação, ao passo que deve ser dada atenção ao usar cimentos resinosos multi-etapas, já que a água pode permear através dos túbulos dentinários, atingindo as interfaces adesivas e dificultar uma reação adequada fixação do cimento em si. Nestes termos, a simulação de uma pressão hidrostática intra pulpar deve ser tomado em consideração adequadamente a realização de procedimentos de cimentação in vitro.

Embora a auto-cimentos adesivos não necessitam de pré-tratamentos da dentina, no capítulo 4, a atenção foi dada a possibilidade de condicionar o substrato dental com leve soluções ácidas (0,1 M de EDTA e ácido poliacrífico 10%) antes da aplicação do cimento, a fim de melhorar as interações cimento / dentina. De acordo com a resistência à microtração as diferenças no desempenho da ligação dos auto-cimentos adesivos foram encontrados. A aderência melhorou quando um cimento auto-adesivo, que mostrou-ionômero de vidro-como em sua composição, foi cimentado em dentina condicionada por 10% de ácido poliacrífico. A técnica de coloração de Masson para microscopia óptica permitiu individualizar as características interfaciais dos três cimentos auto-adesivos testados sob as condições experimentais. No entanto, escassa interação de cimento / dentina foi observada, e, em particular, fibras colágenas expostas foram encontrados no fundo da interfaces do adesivo o que renova as
preocupações quanto à efetiva capacidade de vedação dos cimentos resinosos simplificados.

No capítulo 5, dois estudos com base na avaliação do desempenho da ligação dos cimentos auto adesivos para pinos de fibra foram realizadas. No tópico 5.1 testes de push-out de três cimentos auto-adesivos utilizados para cimentação de pinos de fibra de resina epóxica a dentina radicular foram avaliados. Os resultados do estudo indicam que as diferenças podem ser encontradas entre o mecanismo de ligação dos cimentos resinosos auto-adesivos, principalmente devido às suas diferentes composições químicas, que tornam necessária uma classificação profunda desta nova classe de cimentos resinosos. O modo de aplicação do cimento no espaço do pino também foi considerado um fator importante que influencia os resultados obtidos. A qualidade da interface adesiva pode ser afetada pelo procedimento clínico, portanto, o uso de um alongamento da ponta é aconselhável para a colocação do cimento no espaço do pino. As várias conclusões provenientes dos resultados do estudo descrito anteriormente foram encontradas confirmações no tópico 5.2, em que a durabilidade teste foi realizada. Três diferentes cimentos auto-adesivos foram utilizados para pinos de fibra a força de união foi avaliada antes e após serem submetidos a 5.000 ciclos de envelhecimento térmico. Este estudo revelou que o estresse térmico não afetou os valores de força de push-out bond do RelyX Unicem e Breeze, enquanto aumentaram os de G-Cem. Uma combinação de interações químicas e micro-retenções mecânicas parecem caracterizar o mecanismo de adesão de cimentos auto-adesivos aos pinos de fibra. Embora a incidência de falha adesiva cimento / dentina lado era relevante,
“decimentações” na interface cimento / pino de fibra ocorreu em todos os grupos experimentais.

Mais precisamente, a tecnologia de material reforçado com fibra gerou a sugestão para o pós-teste de diferentes tratamentos superficiais do pino de fibra com o objetivo de obter uma adesão efetiva.

Uma série de observações preliminares foram conduzidos no Capítulo 6. A pesquisa foi realizada e descrita no § 6.1: avaliações de microscopia confocal e microscopia de força atômica foram combinados para avaliar os efeitos de diferentes pós tratamentos químico / mecânico da superfície do pino de fibra na topografia de sua superfície e rugosidade da superfície. Esta abordagem validou com sucesso o uso permanganato de potássio, sódio e jateamento para tratar a superfície do pino entre os procedimentos testados:o aumento da rugosidade da superfície através da remoção parcial / dissolução da matriz de resina epoxídicas melhorou a área de superfície disponível para a adesão através da criação de micro-espacos retentivos. Por outro lado, o ácido fluorídrico foi considerado um método de condicionamento agressivo, como ele causou danos excessivos das fibras de quartzo. No ponto 6.2, a influência dos tratamentos superficiais da fibra sobre a resistência retentiva dos cimentos resinosos auto-adesivos utilizados para cimentação de pinos de resina epóxicas a dentina radicular foi considerada. Abordagens não destrutivas químico / mecânicas foram adotadas para o tratamento de pinos de fibra e dois cimentos auto adesivos foram utilizados. Condicionamentos de superfície do pino não melhorou a retenção de RelyX Unicem aos pinos de fibra, enquanto Max-Cem obteve um adicional benefício da aplicação de um agente de silanizador para otimizar a força de adesão. No entanto, a viscosidade do material parece dificultar a penetração total dos cimentos nos micro-espacos criados pelo
condicionamento. Diferenças também foram encontradas entre os agentes de cimentação. Uma percentagem inferior de defeitos foi detectada quando uma ponta alongada foi utilizado para colocar o cimento no espaço. A utilização de aparelhos de aplicação é altamente recomendável para limitar a ocorrência de defeitos e aprisionamento de ar na massa de cimento.

Conclusões

As seguintes conclusões podem ser tiradas com base nas pesquisas laboratoriais na avaliação do potencial de ligação de diferentes cimentos auto adesivos utilizados para a cimentação de restaurações em compósito e/ou pinos de fibra:

1) A interação limitada com dentina caracteriza o comportamento da ligação do auto-cimentos resinosos a adesiva quando comparado a um total-etch ou sistemas de auto-adesivo. Em particular, a composição química de cada produto influencia o seu mecanismo de adesão.

2) O estado de hidratação da dentina deve ser levado em consideração quando a restauração de um dente vital. O contínuo fluxo de fluido através dos túbulos dentinários pode influenciar a eficácia da adesão de cimentação cimentos, em particular, pode ser prejudicial para cimentos que utilizam sistemas adesivos de vários passos. Por outro lado, a cimentos resinosos auto-adesivos, levam os benefícios da transudação da água como uma reação semelhante à definição de cimentos de silicato pode ser postulada.

3) Considerações devem ser feitas quando a o pré-tratamento da dentina com soluções ácidas antes da cimentação de restaurações indiretas com cimentos auto-adesivos. O condicionamento da dentina com ácido poliacrífico 10% pode ser proposta antes de usar o cimento auto-adesivo.
4) Existem diferenças entre o potencial de ligação de cimentos auto-adesivos utilizados para cimentação de pinos de fibra em dentina radicular. A modalidade de dispensa afeta seu mecanismo de ligação e utilização uma ponta alongada se faz necessária para evitar qualquer defeito dentro da massa de cimento.

5) Envelhecimento térmico não afetar o potencial de ligação cimentos resinoso auto-adesivos. Uma reação melhorada é especulada na presença de altas temperaturas. Uma combinação de reações químicas e micro-retenções mecânicas caracterizam o vínculo de cimentos auto-adesivos para os pinos de fibra.

6) Os processos de condicionamento da seletivo da superfície que reagem com a matriz de resina epóxi do pino de fibra para aumentar e melhorar a rugosidade da superfície disponível para a adesão através da criação de micro-espacios retentivos sem afetar a estrutura interna do pino. Ácido fluorídrico afeta a textura superficial de fibras de quartzo.

7) Tratamentos da superfície de pinos de fibra não melhoram a retenção de cimentos resinosos auto-adesivos aos mesmos. A viscosidade dos materiais dificulta a sua penetração no micro-espacios criados na superfície após as modalidades de condicionamento. A adesão à dentina radicular continua a ser um tema "quente" que precisam ser otimizados.

Futuras perspectivas

Investigações odontológicas orientadas para o desenvolvimento de materiais ideais tem estado em curso há muitos anos. Desde a sua introdução no final dos anos 50, a adesão tem tido considerável maturação, aumentando o exercido na prática diária e investigação dental. O desejo de obter uma proservação duradoura da prótese com simples e práticos
procedimentos de cimentação é uma força motriz por trás da busca contínua de odontologistas e fabricantes o que leva os investigadores a desenvolver continuamente seus pensamentos. A tecnologia de auto-adesão é sem dúvida inovador e aponta a um procedimento simplificado de cimentação. No entanto, muitas limitações podem limitar o seu uso clínico. As inovações devem ser realizadas no mecanismo de adesão à dentina (coronal e radicular).

Algumas características de cimentos auto-adesivos, em termos de viscosidade ou tensão de contração, devem ser melhoradas e testadas. Técnicas destinadas a reduzir a elevada viscosidade dos materiais, a fim de promover uma profunda penetração da resina no tecido dental desmineralizado ou para o tecido condicionado. Estudos futuros deverão avaliar também o percentual de contração dos cimentos resinosos simplificados na presença de um alto fator C (ou seja, no canal de raiz).

Não há dúvidas de que os estudos clínicos são altamente necessárias, a fim de validar os resultados da investigação e validar se esses cimentos são clinicamente recomendáveis.
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Mazzitelli C. Impresión en prótesis fija: el originador para un éxito óptimo. 30° Anniversary FAM Dental, Morelia 2007, October 3-6.


**National Publications**


Abstracts


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