Turun yliopisto
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FIBER-REINFORCED COMPOSITE FIXED DENTAL PROSTHESES

Studies of the Materials Used as Pontics

Leila Perea Mosquera

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To the ones who fulfill my soul,
Michael and Naima

ABSTRACT

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Fiber-reinforced composite fixed dental prostheses – Studies of the materials used as pontics

University of Turku, Faculty of Medicine, Institute of Dentistry, Department of Biomaterials Science, Finnish Doctoral Program in Oral Sciences – FINDOS, Annales Universitatis Turkuensis, Turku, Finland 2015

Fiber-reinforced composites (FRC), a non-metallic biomaterial, represent a suitable alternative in prosthetic dentistry when used as a component of fixed dental prostheses (FDPs). Some drawbacks have been identified in the clinical performance of FRC restorations, such as delamination of the veneering material and fracture of the pontic. Therefore, the current series of studies were performed to investigate the possibilities of enhancing the mechanical and physical properties of FRC FDPs by improving the materials used as pontics, to then heighten their longevity.

Four experiments showed the importance of the pontic design and surface treatment in the performance of FRC FDPs. In the first, the load-bearing capacities of inlay-retained FRC FDPs with pontics of various materials and thicknesses were evaluated. Three different pontic materials were assessed with different FRC framework vertical positioning. Thicker pontics showed increased load-bearing capacities, especially ceramic pontics. A second study was completed investigating the influence of the chemical conditioning of the ridge-lap surface of acrylic resin denture teeth on their bonding to a composite resin. Increased shear bond strength demonstrated the positive influence of the pretreatment of the acrylic surfaces, indicating dissolution of the denture surfaces, and suggesting potential penetration of the monomer systems into the surface of denture teeth.

A third study analyzed the penetration depth of different monomer systems on the acrylic resin denture teeth surfaces. The possibility of establishing a durable bond between acrylic pontics and FRC frameworks was demonstrated by the ability of monomers to penetrate the surface of acrylic resin denture teeth, measured by a confocal scanning type microscope. A fourth study was designed to evaluate the load-bearing capacities of FRC FDPs using the findings of the previous three studies. In this case, the performance of pre-shaped acrylic resin denture teeth used as pontics with different composite resins as filling materials was evaluated. The filling material influenced the load-bearing capacities, providing more durable FRC FDPs.

It can be concluded that the mechanical and physical properties of FRC FDPs can be improved as has been shown in the development of this thesis. The improvements reported then might provide long lasting prosthetic solutions of this kind, positioning them as potentially permanent rehabilitation treatments.

Key words: fiber-reinforced composite, fixed dental prostheses, inlay-retained bridges, adhesion, acrylic resin denture teeth, dental material.

TIIVISTELMÄ

Leila Perea Mosquera

Kuitulujitteiset hammassillat – välihampaan materiaalien ominaisuuksien parantaminen

Turun yliopisto, Lääketieteellinen tiedekunta, Hammaslääketieteen laitos, Biomateriaalitieteen oppiaine, Kansallinen suun terveystieteiden tohtoriohjelma – FINDOS, Annales Universitatis Turkuensis, Turku, Suomi, 2015.

Kuitulujitteinen muovi (engl. fiber-reinforced composite, FRC) on metalliton biomateriaalinakin käytetty materiaali, jonka eräs hammaslääketieteellinen käyttökohde on hammassiltojen runkorakenteissa. Kliininen käyttökokemus on osoittanut, että eräs kuitulujitteisen sillan heikkous on lasikuiturungon ja puuttuvaa hammasta korvaavan ns. välihampaan irtoaminen toisistaan. Tämän tutkimuksen tarkoituksena oli tutkia ja kehittää keinoja välihampaan ja lasikuiturungon välisen liitoksen lujuuden lisäämiseksi materiaaliopillisilla keinoilla. Tavoitteena oli pidentää kuitulujitteisten hammassiltojen käyttöaikaa.

Neljä koesarjaa osoittivat välihampaan kiinnityspinnan materiaalin, muodon ja pintakäsittelyn vaikutuksen sillan kestävyyteen. Ensimmäisessä tutkimusosassa selvitettiin eri materiaalien purupinnan kerrospaksuuden vaikutusta sillan kestävyyteen. Välihampaan purupintamateriaalin paksuuden lisääminen lujitti siltaa erityisesti keraamista välihammasta käytettäessä. Toisessa tutkimusosassa tarkasteltiin muovisen välihampaan kiinnityspinnan kemiallisen pintakäsittelyn vaikutusta liimasauman lujuuteen. Liimasauman lujuutta saatiin lisättyä kemiallisella käsittelyllä, joka liuotti välihampaan kiinnityspintaa. Työssä havaittiin liima-aineen monomeerien imeytymistä välihampaan pintakerrokseen.

Kolmannessa työssä kiinnitettiin huomio liima- ja pintakäsittelyaineiden monomeerien imeytymiseen kiinnityshampaan pintakerrokseen. Aineiden imeytymissyvyys määritettiin konfokaalimikroskopialla ja imeytymissyvyyden ja liimasauman lujuuden välillä havaittiin yhteys. Neljännessä tutkimusosassa mitattiin valmiin hammassillan lujuutta, ns. kuormituksen kantokykyä. Sillan välihammas oli valmistettu etukäteen muotoillusta kuorimaisesta proteesihampaasta, jonka kiinnittyminen sillan lasikuiturunkoon aikaansaatiin erilaisilla täytemuoveilla. Tuloksena todettiin, että pintakäsittelyn lisäksi välihampaan ja sillan rungon välissä olevalla täytemuovilla on keskeinen merkitys sillan kestävyydelle.

Yhteenvetona todettiin, että kuitulujitteisen hammassillan kestävyyttä voitiin lisätä vaikuttamalla välihampaan materiaaliin ja sen kiinnittymiseen sillan lasikuiturunkoon. Kliiniseltä kannalta tuloksia voidaan käyttää hyödyksi valmistettaessa pitkäikäisiä kuitulujitteisia hammassiltoja.

Avainsanat: Kuitulujittenen muovi, hammassillat, välihammas, lujitemuovi, sidostaminen, liimaaminen, komposiitti, proteesihammas

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ABBREVIATIONS

ANOVA	Analysis of variance
bis-GMA	Bisphenol glycidyl methacrylate
bis-MEPP	2,2-Bis (4-methacryloxypolyethoxyphenyl) propane
˚C	Degree celsius
CAD/CAM	Computer-aided design/Computer-aided manufacturing
E-glass	Electrical glass
FDP	Fixed dental prostheses
FEPA	Federation of European producers of abrasives
FRC	Fiber-reinforced composite
HEMA	Hydroxyethylmethacrylate
IPN	Interpenetrating polymer network
min	Minute
MMA	Methylmethacrylate
MPa	Megapascal
µm	Micrometer
N	Newton
n	Number of specimens per group
s	Second
PMMA	poly(methylmethacrylate)
PE	Polyethylene
PEEK	polyetheretherketone
PU	polyurethane
SEM	Scanning electron microscopy
Semi-IPN	Semi-Interpenetrating polymer network
SD	Standard deviation
TEGDMA	Triethylene glycol dimethacrylate
UDMA	Urethane dimethacrylate
UHMWPE	Ultra-high-molecular weight polyethylene
UTMA	Urethane tetramethacrylate

LIST OF ORIGINAL PUBLICATIONS

This thesis is based on the following original publications, which are referred to in the thesis text by the Roman numerals I-IV.

I. **Perea L.**, Matinlinna JP., Tolvanen M., Lassila LV., Vallittu PK. Fiber-reinforced composite fixed dental prostheses with pontics of various kinds. J Adhes Dent 2014 Apr;16(2):161-8. doi: 10.3290/j.jad.a30755

II. **Perea L.**, Matinlinna JP., Tolvanen M., Lassila LV., Vallittu PK. Monomer priming of denture teeth and its effects on the bond strength of composite resin. J Prosthet Dent 2014;112:257-66. doi: 10.1016/j.prosdent.2014.02.019

III. **Perea L.**, Matinlinna JP., Tolvanen M., Mannocci F., Watson T., Vallittu PK. Penetration depth of monomer systems into acrylic resin denture teeth used as pontics. J Prosthet Dent. 2015 May;113(5):480-7. doi: 10.1016/j.prosdent.2014

IV. **Perea L.**, Matinlinna JP., Tolvanen M., Vallittu PK. Fracture behavior of pontics of fiber-reinforced composite fixed dental prostheses. Dent Mater J 2015. doi: 10.4012/djm.2015-081

1. INTRODUCTION

The development of metal-free prosthetic alternatives that meet the increasing demand for esthetic restorations is currently the focus of significant research. The introduction of fiber-reinforced composites (FRC) for restoring missing teeth has enabled the materialization of fixed dental prostheses (FDPs) that offer tooth structure saving, pleasing and cost-effective treatment approaches. Technical complications such as loss of retention or fracture of the materials used as framework are some shortcomings generated by the disproportions between biomechanical stress and material properties in the oral cavity. These drawbacks have been related with some of the alternatives that for many years have been used for replacing teeth. Consequently, a framework material with lower modulus of elasticity than rigid options should reduce the stress at the tooth-restoration interfaces, reducing some of the technical complications commonly seen.

Fiber-reinforced composites are acknowledged by their excellent mechanical properties, which have allowed their applications in diverse fields in dentistry, as is the case of prosthodontics applied in fixed dental prostheses. The mechanical properties of FRC restorations can decline overtime and non-desirable events such as delamination of the veneering composite, debonding and discoloration might be manifested. *In vitro* studies, as well as some reports on the clinical performance of FRC FDPs have shown that framework design, flexural properties and load-bearing capacities of these kinds of prostheses have improved (Behr *et al*., 2005; Dyer *et al*., 2005; van Heumen *et al*., 2008).

Aspects as the fiber orientation, fiber geometry, aspect ratio of the fiber, thickness of the connectors, and good interfacial adhesion between the FRC framework and the veneering material, are of great importance for the final behavior of FRC FDPs and their longevity. Emphasis has been made also on the relevance that the provision of enough amounts of fibers at the pontic area has on the contribution of support for the veneering material. However, our knowledge is still limited on the effect that different materials used as pontics, and the pretreatment of their surfaces may have on the behavior of FRC FDPs as a way to elucidate clinically relevant parameters. Therefore, the work described in this thesis focuses on the improvement of the materials that are suitable to be used as pontics in FRC FDPs.

2. REVIEW OF LITERATURE

2.1 Fixed dental prostheses

Fixed dental prostheses (FDPs) conventionally made with a metal framework represent a primary treatment in dentistry for replacing missing or lost teeth, which have shown a successful clinical performance for many years (Christensen, 2005). These types of prostheses have been the first treatment option for clinicians, who consider them as durable and reliable restorative solutions (DeBacker *et al*., 2006). Some studies have reported the longevity of FDPs with high-noble alloys, showing survival rates of 80 to 98% after 5 years (Glantz *et al*., 1984; Tan *et al*., 2004; Pjetursson *et al*., 2004); 81 to 97% after 10 years (Randow *et al*., 1986; Laurell *et al*., 1991); and 74 to 85% after 15 years (Walton, 2002). Further, excellent long-term results on the success of metal ceramic restorations have been reported, documenting that 79% of the FDPs remained unaffected, and only 3% needed to be replaced after a period of 18 to 23 years (Palmqvist & Swartz, 1993). Other reports also show a survival rate of 74% after 15 years (Creugers *et al*., 1994), as well as a similar one estimating the survival rate at 75% after 15 years (Scuirra *et al*., 1998).

Despite the outstanding reports associated to the metal ceramic FDPs, there are some complications that have been also reported, to mention some, caries has been proclaimed as the most frequent type of failure in FDPs (Randow *et al*., 1986; Walton *et al*., 1986). Loss of abutment vitality, loss of the reconstruction due to periodontitis (Pjetursson *et al.*, 2007; Behr *et al*., 2014), root fractures (Karlsson, 1989) and fracture of the abutments (Walton, 2003) have been reported as biological complications related to FDPs. Furthermore, technical problems, for instance loss of retention due to fracture of the luting cement, fracture of the framework (Pjetursson *et al.*, 2007) and porcelain fractures (Libby *et al*., 1997; Behr *et al*., 2012) are drawbacks related to them. The incidence of toxic effects of the FDPs due to its metal content is not high, however, some reports show allergic reactions for gold-containing and base-metal alloys (Schmalz & Garhammer, 2002; Mjör & Christensen, 1993).

The increasing preference for materials that provide higher esthetic-cosmetic values than the metal-ceramic restorations has driven the prosthetic dentistry into different ways. All-ceramic restorations and materials manufactured by the CAD/CAM systems are a popular alternative thanks to their excellent biocompatibility and pleasing esthetics (Raigrodski, 2005; Donovan, 2008). Yttrium oxide-stabilized tetragonal zirconium dioxide polycrystal (Y-TZP) ceramics, referred to as zirconia, provides new treatment options due to its ability for phase transformation and crack propagation, (Rekow *et al*., 2011).

Restorations made of zirconia provide an improvement on the flexural strength and fracture toughness of all-ceramic dental prosthetic solutions. FDPs made of zirconia have proved to be able to withstand the occlusal stress that occurs in the oral cavity localized in the posterior region. Consequently, they seem to be a reliable alternative to metal ceramic restorations (Bachhav & Aras, 2011; Sailer *et al*., 2007a). Some clinical reports demonstrate high survival rates for FDPs made of zirconia ranging from 92 to 100% followed during 1 to 5 years (Sailer *et al*., 2007b; Heintze & Rousson, 2010). Some disadvantages have been related to zirconia, the most common are chipping or facings failure (Raigrodski, 2005; Raigrodski *et al*., 2012; Donovan, 2008). It is noticeable the differences on the reports related to the incidence of chippings, offering a range that goes from 0% after 2 years, to 54% after 1 year (Heintze & Rousson, 2010; Sailer *et al*., 2009). Observational periods of more that 5 years are needed to have a better understanding of the suitability of FDPs made of zirconia to be considered as a reliable alternative for metal-ceramic restorations (De Backer *et al*., 2008). A few reports provide observational periods of 5 years or more (Schley *et al*., 2010; Bachhav & Aras, 2011; Raigrodski *et al*., 2012). A report that include an observational period of 10 years indicate a survival rate of 67%, a fracture rate of the veneering of 32%, and caries presented in 27% of rehabilitation solutions (Sax *et al*., 2011).

2.2 Dental ceramics

All-ceramic restorations can be divided in two categories: oxide and silicate ceramics (Figure 1). These ceramic materials are produced with cores of glass ceramics, aluminum oxide, or zirconium oxide, being manufactured by heat pressing, slip-casting, milling or sintering (Conrad & Seong, 2007).

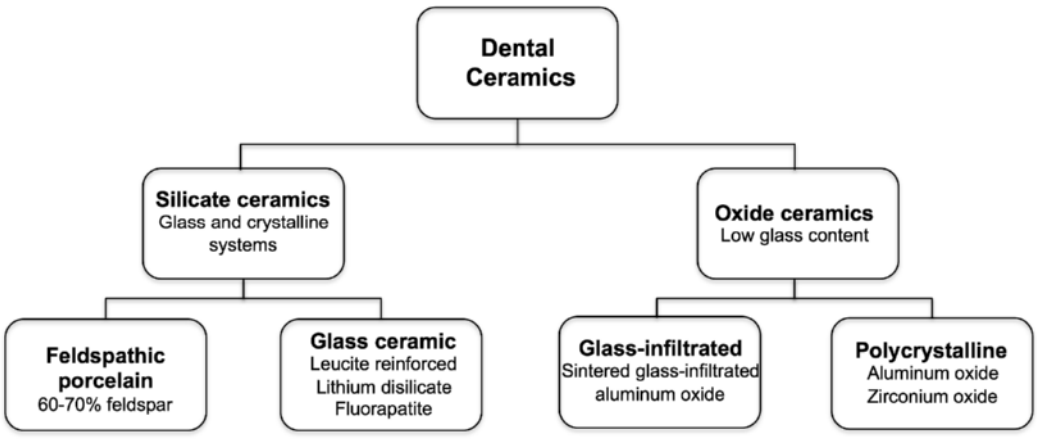

Figure 1. Classification of dental ceramics.

A summary of the ceramic materials with the clinical indications is shown in Table 1.

Table 1. Ceramic materials, systems and clinical indications.

Core Material	System	Clinical Indications
Glass Ceramic		
Lithium disilicate	IPS Empress II (Ivoclar Vivadent)	Crowns, anterior FDPs
	IPS e.max Press (Ivoclar Vivadent)	Onlays, ¾ crowns, crowns, FDPs
Leucite	IPS Empress (Ivoclar Vivadent)	Onlays, ¾ crowns, crowns
	Optimal Pressable Ceramic (Jeneric Pentron)	Onlays, ¾ crowns, crowns
	IPS ProCAD (Ivoclar Vivadent)	Onlays, ¾ crowns, crowns
Feldspathic	VITABLOCS Mark II (VITA Zahnfabrik)	Onlays, ¾ crowns, crowns, veneers
	VITA TriLuxe Bloc (VITA Zahnfabrik)	Onlays, ¾ crowns, crowns, veneers
	VITABLOCS Esthetic Line (VITA Zahnfabrik)	Anterior crowns, veneers
Alumina		
Aluminum oxide	In-Ceram Alumina (VITA Zahnfabrik)	Crowns, FDPs
	In-Ceram Spinell (VITA Zahnfabrik)	Crowns
	In-Ceram Zirconia (VITA Zahnfabrik)	Onlays, ¾ crowns, crowns
	Procera (Nobel Biocare)	Crowns, posterior FDPs
Zirconia		
Yttrium tetragonal zirconia polycrystals	Lava (3M ESPE)	Crowns, FDPs
	Cercon (Dentsply)	Crowns, FDPs
	DC-Zirkon (DCS Dental AG)	Crowns, FDPs
	Denzir (Decim AB)	Onlays, ¾ crowns, crowns
	Procera (Nobel Biocare)	Crowns, FDPs, implant-abutments

Silicate ceramics are glass-based materials with crystalline fillers, commonly leucite or lithium disilicate. These types of ceramics are superb to mimic the translucency of natural teeth. Oxide ceramics contain around 15% of silica with little or no glass phase (Kern, 2009). Due to their stable chemical structure, oxide ceramics have good mechanical properties (Rizkalla & Jones, 2004) and are called “high strength core ceramics” (Ho & Matinlinna, 2011). However, brittleness, crack propagation, low tensile strength, and their abrasiveness that affect the antagonist natural teeth are some disadvantages related to dental ceramics (van Dijken & Hasselrot, 2010; Peumans *et al*., 2004).

2.2.1 Glass ceramics

Glass ceramics are materials that consist of at least one glass phase and one crystalline phase. Due to their composition, glass ceramics provide the materials properties of both, glass and ceramics, contributing with good mechanical properties such as flexural strength and toughness (Albakry *et al*., 2004; Hill *et al*., 2000; Kelly *et al*., 1989). Leucite-reinforced glass ceramics has been used since 1990 in the fabrication of crowns and veneers, being limited to single unit restorations in the anterior zone due to its strength (Fradeani & Redemagni, 2002). This type of ceramic contains leucite, $KAlSi_2O_6$, as the main crystal phase, and it was developed from the SiO_2-Al_2O_3-K_2O-Na_2O chemical system, providing a crystal content of approx. 25%-30% by volume (Matinlinna, 2013). This type of ceramic has been used as a compound of IPS Empress® (Höland *et al*., 2007).

Lithium disilicate ceramics, another glass ceramic material, contain lithium disilicate crystals, $Li_2Si_2O_5$, as the main crystal phase, providing a crystal content of approx. 60%

by volume (Höland *et al*., 2007). Lithium disilicate ceramics (IPS Empress II, Ivoclar Vivadent) were introduced in 1998 as a glass ceramic system for single restorations and anterior three-unit FDPs that can be extended to the second premolar (Höland *et al*., 2000). Although their success (Valenti & Valenti, 2009), survival rates in the range of 50% after 2 years (Taskonak & Sertgoz, 2006) and 70% after 5 years (Marquardt & Strub, 2006) were revealed for bi-layer FDPs.

Consequently, in 2005 IPS e.max Press (Ivoclar Vivadent), which also consist of a lithium disilicate pressed glass ceramic, was made available to the public, offering enhanced mechanical and optical properties. One of its characteristics is a higher translucency that enabled this glass ceramic to be used in restorations in the posterior zone using staining characterization (Stappert *et al*., 2006). In addition, a survival rate of 88% after 10 years is reported for monolithic posterior three unit FDPs (Kern *et al*., 2012). More recently, in 2006, IPS e.max CAD (Ivoclar Vivadent), a computer-aided design/computer-aided manufacturing (CAD/CAM) manufactured version of lithium disilicate glass ceramic was created.

Lithium disilicate glasses, a monolithic restorative material, have a flexural strength higher than leucite-reinforced ceramics, which has been reported in the range of 350 MPa to 450 MPa (Della Bona, 2009). These blocks are available in a pre-crystallized blue state. It displays a flexural strength of 130 ± 30 MPa. In this blue state the ceramic can be processed, followed by the recrystallization in a ceramic oven at 850 ˚C for 25 min. The exposure of the ceramic to this heat allows the dissolution of the metasilicates, resulting in a crystallized and glazed lithium disilicate material, changing also the blue color that characterized it (Culp & McLaren, 2010). This material has been suggested to be used in inlays, veneers, crowns in the anterior and posterior zone, and in implant supported crowns (Tysowsky, 2009).

2.2.2 Alumina-based ceramics

In-Ceram Alumina was introduced in 1989 as an all-ceramic material suitable for single units and 3 units FDPs in the anterior zone (Haselton *et al*., 2000). It is sintered at 1120˚C for 10 hours, followed by a second firing at 1100˚C for 4 hours to remove porosity, increase strength and diminish potential crack propagation (Xiao-ping *et al*., 2002). Feldspathic porcelain then is used for veneering the coping (Haselton *et al*., 2000). In-Ceram Spinell was later introduced in 1994 with the aim of offering an alternative to the opaque core of In-Ceram Alumina. This introduced material has magnesia and alumina to increase translucency (Hefferman et al., 2002), but due to its lower flexural strength than the one offered by In-Ceram Alumina, the cores are recommended only for anterior crowns (Magne & Belser, 1997).

Another modification of the In-Ceram Alumina system is In-Ceram Zirconia. An addition of 35% partially stabilized zirconia oxide to the composition, which strengthen the

ceramic (Sundh & Sjogren, 2004). Due to the core's opacity and lack of translucency, this material is recommended for restorations in the posterior zone and frameworks of FDPs (Sundh & Sjogren, 2004). Procera (Nobel Biocare), that contains 99.9% high purity aluminum oxide (Andersson & Oden, 1993), has the highest strength of the alumina-based materials, strength that is overtaken only by zirconia (Oden *et al*., 1998)

2.2.3 Zirconia

Zirconia was introduced in dentistry with the aim of providing a suitable and esthetic alternative for the traditional restorative materials (Kelly & Denry, 2008). Its mechanical properties and the ease of manufacturing it in the green stage using CAD/CAM technology are some of the reasons for its clinical acceptance and popularity. This material is in a monoclinic phase at a room temperature, becomes tetragonal above 1,170˚C and experiments a third phase, cubic, above 2,370˚C (Subbarao, 1981). It has been a topic of discussion the volume increment that zirconia has in the reversible tetragonal to monoclinic phase transformation. Event that can be prevented by stabilizing tetragonal zirconia at room temperature via allowing with various oxides, which exhibits excellent mechanical properties. The stress induced phase transformation (tetragonal-monoclinic) that is also characterized by a 4.5% volume increase, inhibits the crack propagation, enhancing the toughness of the material (Garvie *et al*., 1975). This phenomenon is called transformation toughening (Piconi & Maccauro, 1999). Further, it is known that zirconia has the highest flexural strength (900-1200 MPa) and fracture toughness (9-10 MPa m^{-1}) between the dental ceramics available at present (Christel *et al*., 1989; Chai *et al*., 2007).

Chipping of the veneering porcelain has been addressed as one of the main drawbacks that zirconia exhibits, however, this issue seems to be diminished by the adoption of slower heating and cooling rates recommended by the manufacturers (Benetti *et al*., 2014). In order to maintain the stability of zirconia, attention has been given to the quality of the surface state in monolithic zirconia (Aboushelib *et al*., 2008). Grinding or sandblasting is associated with the surface phase transformation to monoclinic, bringing as a consequence the development of compressive stresses. Polishing may eradicate the compressive stress but may not remove some micro-craters created by grinding. Additionally, polishing raise the sensitivity to low temperature degradation (Deville *et al*., 2006). Heat treatment after grinding or sandblasting has been reported as a suitable procedure to reverse the phase transformation and eradicate the compressive stresses in the case of low temperature degradation (Denry *et al*., 2010). For instance the use of 650˚C during 1 min has been reported as capable of reversing the transformation with success (Denry *et al*., 2010).

Glazed zirconia is potentially more abrasive than polished zirconia (Janyavula *et al*., 2013; Kontos *et al*., 2013) due to the roughness of the unpolished ceramic surface that is exposed once the glazed is worn (Heintze *et al*., 2008). A study has reported 100% enamel cracks in teeth that are in contact with zirconia (Stawarczyk *et al*., 2013). This said, it is important

to make certain that when monolithic zirconia is used, a very careful surface polishing should be made if grinding adjustments are needed, with the intention of avoiding rougher surfaces that would increase the wear of the antagonist natural teeth (Mitov *et al*., 2012).

2.3 CAD/CAM technology

Computer-aided design/computer-aided manufacturing (CAD/CAM) technology has been introduced as an option for obtaining restorations esthetically improved, using relatively short time in the dental office, what has increased its popularity over the past 30 years (Duret *et al*., 1988, Beuer *et al*., 2008). The all-ceramic prosthetic alternatives made either using the traditional laboratory procedures, or by the implementation of the CAD/CAM technology are in demand (Christensen *et al*., 2006). Alternatives made with CAD/CAM systems are gaining the acceptance of many clinicians due to the unpredictability that the traditional methods may represent, in addition of being time-consuming (Li *et al*., 2014). Further, industrially manufactured blocks offer good homogeneity and CAD/CAM restorations have demonstrated favorability when compared with other restorative alternatives (Hickel & Manhart, 2001; Manhart *et al*., 2004).

Improved fitting accuracy is one of the main technical advantages that the CAD/CAM technology offers compared with traditional manufacturing processes that use press or casting techniques (Tank *et al*., 2004). When evaluating the efficiency of the CAD/CAM technology implemented in prosthetic dentistry is necessary to consider possible inaccuracies from scanning process, designing, milling, and shrinkage effects. These imprecisions may lead to poor restoration fit creating a cervical gap. Decreasing the cervical gap in prosthetic restorations reduces the incidence of periodontal complications, diminishes the rate of cement dissolution, and lowers the recurrence of caries (Rossetti *et al*., 2008). At the same time, it improves the mechanical strength and retention of a ceramic restoration (Thompson & Rekow, 2004).

In vitro studies have reported mean cervical gaps of 64-83 μm in all ceramic single restorations made using CAD/CAM (Sulaiman *et al*., 1997), with comparable values (64-74 μm) for zirconia multiunit frameworks (Tinschert *et al*., 2001). It has been reported also that the fit of CAD/CAM generated crowns is less precise in the internal regions than at the marginal zone (Bornemann *et al*., 2002). The scanning resolution of the measuring system of the CAD/CAM may have an influence on the way the edges of the restoration appear, making them look slightly rounded. The consequence of this is a premature contact at the incisal/occlusal edges. To overcome that, in many cases the technician has to correct that to fit the crown, what results in internal gaps (Pfeiffer, 1999). Another situation that can be presented in the process of scanning during the use of the CAD/CAM technology is the appearance of “overshooters”, that misrepresent

virtual peaks near the edges, resulting also in an increased internal discrepancy of the final restoration (Pfeiffer, 1999).

Despite the aspects reported in the literature related to some slight imprecisions that the CAD/CAM technology has, it is also claimed that this technology offers restorations that meet the requirements to have good clinical performance when homogeneous standardized materials are used (Tinschert *et al*., 2004).

2.4 Dental resin composites

Resin composites are extensively used due to their adhesive and esthetic properties, which position them as one of the first treatment options in preventive approaches, restorative dentistry and minimally invasive dental treatments. The composition of dental composites has changed significantly during the past decades offering important improvements in the material (Figure 2).

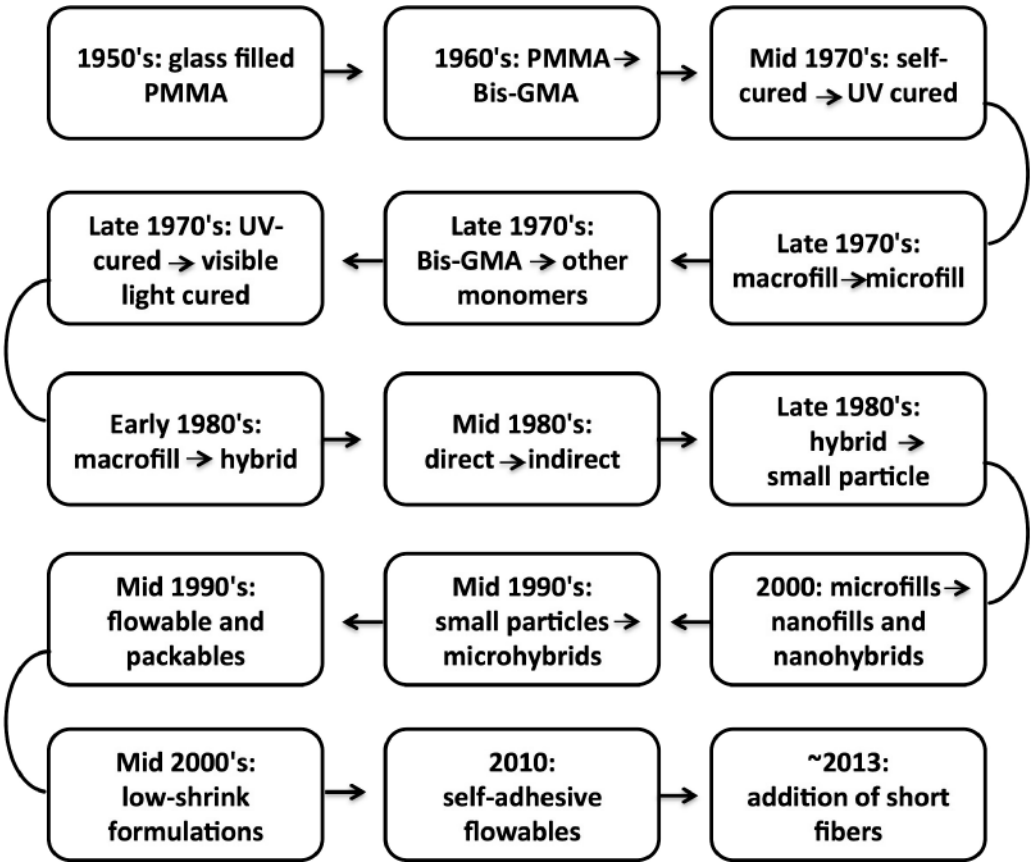

Figure 2. Overview of the evolution of dental composites.

Dental composites are conformed by a polymeric matrix, reinforcing fillers, stabilizers, initiators and activators that contribute with the light-polymerization of the organic matrix to conform cross-linked polymer networks. Additionally, silane coupling agents are involved to bond the fillers to the polymer matrix (Klapdohr & Moszner, 2005; Lung & Matinlinna 2012).

The majority of dental composites commercially available involve bisphenol glycidyl methacrylate (bis-GMA) as their organic matrix, which is highly viscous. Due to the viscosity of bis-GMA it is needed to dilute it using more fluid monomers (Peutzfeldt, 1997). Triethylene glycol dimethacrylate (TEGDMA), which is less viscous, is used as a diluent for bis-GMA and urethane dimethacrylate (UDMA). UDMA and ethoxylated bisphenol-A-dimethacrylate (bis-EMA) have less shrinkage than TEGDMA, for that reason, the utilization of TEGDMA in some dental composites has been replaced by UDMA and bis-EMA with the aim of reducing shrinkage and aging (Yap *et al*., 2000).

The structural and compositional changes that dental composites have experienced have been made with the aim of improving their strength, toughness and shrinkage due to the prevalence of caries and fracture as the primary reasons for replacement of these kinds of dental materials (Sarret, 2005). With regard to flexure, compression and tension, it has been reported that dental composites currently available are nearly as strong as dental amalgams and porcelain, excluding ceramic systems with high strength. New materials have been considered to be included in the composition of dental composites with the aim of increasing their flexural strength, which is the case of whiskers (Xu *et al*., 2003). Short E-glass fibers have been also used in dental composites with the aim of reinforcing its structure to make them suitable to be used in high stress bearing areas particularly in the posterior zone (Garoushi *et al*., 2007). Some reports in this aspect show improved load-bearing capacities, flexural strength and fracture toughness of the reinforced dental composites with the addition of E-glass fibers (Garoushi *et al*., 2011).

The inclusion of dental composites as a material of choice in restorative dentistry is and will continue increasing. Their properties, ease of handling and accessible cost, are some of the reasons that position these materials as a leading choice in many countries. Further, clinical studies confirm their good performance, reporting outcomes from 10 to 20 years with rather low annual failure rates of approx. 2% (Gaengler *et al*., 2001; Pallesen & Qvist, 2003; DaRosa *et al*., 2006).

2.4.1 Fiber-reinforced composites

Fiber-reinforced composites (FRCs) are a group of non-metallic biomaterials that were first used in dental applications in the early 1960s (Smith, 1962). Since then, it has been

used in a variety of disciplines, such as removable and fixed prosthodontics (Narva *et al*., 2001; Vallittu, 1997a; Freilich *et al*., 1998), root canal posts (Lassila *et al*., 2004; Manocci *et al*., 2005), periodontal splints (Freilich & Goldberg, 1997), orthodontic treatments (Freudenthaler *et al*., 2001), and as orthopedic and craniofacial implants (Tuusa *et al*., 2007; Tuusa *et al*., 2008; Mattila 2009).

Fiber-reinforced composites are formed by a polymer matrix reinforced with fibers, which are continuous or discontinuous (Schwartz, 1996). The physical properties of this kind of reinforced composite vary between the most durable phase, which is the fiber, to the weakest phase, the polymer. The purpose of the fibers is to transfer the load from the polymer matrix to the reinforcing fibers (Kolbeck *et al*., 2002). The polymeric matrix functions as a linker to hold the fibers together in a composite structure (Zhang & Matinlinna, 2012). Other functions of the matrix are transferring the stress between the fibers, protecting the fibers from mechanical abrasion, and serving as a barrier against the adversities of the environment (Mallick, 1997). Most FRCs used in dental applications are manufactured with glass fibers owing to their surface chemistry, which benefit their adhesion to the polymer matrix (Freilich *et al*., 1998).

Glass fibers have been used for reinforcing interim restorations, as periodontal splints, in removable prostheses, single crowns and as a component of fixed partial dentures (Vallittu, 1998a). They are a non-crystalline and three-dimensional structure of silica, oxygen, in addition to other atoms (Chang & Chai, 2003). For dental applications, polyurethane, polycarbonate, and acrylic base polymers, such as bisphenol-A glycidyl methacrylate (bis-GMA) and poly(methylmethacrylate) (PMMA) are essentially reinforced with glass fibers and silanized to increase the chemical adhesion between the fibers and the polymer matrix (Uctasli *et al*., 2005).

E-glass (electrical glass) and S-glasses (silica glasses) fibers are the most frequently used type of fibers. These are durable glasses that provide a chemical stability in the pH range 4-11 (Norström *et al*., 2001). Carbon/graphite fibers have been considered to be used in restorative dentistry, but their dark color represents a limitation for their clinical use. Ultra-high-molecular weight polyethylene fibers (UHMWP) have shown some difficulties in adhering the fibers to the resin matrix, which makes them more prone to be colonized by oral microbes (Tanner *et al*., 2000).

A number of publications have demonstrated the relationship between the quantity of fibers in the polymer matrix and the improvements in strength (flexural, impact and transverse) of FRCs (Vallittu & Narva, 1997; Isaac, 1999; Behr *et al*., 2000). By augmenting the quantity of fibers, the flexural strength and modulus of elasticity increases linearly (Furtos *et al*., 2012).

Continuous FRC include unidirectional and bidirectional design. Unidirectional glass fibers consist of 1,000 to 200,000 single glass fibers (Vallittu, 1998a). Unidirectional longitudinal fibers provide their reinforcing aspect to FRC restorations when the stress is applied perpendicularly to the direction of fibers; in other words, unidirectional fibers are anisotropic (Vallittu 2015). On the contrary, woven fibers have their reinforcing properties in two directions and are orthotropic (Vallittu, 1999). The Anisotropic properties of FRC are an important aspect in designing dental prosthetic solutions, considering that the masticatory forces produce stresses that include bending, shear, tensile, compression and torque (Tezvergil *et al*., 2003).

2.4.1.1 Adhesive interface

The transference of load from the polymer matrix to the reinforcing fibers occurs through the adhesive interface, which positions this interface as a key aspect for the durability of FRCs (Bouillaguet *et al*., 2006). A chemical bond is achieved between the polymer matrix and the exposed glass fibers due to the use of silane coupling agents. Some studies report the capabilities of silanes to increase the surface wettability of the glass fibers, resulting in chemical bridges formation and an increased physical attachment of the resin to the surface of glass (Goracci *et al*., 2005). Some other aspects have an effect on the durability of FRC restorations, for instance, the properties of the fibers; resin matrix; the quantity of fibers; the direction, orientation, distribution, construction and position of the fibers and the impregnation of the fibers with the resin (Pensler *et al*., 1997).

Two types of resins are used in FRCs, forming a cross-linked (thermoset) polymer matrix, or a linear (thermoplastic) polymer matrix. Multifunctional or dimethacrylate resins are involved in the formation of the cross-linked matrix, contrary to the non-cross-linked polymer matrix where monofunctional methacrylates are included (Väkiparta *et al*., 2007). A combination of thermoset and thermoplastic resins has been used as impregnation methods. In this case, the polymer matrix is a semi-interpenetrating polymer network (semi-IPN), where a cross-linked polymer and a linear polymer are merged (Kallio *et al*., 2001). Cross-linked polymer matrix results in FRCs with higher modulus of elasticity than the achieved by thermoplastic or semi-IPN polymers (Lassila *et al*., 2002). Additionally, higher toughness is one of the advantages that thermoplastic and semi-IPN polymer matrices contribute over FRCs manufactured from highly cross-linked thermosets. The semi-IPN polymer matrix of FRC provides some benefits over cross-linked dimethacrylate regarding its handling properties and the adhesion of indirectly made restorations to resin luting cements and veneering composites (Kallio *et al*., 2001).

Polyethylene (PE), polyetheretherketone (PEEK), polyacetal (PA), and polyurethane (PU) are examples of thermoplastics that are used in medical applications. Examples

of thermosets are epoxy polymer, bis-GMA, and TEGDMA copolymer (Tuusa *et al*., 2007).

2.4.1.2 Impregnation of fibers

Resin impregnation allows an optimal reinforcement and transfer of stresses from the polymer matrix to the reinforcing fibers (Vallittu, 1998b). Effective impregnation is a requirement to achieve a good contact between the matrix and each fiber, which is often performed using various monomers. Some of these monomers are bis-GMA, UDMA, urethane tetramethacrylate (UTMA), poly(methylmethacrylate) (PMMA) or triethylene glycol dimethacrylate (TEGDMA) (Lastumäki *et al*., 2003). The use of only monomers for the impregnation of fibers brings as a consequence a high polymerization shrinkage, which may affect the mechanical properties of the FRC (Vallittu, 1996). Then, pre-impregnated FRC systems have been developed, which facilitates the handling properties.

Deficiencies in the impregnation of fibers increase the water sorption in FRC, reducing their strength and modulus of elasticity (Miettinen *et al*., 1999). The use of high viscosity resin systems and the polymerization shrinkage of the resin can have as consequence an inadequate degree of impregnation of fibers (Vallittu 1995a; Vallittu 1995b). This can facilitate water sorption through voids leading to decreased mechanical properties of FRC (Miettinen & Vallittu, 1997). In addition, voids originated due to poorly impregnated fibers serve as oxygen reservoirs that inhibit the radical polymerization of the polymer matrix (Vallittu, 1999). Pre-impregnated FRCs also show good mechanical properties due to the reduction in voids and cracks in its composition, which can limit water sorption. Water sorption due to poor impregnation can have detrimental consequences in the bond strength and lead to hydrolytic degradation of polysiloxane network of FRC (Miettinen & Vallittu, 1997).

2.4.1.3 Mechanical properties

Mechanical properties in the matter of strength, toughness, stiffness and fatigue resistance rely upon the geometry of the reinforcement. The provision of strong fiber-resin interfaces contributes to greater static and fatigue properties. Increased hardness and tensile strength are influenced by the incorporation of silanated filler particles of fiber (Debnath *et al*., 2004). The effectiveness of the fiber reinforcement, as described on the Krenchel's factor, differs according to the length and fiber orientation. The reinforcing efficiency factor for the fiber reinforcement goes against the known direction of stress (Krenchel 1963). Many other parameters contribute to the reinforcing efficiency of fibers, between them interfacial adhesion, fiber volume fraction and elongation of fibers (Murphy, 1998).

Fiber-reinforced composites are classified as short discontinuous and long continuous FRCs. They show differences in their mechanical properties even when their fiber volume fraction is similar (Kardos 1993). For instance, if continuous unidirectional fibers are replaced by longitudinally oriented discontinuous short fibers of lower aspect ratio, a reduction of the ultimate tensile strength of the composite will be the result (Vallittu, 2015). Here is when the isotropicity-anisotropicity of the material plays a role. Continuous and discontinuous FRCs are anisotropic, however, they become isotropic when the orientation of short fibers is random, with the outcome of reducing the tensile strength.

In a polymeric structure the provision of fiber reinforcement increase its modulus of elasticity or stiffness and toughness (Wright *et al*., 1997). The modulus of elasticity (*E*) is a measure of the stiffness, defined by the slope of the stress-strain curve linear segment before plastic deformation. According to that, a direct influence exists between the *E* and the stiffness, providing that the higher the *E*, the stiffer the material will be. The greater the *E*, the less the elastic deformation will be as a resultant of the stress application. Toughness is the ability of a material to absorb energy and have the capacity to plastically deform without fracturing (Callister, 1991). Some investigations have been focused on the effects of fracture toughness and *E* on FRCs. One of them found that the *E* of particulate reinforced composites increases when cross-sectional designs included 1 or more fiber reinforcements located at the compression side (Dyer *et al*., 2005).

2.4.1.4 Clinical relevance of FRC FDPs

The consideration of FRC FDPs to be used as a reliable prosthetic solution is justified by their advantageous elastic modulus compared with metal, and improved adhesion of the composite luting agent to the framework (Vallittu & Sevelius, 2000). Some reports question the benefit of FRC prostheses due to its anisotropicity, which does not strengthen the restoration in all directions, contrasting with their metal counterparts (Jokstad *et al*., 2005). Nonetheless, some *in vitro* studies have reported that fiber reinforcements have a positive influence in the mechanical properties of a resin composite due to an increment in its fracture strength, which justify the clinical use of this material (Ellakwa *et al*., 2002; Alander *et al*., 2004; van Heumen *et al*., 2008). Furthermore, besides the *in vitro* studies, some clinical reports elucidate the benefits of FRC FDPs (Vallittu, 2004; Göhring & Roos, 2005), but long-term clinical studies in this matter are lacking.

The main drawbacks related to FRC FDPs have been reported as delamination of the veneering composite, wear and debonding. These are some of the reasons given by some authors (Jokstad *et al*., 2005; Edelhoff *et al*., 2001; Behr *et al*., 2003) for questioning the

use of FRC FDPs of that time as a long-term prosthetic solution, concluding that these kinds of prostheses need further improvements to overcome the related disadvantages.

Location, type and length of the FRC FDPs have an influence on their survival rates (Prösber & Henrich, 1997; Creugers *et al*., 1998). Some reports show that FRC FDPs with a long span and located in the posterior zone had a similar or even higher behavior than prostheses with only one pontic located in the anterior zone (Vallittu & Sevelius, 2000), concluding that if enough vertical space is available and the amount of fibers can be increased, in addition to good periodontal conditions, FRC bridges can be made even with two or three pontics.

3. AIMS OF THE THESIS

The current series of studies were performed to investigate ways of enhancing the mechanical and physical properties of fiber-reinforced composites fixed dental prostheses, by improving the materials used as pontics, to then heighten their longevity. Thus, the working hypothesis was that by changing the material or modifying the adhesive properties of the pontics, the durability of FRC FDPs could be improved.

The following specific aims were set to:

1. Evaluate the influence of a variety of materials used as pontics and their characteristics in the load-bearing capacities of fiber-reinforced composites fixed dental prostheses.

2. Investigate the effect of the chemical surface treatment of acrylic resin denture teeth on their surface hardness and shear bond strength to a composite resin.

3. Analyze the dissolving capabilities and penetration depth of different monomer systems on acrylic resin denture teeth.

4. Assess the fracture behavior and load-bearing capacities of pontics made of pre-shaped acrylic resin denture teeth in combination with different composite resins.

4. MATERIALS AND METHODS

The materials used for the fabrication of the specimens in study I-IV are listed in Table 2.

Table 2. Materials used in the fabrication of the specimens in studies I-IV.

Brand	Manufacturer	Composition	Study
Palapress	Heraeus Kultzer	Powder: PMMA-copolymer Liquid: MMA, dimethacrylate, butanediol dimethacrylate, alcoxylated polyoltetraacrylate, additives	I, II
Artic 8	Heraus-Kulzer	PMMA, dimethacrylate	I, II, III
Vitapan Cuspiform	Vita	PMMA, dimethacrylate	II, III
GC (experimental teeth)	GC	PMMA, dimethacrylate	III
Creopal	GC	Occlusal layer: UDMA with fillers. Bonding layer: PMMA	IV
everStick C&B	Stick Tech-GC	PMMA, bis-GMA, E-glass fibers	I, IV
IPS Empress CAD	Ivoclar Vivadent	SiO_2, Al_2O_3, K_2O, Na_2O, CaO	I
IPS Ceramic etching gel	Ivoclar Vivadent	Hydrofluoric acid	I
Monobond-S	Ivoclar Vivadent	Alcohol solution of silane methacrylate, phosphoric acid methacrylate and sulphide methacrylate	I
G-ænial Anterior	GC	Methacrylate monomers; pre-polimerized fillers: silica, strontium; inorganic fillers: silica, fumed silica	I
G-ænial Posterior	GC	Methacrylate monomers; pre-polimerized fillers: silica, strontium; inorganic fillers: silica, fumed silica	IV
G-ænial Universal Flo	GC	UDMA, bis-MEPP, TEGDMA, Silicon dioxide, Strontium glass	IV
everX Posterior	GC	bis-GMA, PMMA, TEGDMA, short E-glass fiber filler	IV
Stick Flow	Stick Tech-GC	bis-GMA, TEGDMA	I, II, III
Stick Resin	Stick Tech-GC	bis-GMA, TEGDMA	I, II, III, IV
Scotchbond Universal	3M ESPE	bis-GMA, HEMA	I, IV
Methylmethacrylate, 99%	Sigma-Aldrich	MMA	I, II
Composite primer	GC	Monofunctional methacrylate, UDMA, camphorquinone	II, III
Tetrahydrofuran	Sigma-Aldrich	Tetrahydrofuran	II
Rhodamine B	Sigma-Aldrich	Dye content 97%	III

4.1 Study I

4.1.1 Fabrication of the fiber-reinforced composite fixed dental prostheses

Inlay preparations were made in a polymer phantom model where a situation of a missing first molar was replicated (Figure 3).

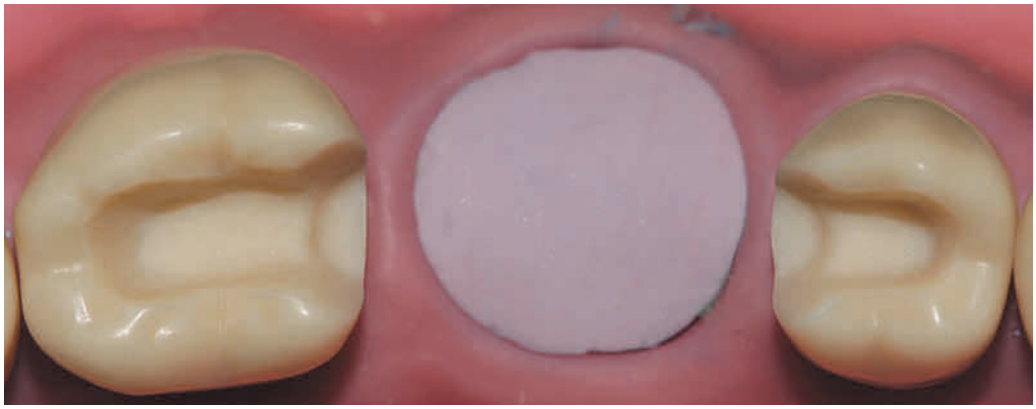

Figure 3. Inlay preparations made in a phantom model.

Inlay-retained FRC FDPs were made using two continuous unidirectional E-glass fiber reinforcements attached with a flowable composite. Three positions were chosen to vertically curve the FRC frameworks that allowed the fabrication of pontics with three different thicknesses (2.5 mm, 3.2 mm, and 4.0 mm) (Figure 4).

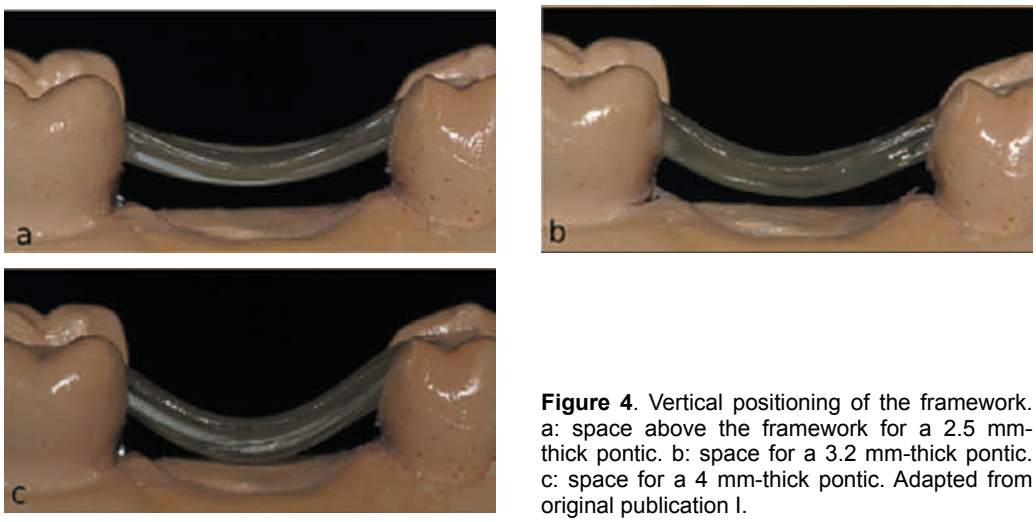

Figure 4. Vertical positioning of the framework. a: space above the framework for a 2.5 mm-thick pontic. b: space for a 3.2 mm-thick pontic. c: space for a 4 mm-thick pontic. Adapted from original publication I.

An index made of a composite resin was made and attached to the working model to standardize the bending of the framework (Figure 5). A 1.5 mm-thick index was used to make a framework that allowed the fabrication of a 3.5 mm-thick pontic measured in the gingival-occlusal direction. A 0.8 mm-thick index was made for the 2.5 mm-thick pontics and no index was used for the lowest position of the framework. In this case, the framework was curved until reaching the gingival side of the working model.

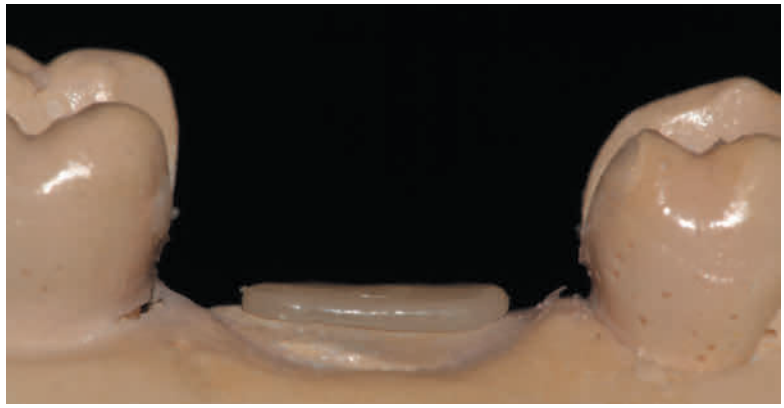

Figure 5. Index used to control the vertical positioning of the framework.

Three groups were formed based on the material used for the fabrication of the pontics: composite resin, acrylic resin denture teeth and leucite-reinforced glass ceramic blocks. Each group was subdivided according to the pontics thicknesses (n=8 per subgroup). A Cerec-3 unit was used to scan the fiber frameworks to design the ceramic pontics with a space on their gingival side for positioning the frameworks (Figure 6).

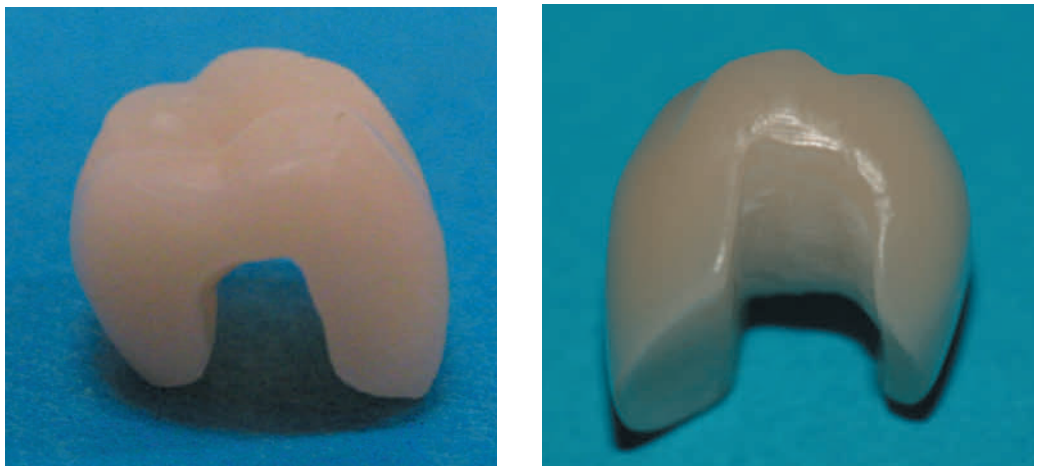

Figure 6. A space on the gingival side of the milled ceramic pontic for positioning the fiber framework.

An acrylic denture tooth was scanned by Cerec to reproduce its shape on the ceramic pontic. The shape of the ceramic pontic was mirrored on the composite pontic by using a silicone mold. In that way, standard pontics in the three different materials were achieved.

In the composite resin group additional transversal fiber reinforcement was used (Figure 7). Next, the FDP was made using the same composite resin.

Figure 7. An additional transversal fiber reinforcement used in the composite resin group.

The gingival side of the polymer denture teeth was modified to fit the framework according to its vertical position. A flowable composite was subsequently used to attach the pontic to the framework, followed by the addition of composite resin to complete the FDP. The gingival surfaces of the ceramic pontics were etched, silanated and bonded to the framework by using a bonding agent and a flowable composite (Figure 8). After that, a composite resin was used to complete the FDP (Figure 9). Each step of the fabrication of the FRC FDP was light polymerized for 40 s per side (Elipar S10, 3M ESPE, Seefeld, Germany). The irradiance was 950 mW/cm^2, as measured using curing radiometer. A zirconia model was used for cementation of the FRC FDPs to perform the fracture test.

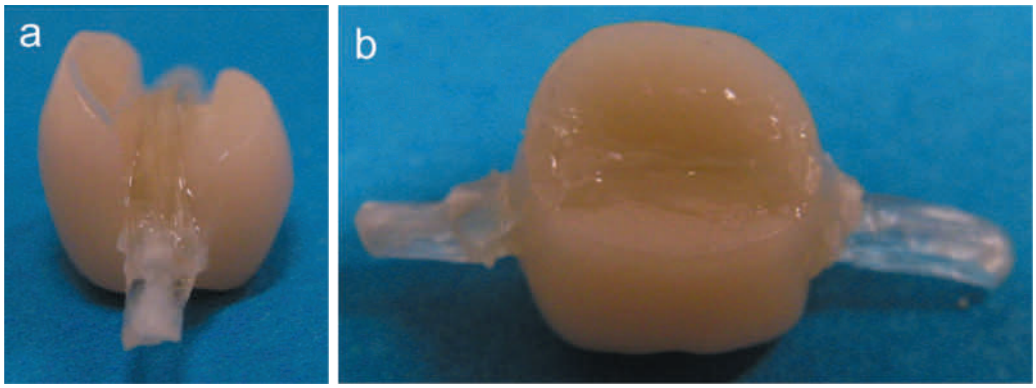

Figure 8. a: fiber framework placed in the milled ceramic pontic; b: gingival side of the milled ceramic pontic filled with composite resin.

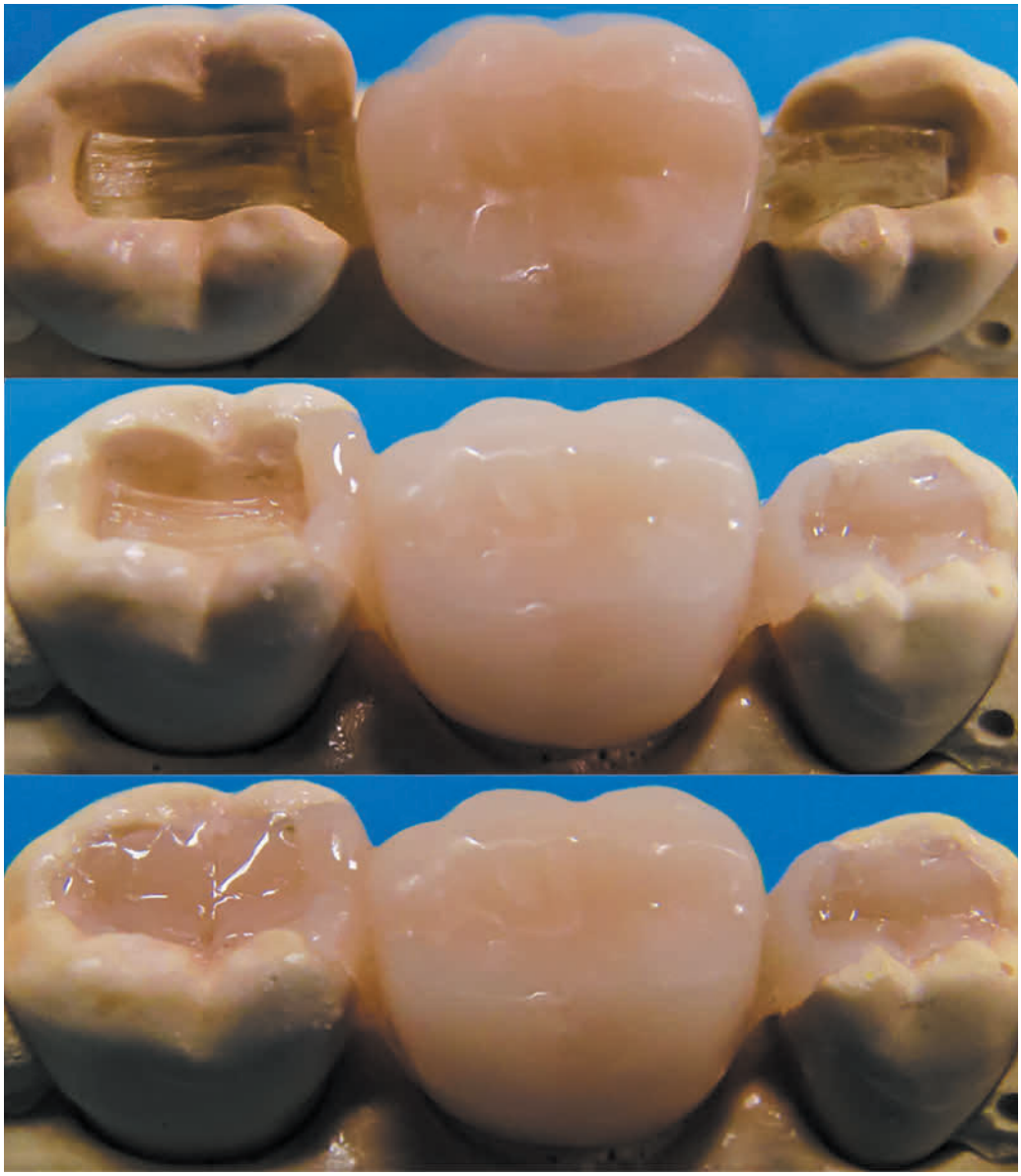

Figure 9. Completion of the FRC FDP with a ceramic pontic using a composite resin.

4.1.2 Mechanical testing

The FRC FDPs were statically loaded to failure at a crosshead speed of 0.5 mm/min (Model LR 30K plus, Lloyd Instruments; Fareham, UK). A 6mm diameter steel ball was used for the application of the load to the occlusal surface (Figure 10). The load was applied until the final fracture. The load-deflection graph was used to determine the initial fracture.

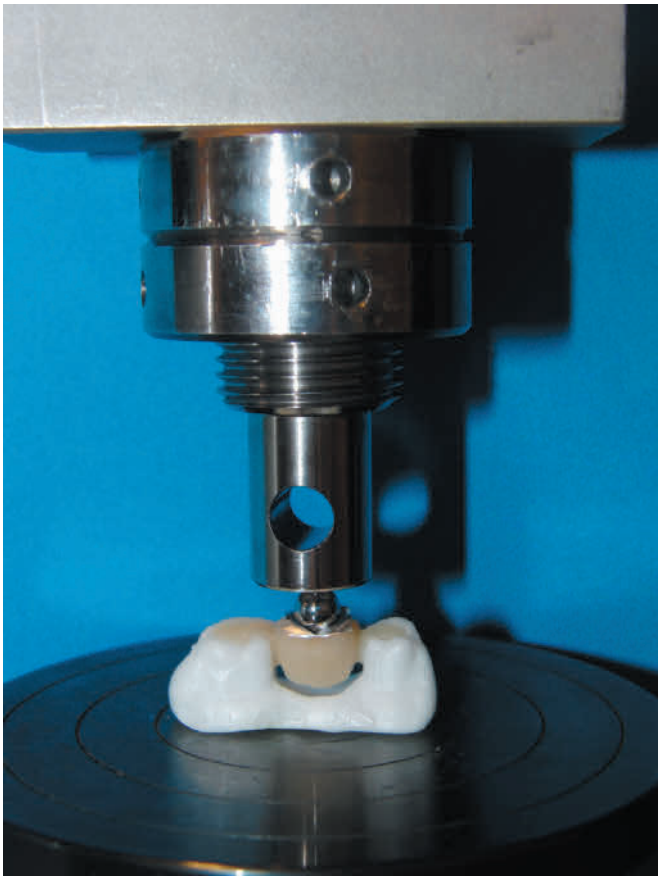

Figure 10. Static compressive fracture test.

4.1.3 Analyzing methods

The fracture mode of the FDPs were visually analyzed and determined after testing. The fracture mode was categorized as fracture of pontic with and without fiber exposure.

4.2 Study II

4.2.1 Fabrication of the substrates investigated

An autopolymerizing acrylic resin (Palapress; Heraeus Kultzer GmbH) was used as a base material to bond the acrylic resin denture teeth (Artic 8; Heraeus Kultzer GmbH and Vitapan cuspiform; Vita). A plane and even bonding surface was obtained by using a silicon carbide grinding paper (1200-grit, FEPA) on the ridge-lap surfaces of the denture teeth (Figure 11).

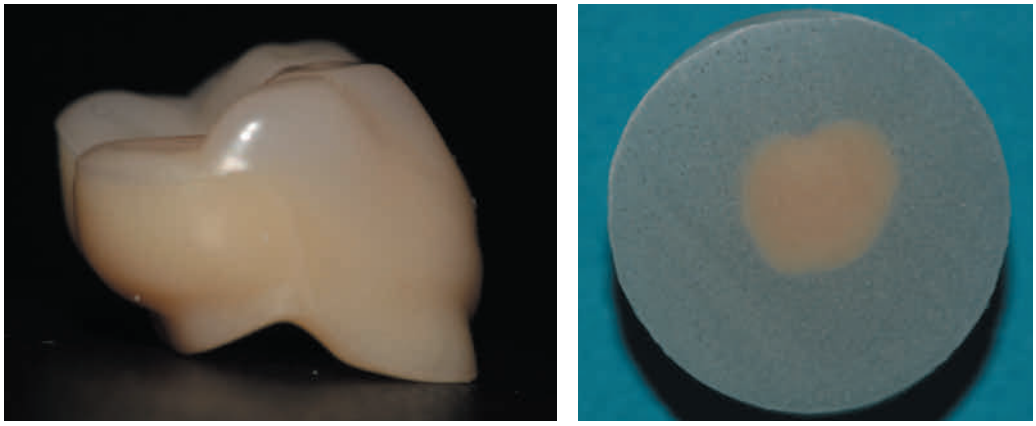

Figure 11. Modification of the ridge-lap surface of the acrylic denture teeth to obtain a flat bonding surface.

A Flowable composite (Stick Flow; Stick Tech-GC), Methylmethacrylate (Sigma-Aldrich), Composite primer (GC) and a photopolymerizing dimethacrylate resin (Stick Resin; Stick Tech Ltd.) were used for the pretreatment and conditioning of the acrylic teeth surfaces. The two brands of acrylic teeth were divided based on the four-monomer systems (n=15). They were then subdivided according to the exposure times used for the chemical pretreatment: no pretreatment (control), 1, 5, 15 and 60 min.

4.2.2 Surface microhardness and debonding test

A Vickers hardness testing machine was used to perform a surface microhardness testing on the untreated acrylic denture teeth surfaces (Duramin-5; Struers), at a 245.2 mN force for 15 s. This was done to evaluate the initial surface hardness values before a surface conditioning was carried out. Next, a flowable composite resin (Stick Flow; Stick Tech-GC) was used to prepare cylindrical shear bond test specimens with a diameter of 3.6 mm and a height of 4 mm using a polyethylene transparent mold. Light polymerization was followed for 40 s (Elipar S10, 3M ESPE, Seefeld, Germany). Twenty-four hours later a debonding testing was performed (Model LR 30K plus; Lloyd Instruments, Fareham, UK) at a 1.0 mm/min crosshead speed until failure (Figure 12). Debonding test was so-called “shear bond strength” test, where the major stress was considered to be shear stress. After this testing, new microhardness values were recorded to measure potential changes in the surface hardness of the acrylic teeth after being exposed to the light polymerization.

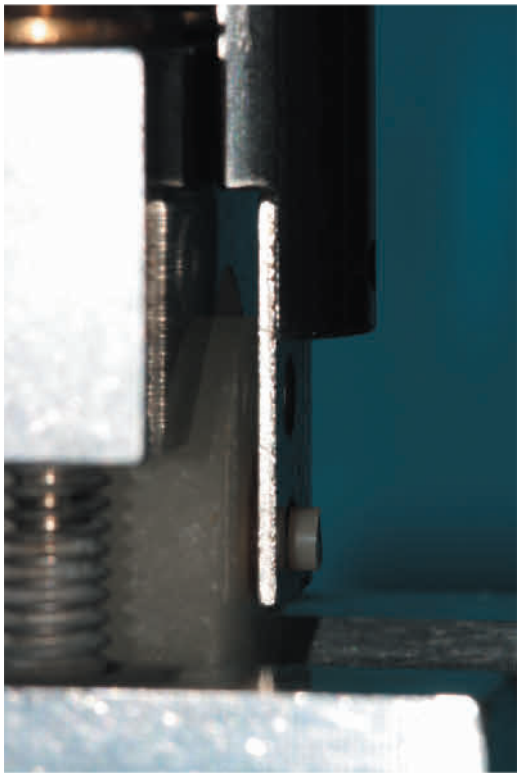

Figure 12. Debonding test.

Afterwards, the four monomers systems were used respectively as surface conditioners on the acrylic teeth selected for the remaining exposure times (1, 5, 15 and 60 min). These monomers were used unpolymerized and protected from light during each exposure time. Surface hardness values were immediately taken after the unpolymerized monomers were removed from the acrylic denture teeth surfaces. Next, the flowable composite was used to make the stub for the shear bond testing in the same way that it was described previously. Shear bond testing was performed 24 hours later followed by a new surface microhardness testing. Visual analysis was carried out to evaluate the fracture type.

4.2.3 Scanning electron microscopy

SEM (JSM 5500, JEOL Ltd., Tokyo, Japan) was used to assess the polymer structures of the acrylic denture teeth. Tetrahydrofuran (Sigma-Aldrich) was used for 20 s to identify the inner part of the polymer beads of the tooth. Specimens were then attached to metal holders using carbon tape and sputter-coated with gold. SEM images were then taken at 250x magnification.

4.3 Study III

4.3.1 Fabrication of the substrates investigated

Three brands of acrylic resin denture teeth (Artic 8, Heraeus Kultzer; Experimental teeth, GC; Vitapan, Vita) were selected and merged in an autopolymerizing acrylic resin (Palapress; Heraeus Kultzer GmbH). Flat ridge-lap acrylic teeth surfaces were obtained using a silicon abrasive paper (1200-grit, FEPA). Different monomer systems were selected to be used on the surface of the acrylic resin denture teeth: a flowable composite resin (Stick Flow; Stick Tech-GC), Methylmethacrylate (Sigma-Aldrich), Composite primer (GC) and a photopolymerizing dimethacrylate resin (Stick Resin; Stick Tech Ltd.). 1, 5, 15 and 60 min were the exposure times selected to use the monomer systems on the acrylic teeth surfaces.

The monomers were combined with a florescent dye (Rhodamine B; Sigma-Aldrich), applied to the specimens' surfaces, and left unpolymerized for the respective exposure time protected from the light. This was done to allow the monomers to dissolve and penetrate the acrylic denture teeth surfaces. A 5 min light polymerization was followed (Optilux 500; Demetron-Kerr; 450 mW/cm^2 irradiation). Each specimen was then sectioned in 4 slices of 1 mm-thick, including 8 slices per experimental group (Figure 13). 2400-grit (FEPA) silicon carbide paper was used to polish the slices surfaces.

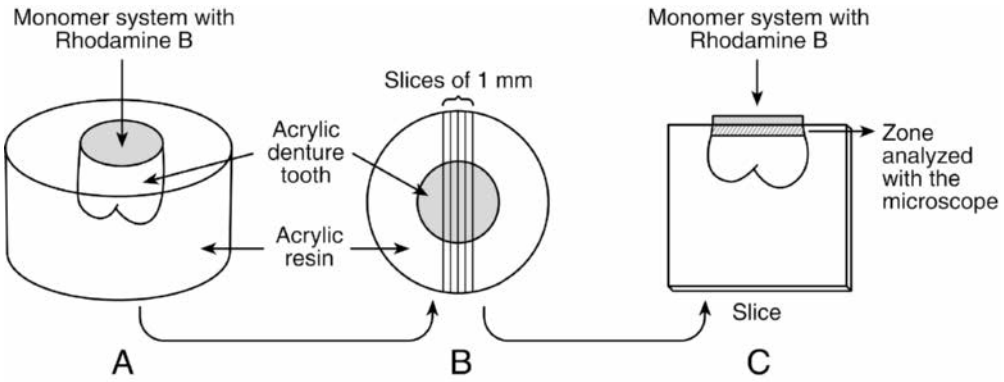

Figure 13. Schematic representation of the specimen. A: Before sectioning, B: Sectioning in 1 mm slices, C: Vertical slice. Adopted from original publication III.

4.3.2 Confocal scanning microscopy

A confocal scanning type microscope (TSM; Noran Instruments) was used to measure the penetration depth of the monomers into the acrylic denture teeth surfaces. x60 and x100 oil immersion objectives were used for the specimens examinations. Images from the deepest penetrations were taken using an electron multiplying charge-coupled device (iXon 885 EM-CCD; Andor Technology).

4.4 Study IV

4.4.1 Fabrication of the fixed dental prostheses

Inlay retained FRC FDPs were made using two bundles of continuous unidirectional E-glass fibers (everStick C&B, Stick Tech-GC; Turku, Finland), in addition to a third fiber reinforcement placed transversally in the pontic space. Acrylic resin denture teeth (Creopal, GC, Meiningen, Austria) with the shape of a shell were used for the fabrication of the pontics (Figure 14).

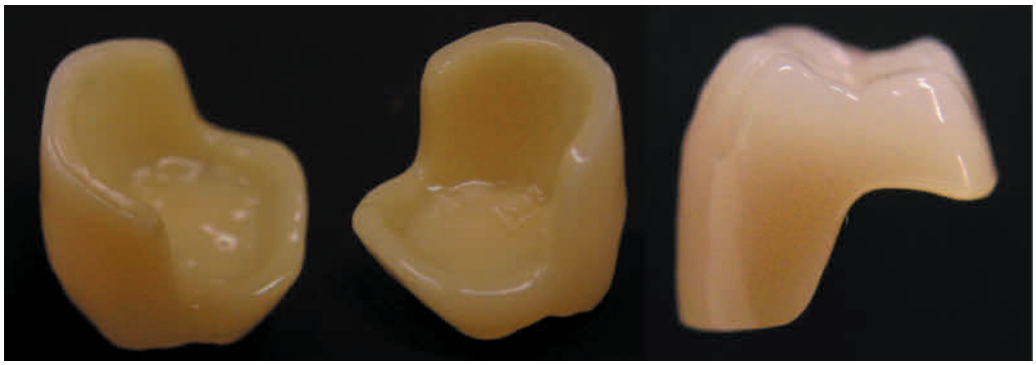

Figure 14. Shell-shaped acrylic resin denture teeth.

Three composite resins were used as filling material to fabricate the final shape of the pontics: a universal flowable composite (G-ænial Universal Flo, GC), a hybrid composite (G-ænial Posterior, GC), and a discontinuous short fiber-reinforced composite resin (everX Posterior, GC). Based on the filling material, three experimental groups were made using the FRC framework with the characteristics previously described (Figure 15a). A fourth experimental group was made with everX Posterior without the transversal fiber reinforcement in the pontic area (Figure 15b).

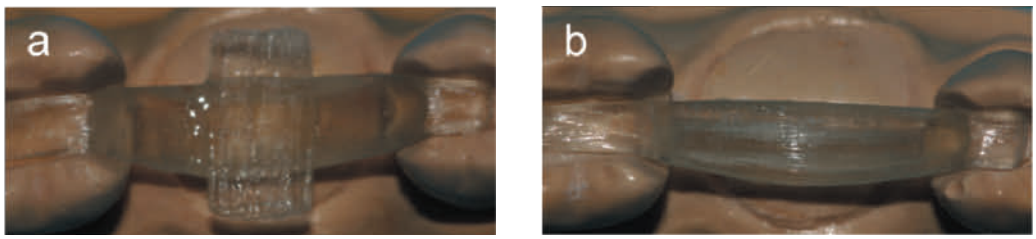

Figure 15. a: FRC framework with a transversal reinforcement. b: FRC framework without the transversal reinforcement.

A dimethacrylate resin (Stick Resin, StickTech-GC, Turku, Finland) was applied to the FRC frameworks and left unpolymerized for 5 min protected from the light to allow monomers to dissolve into the FRC (Wolff et al. 2012). Next, a light polymerization was undertaken on the FRC frameworks for 40 s (Elipar S10, 3M ESPE; Seefeld, Germany) with a light intensity of 680mW/cm^2. After that, the inlay restorations were made using a flowable composite in the cervical side of the box preparations, and a hybrid composite (G-ænial Posterior, GC) until completing the remaining box cavity.

The internal surface of the shell-shaped acrylic teeth was pretreated with a dimethacrylate resin (Stick Resin, StickTech-GC). This resin was kept unpolymerized on the acrylic teeth surface during 15 min for dissolving and penetration of the resin in the denture teeth surface. A light polymerization was made for 5 min. The shell-shaped pontics were adhered to the FRC frameworks and built their shape using the composite resin filling materials described previously.

Four groups were formed (n=21/group) and subdivided in three subgroups to perform the testing to the plane of the occlusal surface as follow: a) dry specimens at 90˚, b) dry specimens at 30˚ and c) specimens stored in water for one month and tested at 90˚. The FRC FDPs were bonded to a zirconia model and fixed to the testing device.

4.4.2 Mechanical testing

A universal testing machine (Model LR 30K plus, Lloyd Instruments; Fareham, UK) was used for the static compressive fracture test at a crosshead speed of 0.5 mm/min. 90˚ and 30˚ were used for the application of the load. The load was applied until the final fracture. The load deflection graph was used to determine the initial fracture. The fracture types were determined visually.

4.4.3 Microscopic analysis

The polymer structure of the acrylic resin denture teeth used was determined using SEM. The crack propagation of the tested FRC FDPs was also examined with SEM.

4.5 Statistical analysis

Statistical analyses were conducted using Statistical Package for the Social Sciences software (SPSS. Inc, Chicago, Illinois). The distribution of data was normal, which was determined by the Kolmogorov-Smirnov test. Therefore, parametric tests were used for testing statistically significant differences. Differences were considered significant at 95% confidence level.

In study I, the data were analyzed using ANOVA. The independent variables were material, thickness, and their mutual interaction. The dependent variables were initial and final fracture load. Curve estimation regression models were also used. In study II, statistically significant differences were evaluated with a 3-way ANOVA. The independent factors were tooth brand, monomer, exposure time, and their interactions. The dependent factors were shear bond strength and hardness before and after testing.

In study III, ANOVA was used to evaluate mean differences according to exposure times and to the acrylic resin denture teeth brand, monomer system, and their interaction. Differences between the exposure times were also evaluated by using curve estimation. In study IV, statistically significant differences between the initial and final fracture loads for each filling material, angle and storage were evaluated with a 3-way ANOVA. The independent factors were filling material, angle, storage and their interactions. The dependent factors were initial and final fracture load.

5. RESULTS

5.1 Pontic materials and thicknesses (Study I)

The materials used in pontic fabrication, their thicknesses as well as their interaction, significantly affected the load-bearing capacities of FRC FDPs ($p<0.001$). The results of testing are illustrated in Table 3. In general the ceramic pontics had the highest initial and final fracture values for pontics with thicknesses of 4.0 mm.

Table 3. Initial and final fracture loads in N (SD) for thicknesses and material.

		Material		
Fracture	**Thickness**	**Composite pontic**	**Polymer denture tooth pontic**	**Glass ceramic pontic**
Initial	2.5 mm	613 (103)	537 (139)	716 (95)
	3.2 mm	794 (145)	593 (129)	707 (76)
	4.0 mm	853 (94)	673 (174)	1331 (158)
Final	2.5 mm	832 (88)	1210 (258)	725 (80)
	3.2 mm	1021 (196)	1871 (210)	708 (76)
	4.0 mm	996 (126)	1589 (136)	1667 (147)

Using quadratic curve estimation allowed showing that in the initial fracture, thickness affected the composite and ceramic pontics, but did not have an effect on the polymer denture tooth pontics (Figure 16). At the point of final fracture, pontic thickness affected all the materials tested (Figure 17).

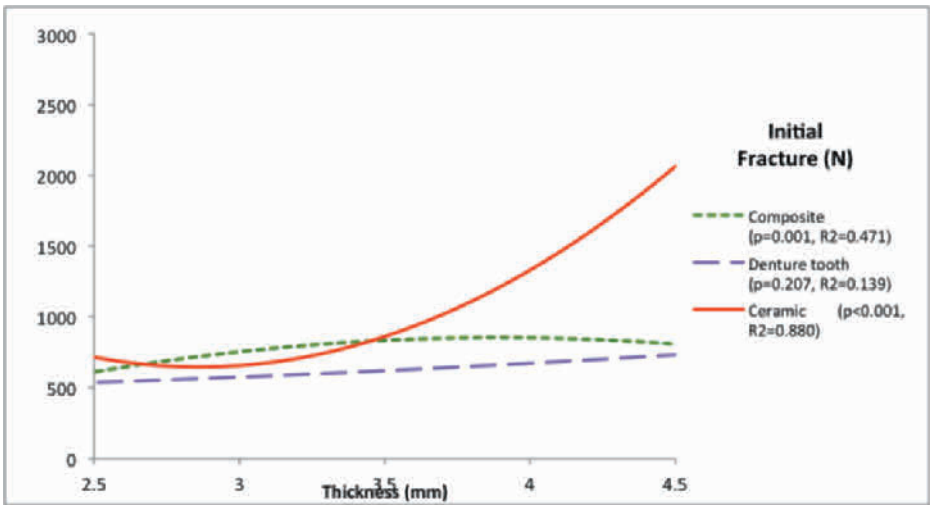

Figure 16. Estimation of quadratic curve for initial fracture in the tested specimens. Adapted from original publication I.

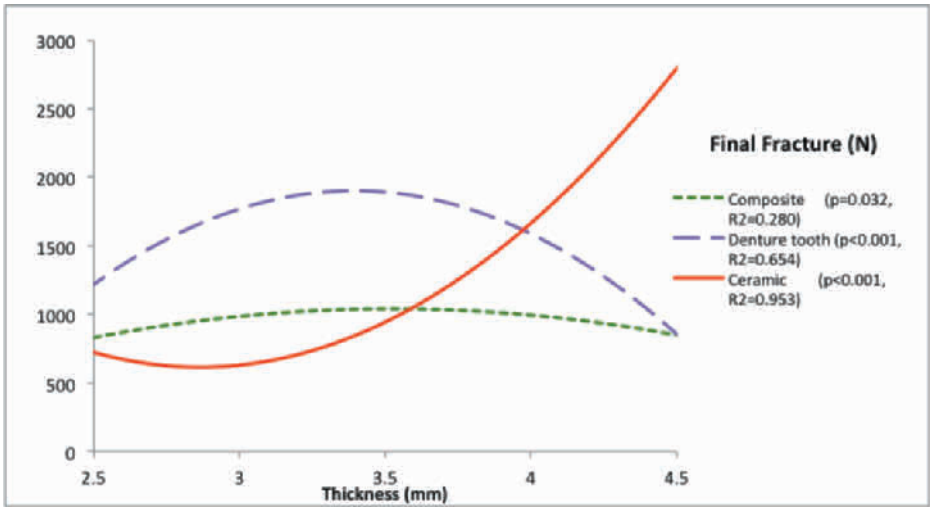

Figure 17. Estimation of quadratic curve for final fracture in the tested specimens. Adapted from original publication I.

Pontic fractures types, which exposed fibers, and those not exposing fibers were observed in the tested specimens (Table 4, Figure 18 and Figure 19). No cases of framework fracture or detachment of the framework were observed.

Table 4. Observed fracture types in fiber-reinforced composite fixed dental prostheses.

Fracture type	Composite pontic	Polymer denture tooth pontic	Ceramic pontic
Fracture without fiber exposure	23	16	16
Fracture with fiber exposure	1	8	8

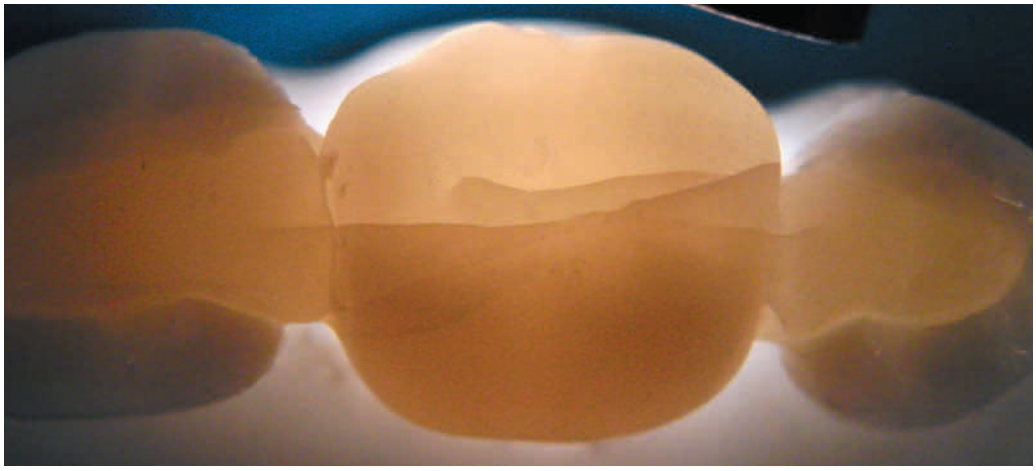

Figure 18. Fracture of ceramic pontic without fiber exposure.

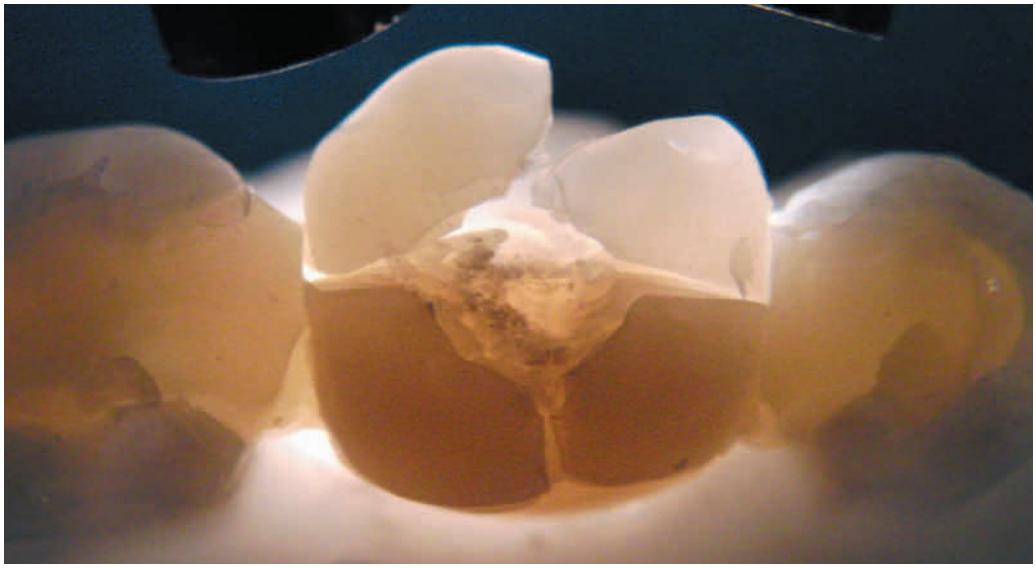

Figure 19. Fracture of ceramic pontic with fiber exposure.

5.2 Debonding stress and surface hardness measurements (Study II)

Of the selection of monomers used in surface-conditioning, Composite Primer® had the highest MPa values when it was used in Artic 8® resin denture teeth during 1 min. The best performing monomer in the Vitapan® acrylic resin denture teeth was Stick Resin® used during 15 min. Table 5 illustrates means and standard deviations for debonding stress of tested acrylic resin denture teeth.

Table 5. Debonding stress (MPa) for monomer systems and exposure times in Artic 8 and Vitapan acrylic denture teeth.

Acrylic teeth brand	Monomers	Exposure time				
		0 min	1 min	5 min	15 min	60 min
Vitapan	Stick FLOW	0.47 (0.10)	1.38 (0.16)	0.58 (0.24)	2.56 (0.93)	2.73 (0.70)
	Methylmethacrylate	0.54 (0.36)	0.75 (0.25)	0.96 (0.27)	1.42 (0.58)	1.79 (1.21)
	Composite primer	1.78 (0.61)	2.81 (0.84)	1.96 (0.72)	4.45 (1.49)	3.77 (1.12)
	Stick Resin	2.34 (1.55)	3.41 (0.98)	2.71 (0.78)	7.98 (0.22)	6.19 (1.27)
Artic 8	Stick FLOW	1.05 (0.23)	1.24 (0.26)	1.73 (0.16)	1.75 (0.43)	1.72 (0.26)
	Methylmethacrylate	1.26 (0.38)	0.94 (0.34)	1.04 (0.24)	0.67 (0.16)	1.27 (0.32)
	Composite primer	6.09 (1.99)	9.08 (1.35)	7.96 (1.35)	6.59 (1.43)	5.40 (1.70)
	Stick Resin	7.70 (2.81)	6.65 (2.05)	7.29 (1.76)	6.96 (0.71)	5.21 (0.36)

Values are presented as mean (SD)

An illustration of the maximum bond strength values from evaluated acrylic resin denture teeth as shown in Figure 20.

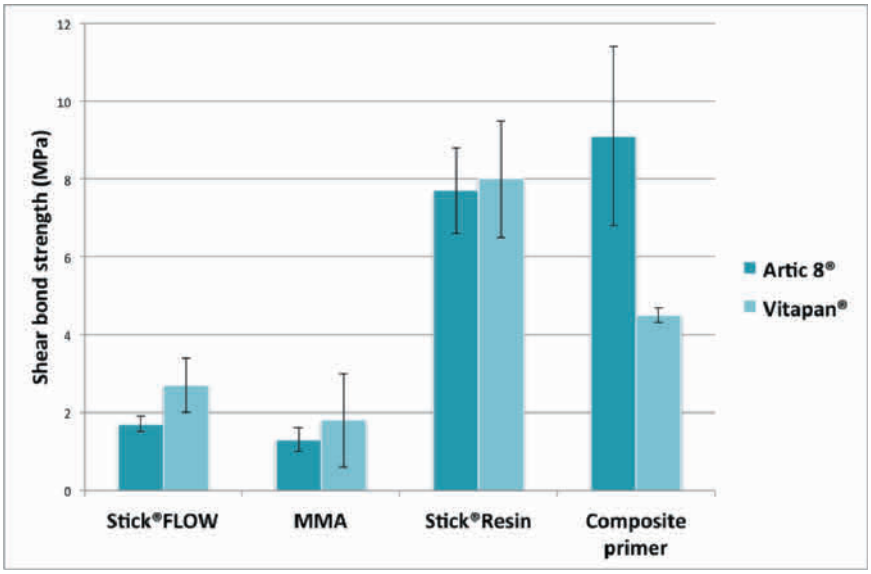

Figure 20. Maximum bond strength values (MPa) from Vitapan and Artic 8. Adapted from original publication II.

Testing revealed that the most significant changes in surface hardness of Artic 8 teeth were found in the test group where Stick Resin was used as a surface-conditioning monomer for 60 min. Similar performance was observed in Vitapan teeth using Composite Primer for 60 min. In both scenarios surface micro-hardness measures were taken prior to the shear bond test being performed. Means and standard deviations of the surface hardness of the tested acrylic resin denture teeth measured before and after the shear bond test are described in Table 6.

Table 6. Surface hardness in VHN for the monomers systems used as surface-conditioners and exposure time.

Brand	Monomer	Testing period	Exposure time				
			0 min	1 min	5 min	15 min	60 min
Vitapan	Stick Flow	B	21.3 (0.6)	20.2 (0.3)	19.4 (1.0)	20.1 (0.4)	19.6 (0.3)
		A	20.2 (0.9)	22.7 (1.3)	23.2 (1.1)	24.7 (0.2)	21.8 (0.5)
	Methylmethacrylate	B	19.6 (0.6)	19.5 (0.6)	18.4 (0.6)	19.9 (0.8)	19.2 (0.6)
		A	20.4 (0.8)	21.6 (0.5)	22.6 (1.1)	24.9 (0.6)	20.8 (0.6)
	Composite primer	B	18.7 (0.4)	18.5 (0.4)	19.9 (0.2)	20.2 (0.2)	17.6 (0.4)
		A	21.8 (0.9)	21.4 (0.5)	22.7 (0.7)	24.4 (0.5)	21.0 (0.5)
	Stick Resin	B	20.1 (0.9)	18.6 (0.5)	20.1 (0.4)	20.0 (0.9)	18.7 (0.5)
		A	22.1 (0.7)	22.2 (0.8)	23.2 (0.6)	33.1 (1.7)	23.4 (0.4)
Artic 8	Stick Flow	B	23.3 (1.2)	20.9 (0.7)	19.8 (0.3)	19.3 (0.2)	18.3 (0.9)
		A	22.8 (0.2)	22.4 (1.2)	21.5 (1.2)	22.3 (1.9)	22.8 (0.7)
	Methylmethacrylate	B	23.8 (1.5)	22.3 (0.5)	20.2 (0.7)	19.9 (0.7)	19.0 (0.9)
		A	22.9 (1.7)	23.1 (2.0)	22.6 (0.7)	23.5 (0.1)	22.8 (1.7)
	Composite primer	B	22.6 (0.4)	22.0 (0.8)	20.3 (0.9)	20.9 (0.3)	19.1 (1.4)
		A	24.0 (1.7)	22.1 (1.1)	22.8 (1.0)	23.3 (1.1)	22.2 (0.9)
	Stick Resin	B	22.9 (1.4)	21.4 (1.9)	19.4 (1.1)	18.6 (0.9)	17.3 (1.3)
		A	24.4 (1.8)	23.7 (0.6)	23.7 (1.4)	22.3 (1.2)	24.3 (1.1)

B: After monomer application and before shear bond testing; A: after shear bond testing (SD)

Graphical representation of the shear bond strength (MPa) and surface hardness (VHN) prior to and after the shear bond testing is shown in Figure 21.

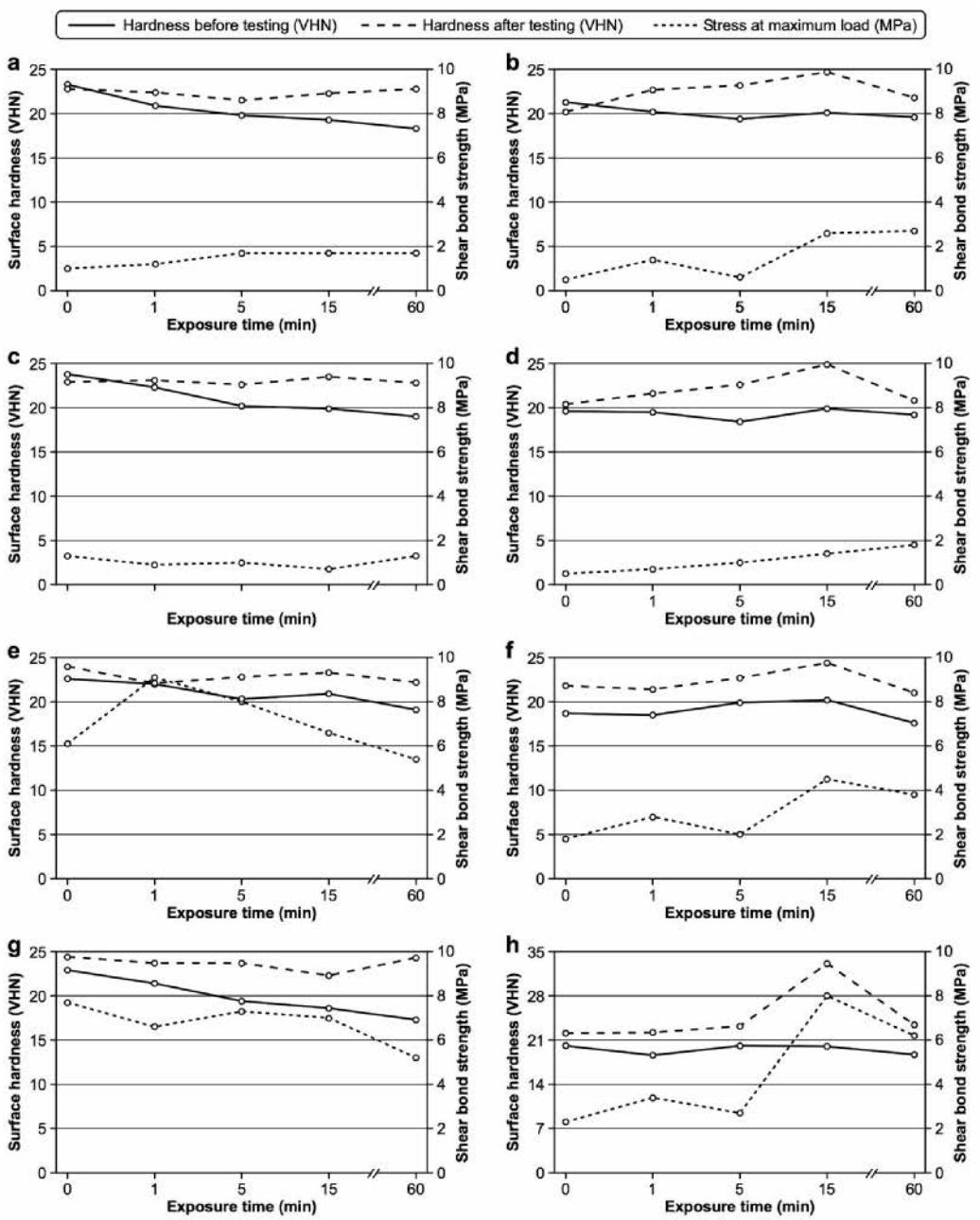

Figure 21. Shown are the debonding strength (MPa) and surface hardness (VHN) of treated surfaces before bond testing (hardness before testing) and after bond testing (hardness after testing). a, Stick Flow in Artic 8. b, Stick Flow in Vitapan. c, Methylmethacrylate in Artic 8. d, Methylmethacrylate in Vitapan. e, Composite primer in Artic 8. f, Composite primer in Vitapan. g, Stick Resin in Artic 8. h, Stick Resin in Vitapan. Adopted from original publication II.

Tooth type, monomer system, exposure time along with their two- and three-way interactions demonstrated a significant effect on the shear bond strength and surface hardness prior and after testing. This was with the exception of the three-way interaction on hardness prior to testing (Table 7).

Table 7. *P* values from three-way ANOVA for effects of tooth brand, monomer system, exposure time, in addition to their interactions on the shear bond strength and surface hardness prior and after the shear bond testing.

Variables	Shear Bond Strength	Hardness Before Testing	Hardness After Testing
Tooth brand (Artic8, Vitapan)	<0.001	<0.001	0.680
Monomer system	<0.001	0.066	<0.001
Exposure time	<0.001	<0.001	<0.001
Tooth brand × monomer system	<0.001	<0.001	0.001
Tooth brand × exposure time	<0.001	<0.001	<0.001
Monomer system × exposure time	0.026	0.013	0.001
Tooth brand × monomer × exposure time	0.013	0.365	<0.001

Adopted from original publication II

Measurements taken of surface hardness subsequent to the longest exposure time (60 min) are illustrated in Figure 22. Shown is the initial surface hardness of the acrylic denture teeth prior to any surface treatment. Also shown are the differences after exposure to the monomer systems for 60 min.

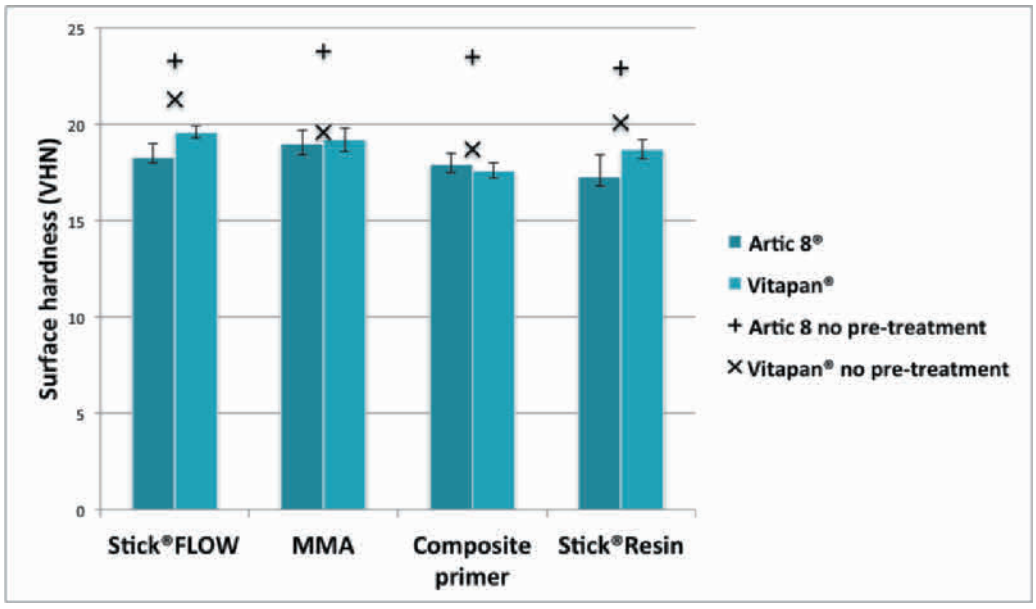

Figure 22. Measurements of surface hardness (VHN) of acrylic teeth after 60 min of being exposed to monomer systems in Artic 8 and Vitapan acrylic resin denture teeth. Adapted from original publication II.

5.3 Scanning electron microscopy evaluation (Study II)

A SEM analysis of the influence of the various monomer systems used as conditioners in the surface of acrylic denture teeth revealed differences in the structure of Artic 8 and Vitapan teeth. Among the Vitapan teeth, it was possible to view a multiphasic structure of cross-linked matrix, semi-IPN, and linear polymeric phases. These were not visible in the Artic 8 teeth (Figure 23).

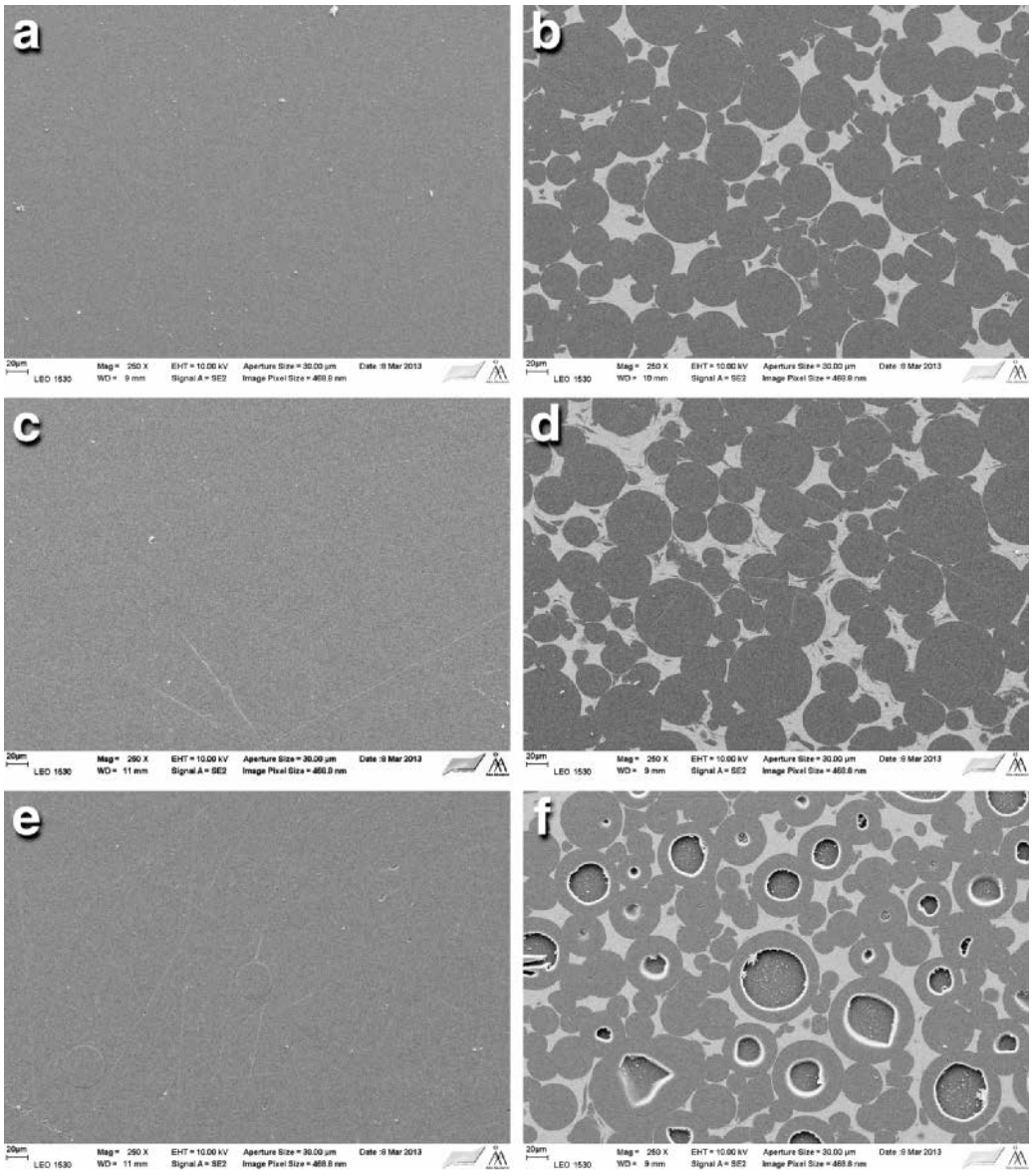

Figure 23. Shown are SEM micrographs 250x. a, Artic 8 without treatment surface. b, Vitapan without treatment surface. c, Artic 8 treated with Stick Flow. d, Vitapan treated with Stick Flow. e, Artic 8 treated with Methylmethacrylate. f, Vitapan treated with Methylmethacrylate. g, Artic 8 treated with Composite primer. h, Vitapan treated with Composite primer. i, Artic 8 treated with Stick Resin. j,Vitapan treated with Stick Resin. Adopted from original publication II.

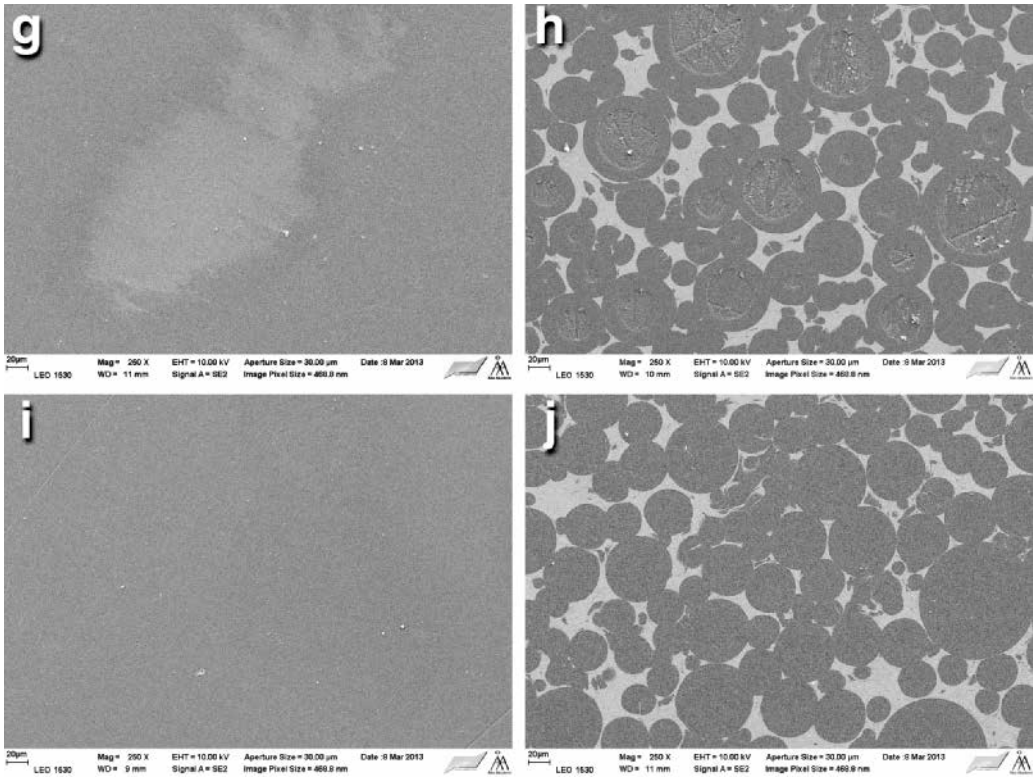

Figure 23. (Continued) g, Artic 8 treated with Composite primer. h, Vitapan treated with Composite primer. i, Artic 8 treated with Stick Resin. j,Vitapan treated with Stick Resin. Adopted from original publication II.

5.4 Penetration depth of monomer systems (Study III)

The results showed a significant association between penetration depth and acrylic resin denture tooth brands, monomer systems used on each, and time under which they were exposed. Observed differences in the monomer penetration depths into the acrylic resin denture teeth are illustrated in Table 8.

Table 8. Dissolving depths in μm (SD) of monomer systems into acrylic resin denture teeth as a function of time.

Brand	Monomer system	1 min	5 min	15 min	60 min
Vitapan	Composite Primer	1.69 (0.30)	1.64 (0.42)	1.71 (0.40)	2.09 (0.24)
	Methylmethacrylate	0.67 (0.20)	0.94 (0.48)	0.89 (0.40)	3.95 (1.12)
	Flowable composite	2.82 (0.48)	3.26 (0.63)	3.32 (0.73)	5.08 (1.21)
	Stick Resin	0.21 (0.14)	1.58 (0.23)	1.98 (0.36)	1.90 (0.29)
Artic 8	Composite Primer	1.01 (0.28)	2.74 (0.52)	2.78 (0.26)	2.78 (0.33)
	Methylmethacrylate	0.24 (0.08)	0.28 (0.10)	0.25 (0.08)	0.68 (0.25)
	Flowable composite	2.10 (0.62)	2.29 (0.35)	2.66 (0.37)	4.47 (0.71)
	Stick Resin	1.80 (0.23)	2.21 (0.28)	2.30 (0.57)	2.26 (0.28)
GC	Composite Primer	1.17 (0.36)	1.20 (0.35)	1.28 (0.31)	1.98 (0.41)
	Methylmethacrylate	0.56 (0.23)	0.94 (0.38)	1.15 (0.40)	2.15 (1.33)
	Flowable composite	2.46 (0.30)	3.77 (0.53)	3.70 (0.88)	3.72 (0.49)
	Stick Resin	1.93 (0.42)	2.53 (0.34)	2.63 (0.28)	2.65 (0.29)

The ANOVA test (R^2=0.699) indicated that there existed differences which varied according to the acrylic resin denture tooth brand (p=0.047), monomer system used (p<0.001), exposure time (p<0.001), and their interaction (p<0.001). The results indicated that the highest penetration depth value (5.08 µm) occurred in the Vitapan group while using a flowable composite resin as a monomer for 60 min.

The interaction between penetration depths and the four monomer systems by exposure time in Artic 8 is illustrated in Figure 24a. Figure 24b illustrates the results found among GC teeth, and Figure 24c shows the results for Vitapan teeth.

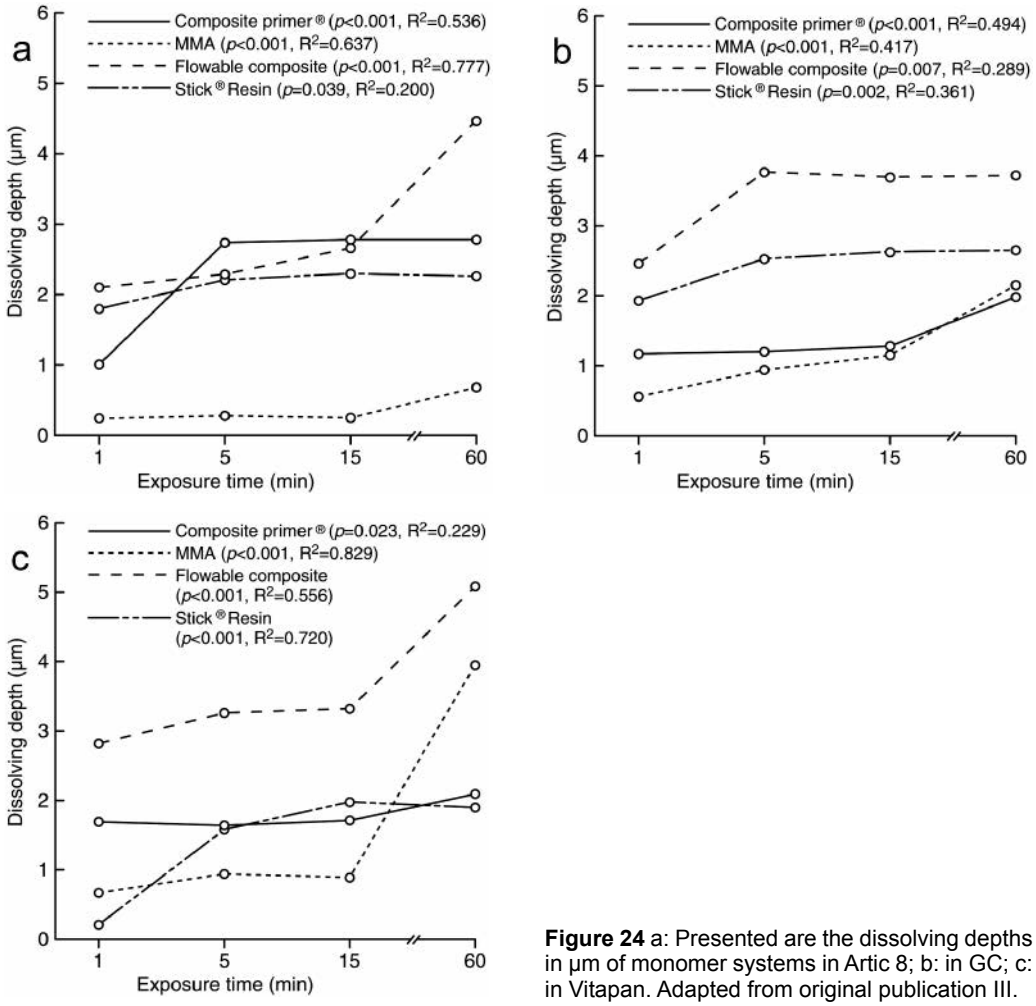

Figure 24 a: Presented are the dissolving depths in µm of monomer systems in Artic 8; b: in GC; c: in Vitapan. Adapted from original publication III.

A continuous dissolution layer of different thickness was found as a morphological pattern of interdiffusion in the test groups (Figure 25).

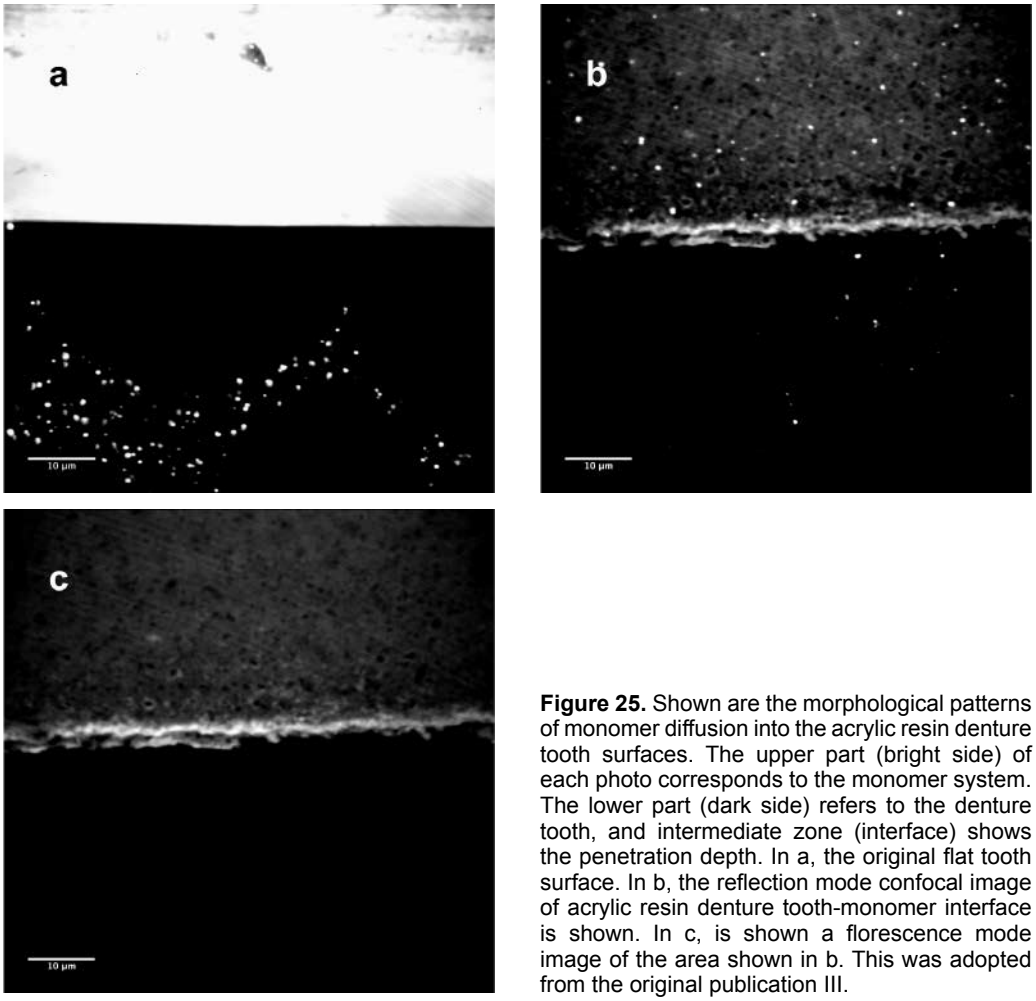

Figure 25. Shown are the morphological patterns of monomer diffusion into the acrylic resin denture tooth surfaces. The upper part (bright side) of each photo corresponds to the monomer system. The lower part (dark side) refers to the denture tooth, and intermediate zone (interface) shows the penetration depth. In a, the original flat tooth surface. In b, the reflection mode confocal image of acrylic resin denture tooth-monomer interface is shown. In c, is shown a florescence mode image of the area shown in b. This was adopted from the original publication III.

5.5 Fracture behavior of pontics (Study IV)

The ANOVA statistical test results showed significant differences in the load-bearing capacities of each tested inlay-retained FRC FDP according to the filling material ($p=0.002$), angle ($p<0.001$), but not storage ($p=0.263$). Differences which were observed in the load-bearing capacities of the tested FRC FDPs are listed in Table 9.

Table 9. Shown are the load-bearing capacities in N (SD) by filling material, angle and storage.

Filling material	Angle used for testing	Storage	Initial Fracture	Final Fracture
Flowable composite	90°	Dry	802 (97)	1635 (262)
	90°	Water	581 (180)	1569 (281)
	30°	Dry	555 (91)	1342 (264)
Hybrid composite	90°	Dry	655 (47)	1629 (191)
	90°	Water	767 (83)	1655 (148)
	30°	Dry	420 (73)	802 (192)
everX Posterior[1]	90°	Dry	1112 (169)	1710 (134)
	90°	Water	814 (129)	1739 (216)
	30°	Dry	418 (72)	1246 (102)
everX Posterior[2]	90°	Dry	897 (162)	1572 (130)
	90°	Water	758 (174)	1847 (182)
	30°	Dry	451 (68)	1291 (228)

[1]FRC framework with a transversal reinforcement in the pontic area. [2]FRC framework without the transversal reinforcement. Adopted from original publication IV

A graphical illustration of the results is shown in Figures 26, 27 and 28. FDPs which utilized everX Posterior as a filling material had the highest load-bearing capacities when tested at 90°, and G-ænial Universal Flo provided the most durable FDP when tested at 30˚.

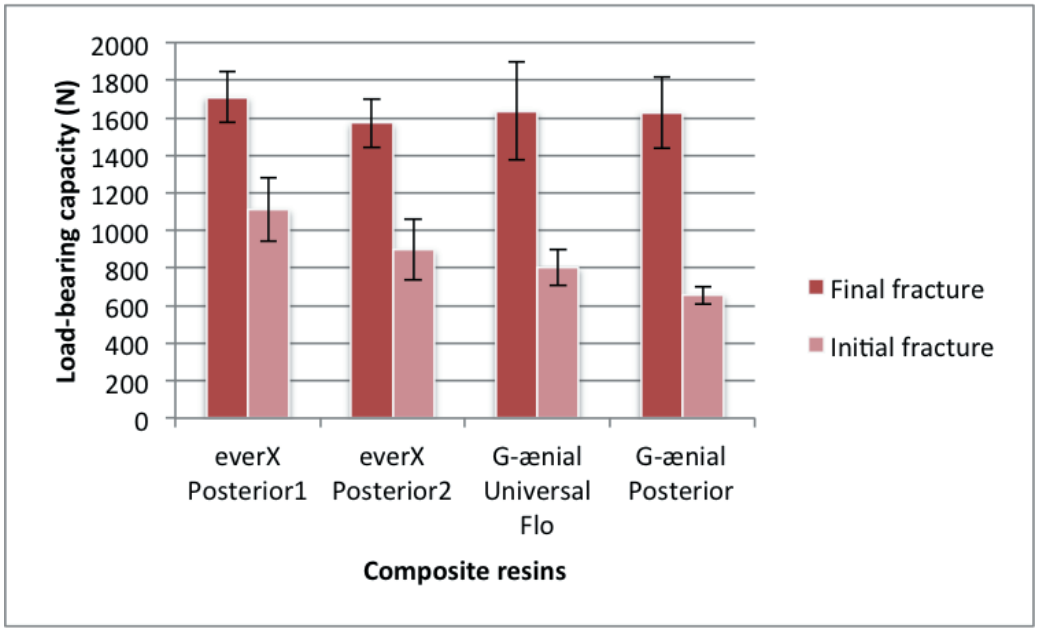

Figure 26. Load bearing capacities (N) by filling composite for specimens tested when dry at 90°. everX Posterior1:FRC framework with a transversal reinforcement in the pontic region. everX Posterior2: FRC framework without transversal reinforcement.

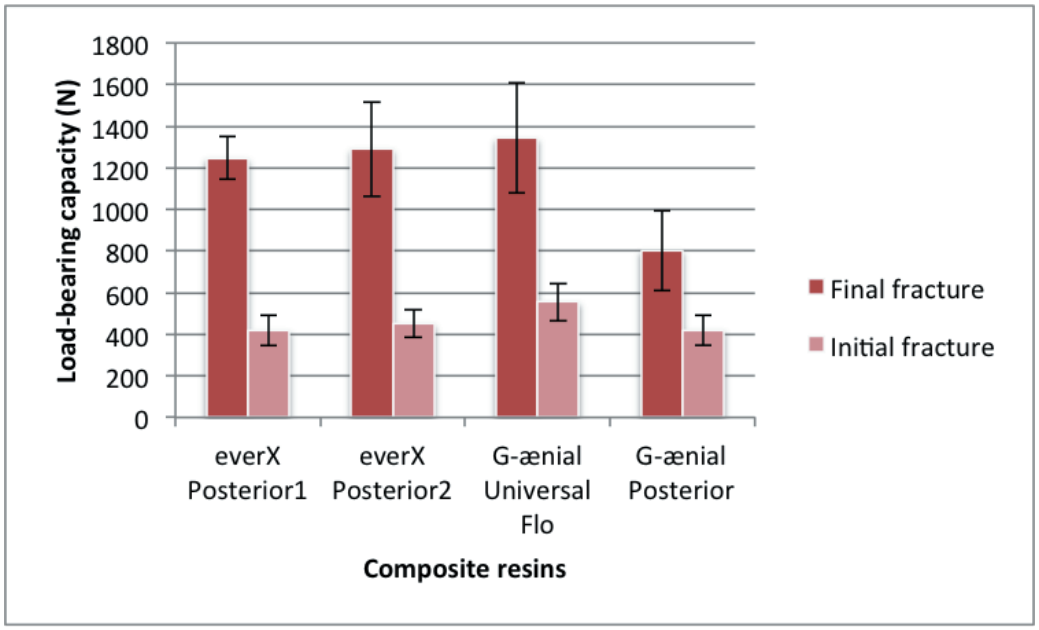

Figure 27. Shown are the load-bearing capacities (N) according to the filling composites for the specimens tested dry at 30˚. everX Posterior1:FRC framework with a transversal reinforcement in the pontic area. everX Posterior2: FRC framework without the transversal reinforcement.

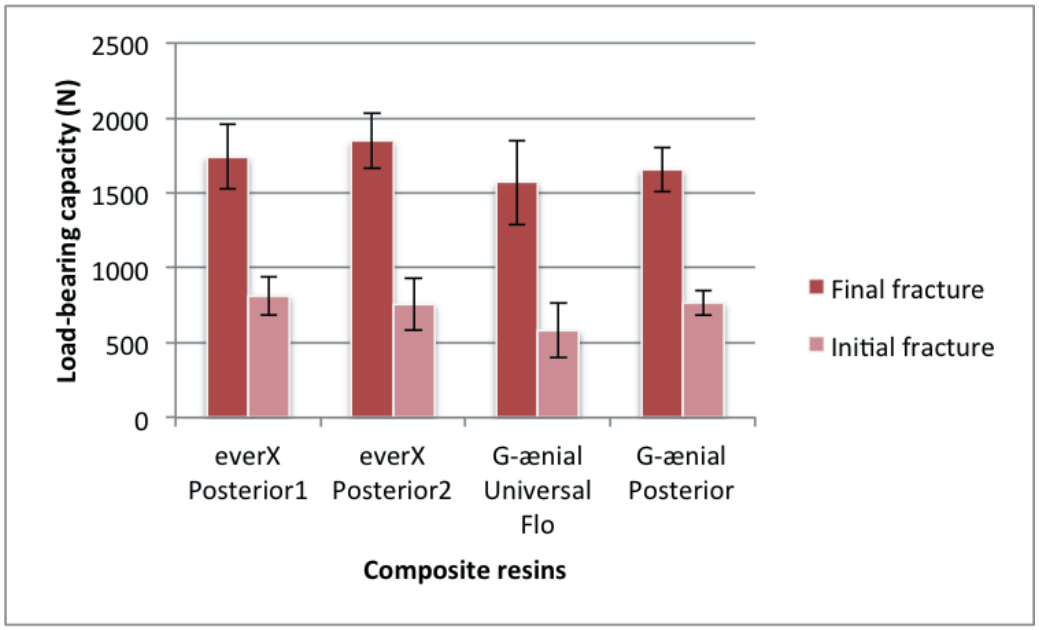

Figure 28. Shown are the load-bearing capacities (N) according to the filling composites for the specimens stored in water during 30 days and tested at 90˚. everX Posterior1:FRC framework with a transversal reinforcement in the pontic area. everX Posterior2: FRC framework without the transversal reinforcement.

Table 10 provides the *p* values for the initial and final fracture from a 3-way ANOVA for effects of filling material, storage, angle and their interactions.

Table 10. Shown are the *P* values from 3-way ANOVA for the initial and final fracture for effects of filling material, storage, angle and their interactions.

Variables	Initial Fracture	Final Fracture
Filling material	0.041	0.002
Angle	<0.001	<0.001
Storage	<0.001	0.262
Material-Angle	<0.001	0.004
Material-Storage	0.001	0.204

Adopted from original publication IV

The analyses of failure types showed that there was no debonding of any of the FDPs from the inlay preparations. No catastrophic fracturing of the pontics was observed. The failure types for each of the experimental groups are illustrated in Table 11.

Table 11. Failure types per experimental group.

Filling material	Angle used for testing	Storage	Failure types			
			I	II	III	IV
Flowable composite	90°	Dry	7	0	0	0
	90°	Water	1	0	6	0
	30°	Dry	0	3	4	0
Hybrid composite	90°	Dry	2	1	4	0
	90°	Water	2	1	4	0
	30°	Dry	4	3	0	0
everX Posterior[1]	90°	Dry	2	2	3	0
	90°	Water	0	2	5	0
	30°	Dry	1	6	0	0
everX Posterior[2]	90°	Dry	3	2	2	0
	90°	Water	0	3	4	0
	30°	Dry	0	3	4	0

[1]FRC framework with a transversal reinforcement in the pontic area. [2]FRC framework without the transversal reinforcement. I: Cohesive fracture in the shell-shaped acrylic resin denture tooth. II: Cohesive-adhesive fracture in the shell-shaped acrylic resin denture tooth. III: Cohesive fracture that includes the filling composite without fiber exposure. IV: Cohesive fracture that includes the filling composite with fiber exposure. Adopted from original publication IV

SEM examination showed the structural composition of the shell-shaped acrylic resin denture teeth (Figure 29). Two layers can be identified, the one that correspond to the occlusal surface clearly shows fillers, in contrast to the layer below, that is conventional PMMA.

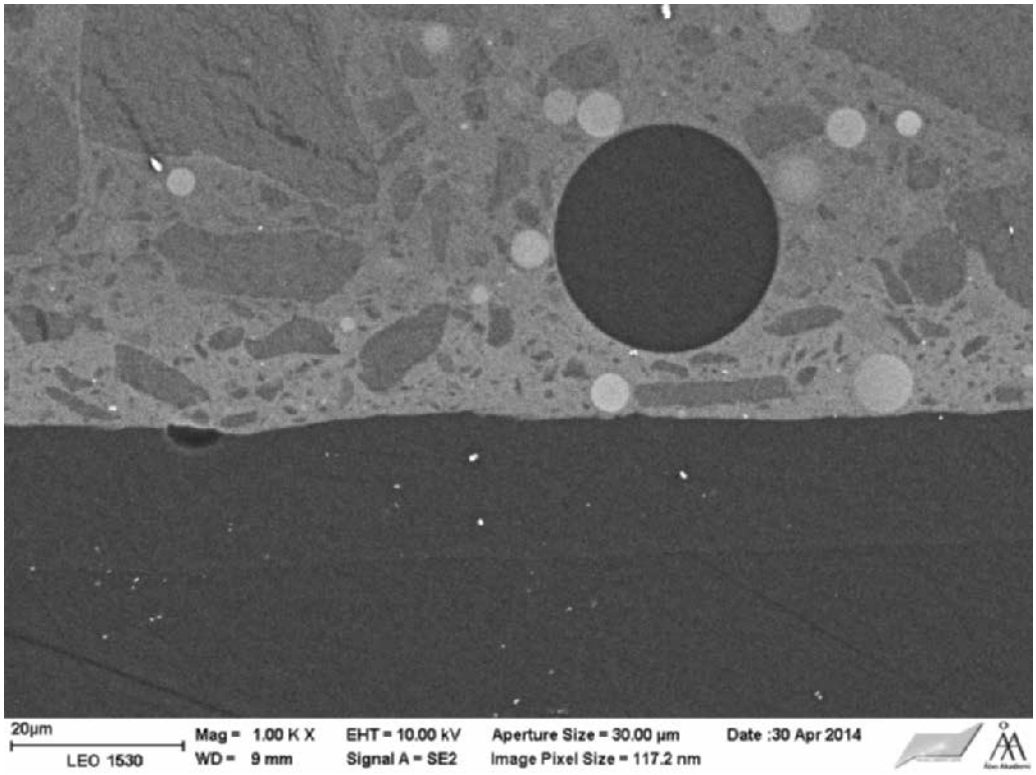

Figure 29. Structure of the shell-shaped acrylic resin denture teeth. The upper part corresponds to the occlusal surface (Urethane dimethacrylate with fillers). The lower part correspond to the bonding layer made of PMMA (x1000 magnification). Adopted from original publication IV.

SEM images are also provided from the orthogonally sectioned pontic which was loaded until the final failure. The image shows the crack propagation through the pontic (Figure 30). The fracture progressed from the main FRC framework towards the occlusal surface.

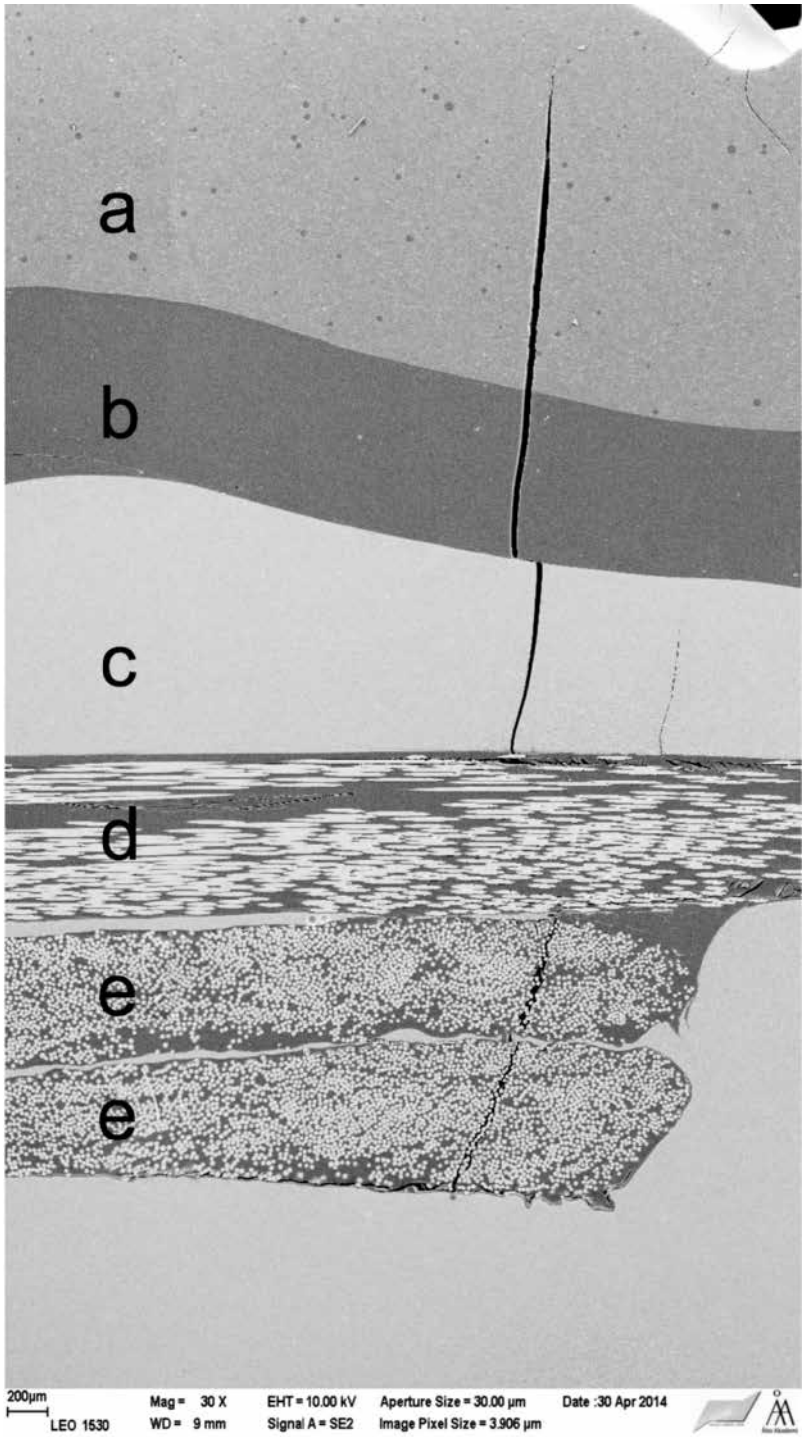

Figure 30. Cross-sectional view of crack propagation through the pontic. From the top to the bottom, the first two layers (a, b) correspond to the shell-shaped acrylic denture tooth, the third part (c) is the filling composite, below (d) is the transversal bundle of the FRC framework followed by (e) two bundles of the main FRC framework. At the bottom of the photo the filling composite is seen again (x30 magnification). Adopted from original publication IV.

6. DISCUSSION

This thesis is based on investigations, which aimed to enhance the mechanical and physical properties of inlay-retained fiber-reinforced composite fixed dental prostheses. This was achieved by improving the materials used as pontics, with the goal of increasing the longevity of these kinds of prostheses.

In vitro studies and clinical data demonstrate that FRC restorations are reliable alternatives to conventional prosthetic approaches, such as metal-based FDPs (Freilich *et al*., 2002; Kangasniemi *et al*., 2003). However, due to clinical shortcomings such as delamination of the veneering material and worn occlusal surfaces, improvements in these kinds of prostheses have become necessary. In an effort to achieve this, **Study I** was designed. **Study I** focused on evaluating the effects of different pontic materials and thicknesses on the abilities of inlay-retained FRC FDPs to withstand stress. Statistically significant differences were found between the materials and varying occlusal thicknesses. The highest interaction between thickness and load-bearing capacity in the pontics was found in those made of ceramic.

Unfavorable events such as chipping fracture in the veneering composite and delamination were found in the restorations evaluated in this study. This was in accordance with events described in the literature (Vallittu, 1997a; Freilich *et al*., 2002; van Heumen *et al*., 2010). At first, the cusp area fractured, followed by the whole construction. This indicates that the cohesive inner strength of the veneering material was lower than the adhesion of the pontic material to the FRC framework. In this study no failures were observed at the cementation joint. Previous studies have reported problems in the adhesion of the veneering material to the fiber framework (Behr *et al*., 2002). Natural teeth were used as abutments in some of the studies that have reported this event.

In this thesis, a zirconia model was used instead of natural teeth to cement the inlay-retained FRC FDPs. The differences in Young's moduli between zirconia and natural teeth may lead to overestimation of the fracture forces. The inclusion of natural teeth in this study might have represented a closer situation to the one presented in the oral cavity. This is then a limitation of this study.

Additional reasons for failure of FRC FDPs include faulty framework design. Low-structure frameworks are said to lead to higher failure modes. The positioning of the fibers is also a crucial parameter in framework design, optimal fiber positioning heightens framework strength and increases the mechanical properties of FRC FDPs (Dyer *et al*., 2005; Lassila & Vallittu, 2004).

The quality of direct FRC FDPs depends highly on the clinical skills of the operator to correctly replicate the shape and anatomical contour of the pontics. The provision of prefabricated pontics may represent a simplification of the technique, in addition to the achievement of pontics with standardized features (Belli & Ozer, 2000). In this first study, acrylic resin denture teeth and ceramic pontics were included and compared with pontics made of composite resin that has traditionally been used in FRC FDPs. The inclusion of ceramic pontics was intended to overcome the wear and discoloration that the conventionally used veneering materials of FRC FDPs have shown. In this case, CAD/CAM technology was included for designing and manufacturing the ceramic pontics. This was done to achieve a good stability of the pontics and ideally provide good adhesion to the framework.

Some reports have demonstrated the importance of reinforcing FRC FDPs on the gingival side of the pontics due to the high tensile stresses found in this zone (Narva *et al*., 2005). *In vitro* tests have reported the advantages of using long continuous fibers located in the tensile area of the FRC FDP, with strands perpendicular to the direction of the applied load (Dyer *et al*., 2004). From the different framework positioning assessed in this first study, the one with a positioning closer to the gingiva showed higher values for the initial and final fracture loads. Closer positioning of the FRC framework to the gingiva provides a bigger space above the framework, resulting in thicker pontics. A direct correlation was found between occlusal thickness of the pontics and their capacities to withstand the load. Thicker pontics showed more performant behavior. Thicker pontics also need to be combined with thick FRC frameworks to change the moduli of elasticity of the FRC. A stiffer FRC substructure attracts more loading onto itself, lowering the stresses in the veneering material, and optimizing the whole structure. In general, increasing the fiber volume will increase the load-bearing capacity, and therefore the clinical success rate of the FDP. By increasing the fiber thickness, the moment of inertia on the fiber cross-section increases, which lowers the stress concentration in the veneering material and reinforces the whole structure.

The average masticatory forces reported in the posterior zone are in the range of 500N to 900N (Behr *et al*., 2002; Waltimo & Könönen, 1993). In this first study, the mean value for the maximum fracture load was 1667N. Differences in design might make difficult to make comparisons with previous studies, but the fracture-strength values of the present study were higher than in other report (Behr *et al*., 2000). In that report values of 696 N and 722 N were obtained. In that case, the glass fibers used as the framework (Vectris; Ivoclar Vivadent) were different than those used in this study. The fiber system used in the first study of this thesis was based on impregnation of the reinforced fibers with polymer-monomer gel, different from the monomer resin impregnation of the Vectris system. The values obtained in this study might indicate that these kinds of FRC FDPs may be strong enough for clinical applications in terms of their load-bearing capacities.

However, dental restorations are not only subjected to static load, but also to cyclic load, the latter known as fatigue loading. Fatigue is a mode of failure where a material or structure is subjected to repeated loads, which leads eventually to failure. For that reason, dynamic load instead of static load may provide a better understanding of the clinical behavior of FRC FDPs.

Evaluations of the performance of acrylic resin denture teeth used as pontics in FRC FDPs were undertaken. Due to their shape and shade esthetics, good mechanical strength, and the ease at which their occlusal surfaces can be adjusted, these kinds of teeth are the first choice in many dental applications (Anusavice & Phillips, 2003). Furthermore, the incorporation of acrylic resin denture teeth as pontics in FRC FDPs may simplify the technique and perhaps increase their performance.

Chemical and mechanical retention systems have been suggested to improve the bond strength of acrylic resin denture teeth. Based on the composition of these teeth, the use of monomers to soften or dissolve their ridge-lap surfaces has been suggested (Cardash *et al*., 1986; Cunningham, 2000). Therefore, the **Study II** developed in this thesis aimed at evaluating the influence of monomer systems on the ridge-lap surfaces of acrylic resin denture teeth. This influence was assessed by determining their shear bond strength to a composite resin. Additional, variations on the surface microhardness of acrylic teeth due to the use of monomer systems were evaluated. The goal was to enhance the adhesive properties of acrylic resin denture teeth to increase their success when used as pontics in FRC FDPs.

Statistically significant differences were found in the bond strength and surface hardness between the monomers used on the acrylic resin denture teeth. The effectiveness of the chemical pretreatment of acrylic resin denture teeth to improve their bond strength was demonstrated, comparable with results reported in other studies (Cardash *et al*., 1986; Cunningham, 2000). This study found that the brand of acrylic resin denture teeth was an influential factor on the bond strength due to the differences in their polymeric structures. SEM examination of the acrylic teeth was performed. This showed a multiphasic polymeric structure, where likely PMMA beads were identified. These beads may have influenced the bonding to these kinds of teeth. The core of the beads, which retained their linear polymeric structure, may have provided a good bonding site between the acrylic resin denture teeth and the monomer used.

Acrylic resin denture teeth are composed of PMMA beads in a cross-linked polymer matrix. Between the beads and the matrix there is an intermediate layer called a semi-interpenetrating polymer network (IPN). The bonding mechanism of acrylic resin denture teeth to composite resin is based on the dissolution of the ridge-lap surface of the tooth by monomers, and on the formation of secondary IPN bonding. The polymer is dissolved

during this secondary IPN by the solvent molecules of the monomer, and the monomers penetrate the solvent-rich surfaces (Lastumäki *et al*., 2002; Lastumäki *et al*., 2003). The results obtained in this study using the monomer systems demonstrated their capabilities on dissolving the acrylic teeth ridge-lap surfaces. It was also demonstrated that the use of the monomers on the acrylic teeth surfaces provided active sites that reacted with the composite resin.

The dissolution gradient varies depending on how effectively the monomeric solvent swells and dissolves the PMMA, the contact wetting time and the polymeric structure of the substrate (Vallittu *et al*., 1994; Vallittu *et al*. 1997b). In this study, the time used for the pretreatment of the acrylic resin denture teeth surfaces with the monomer systems was an influencing factor on the bond strength. The intention was to investigate the dissolving capabilities of the different monomer systems used on the acrylic teeth according to their exposure times. Measurements of the acrylic teeth surface microhardness provided a better understanding of this phenomenon. It was found that the surface microhardness of the acrylic teeth decreased proportionally to the prolongation of the exposure time of the monomer systems. A deeper penetration of the monomers systems into the dissolved acrylic teeth surfaces could be a possible explanation for the changes in the surface hardness. This finding needed to be further investigated.

Consequently, **Study III** was designed to investigate the penetration depth of the same monomer systems that were previously tested in the **Study II**. This was done to find explanations to the surface microhardness changes found on the acrylic resin denture teeth previously tested. Statistically significant differences were found in the penetration depths of the monomer systems used according to the exposure time applied and the different teeth brands tested. Thus, the dissolving and penetration capabilities of the monomer systems used into the surfaces of acrylic resin denture teeth tested were demonstrated.

Previous reports have shown the use of confocal scanning microscopy for measuring the penetration depth of monomer systems into FRC-based polymers (Mannocci *et al*. 2005; Wolff *et al*., 2012). In this third study, a confocal scanning type microscope was used for evaluating the penetration depths of the monomer systems into the acrylic teeth surfaces. Three different acrylic resin denture teeth brands were included. Potential differences in their structural composition were considered, which may have an effect on the performance of the monomer systems. Rhodamine B was used for tracing the diffusion of the monomers into the surfaces. Some reports provide evidence that the confocal scanning microscopy may misrepresent the real penetration of the monomers due to the dispersed fluorescent light (Pioch et al., 1997). Scattered fluorescent light might simulate broader infiltration zones than are actually present under reflected light imaging. For that reason, reflected light imaging is needed in addition to the fluorescent

light to provide a more accurate description of the diffusion of the monomer systems. Taking that into account, in this third study, the penetration depths were analyzed and measured under the fluorescence and reflection modes.

It has been reported that the diffusion of monomers into a polymeric structure can be possible. This is developed for instance when the substrate consists of a linear polymer, such as PMMA. Also the monomers need to have the potential of dissolving the linear polymer phases of the substrate (Lastumäki *et al*., 2002; Vallittu *et al*., 1997b). The time used for the exposure of the monomers on the surface of acrylic resin denture teeth has an influence on the dissolving potential of the PMMA (Vallittu *et al*., 1994).

The capabilities of methylmethacrylate (MMA) to enhance the bond strength in denture tooth repairs have been reported. Three minutes have been recommended to expose the denture tooth to this monomer to form a secondary IPN layer (Vallittu *et al*., 1994). MMA was used in this third study; however, its performance provided the lowest penetration depth values, excepting for one of the brands used (Vitapan teeth), where MMA achieved a mean penetration depth of 3.95 μm when used for 60 minutes. The difficulties in maintaining the MMA on the surface of the acrylic teeth during the pretreatment time due to its volatility, may have played a role on the low penetration of this monomer into the surfaces.

In this third study, a flowable composite exhibited the highest penetration depth (mean value 5.08 μm) when used during 60 minutes. A potential explanation of this might be found in the increase in temperature generated by the light-curing unit. The heat generated during the 5 minutes used for the photo-polymerization of the monomer systems might have allowed the activators, initiators and monomers of the flowable composite to propagate into the acrylic teeth surfaces. This assumption is based on the penetration found among some monomers in denture base polymers achieved when exposed to high temperatures (Vallittu, 1995c). However, these temperatures were not measured in this thesis and very likely they were lower than those reported by Vallittu (Vallittu, 1995c).

An understanding of the influence of fiber framework positioning was obtained from **Study I**. **Studies II** and **III** provided some knowledge on the performance of acrylic resin denture teeth. **Study IV** was then designed to assess the load-bearing capacities and fracture behavior of FRC FDPs. In this case, pre-shaped acrylic resin denture teeth were used as pontics, using different composite resins as filling materials.

In oral conditions, FDPs are subjected to variations in magnitude and direction of forces by chewing. The forces occurring during physiological function have a vertical as well as a horizontal component. Inlay-retained FRC FDPs were evaluated in **Study IV** using two

different angulations, 90˚ and 30˚. The inclusion of these two angulations had the goal of replicating the multiple force vectors found in the oral cavity. Dry and those specimens stored in water during 30 days were tested as an approximation of clinical situations. These 30 days were chosen based on reports that revealed that water sorption occurred primarily during the first 20 to 25 days. After that time, the material immersion in water remained constant to 90 days (Cal *et al*., 2000). The positioning of the framework was determined in the closest location to the gingiva, reserving a space of 2 mm underneath for the veneering material. Additionally, the internal surfaces of the acrylic denture teeth used for the fabrication of the pontics were pretreated with a monomer system. This was done based on the findings of **Studies II** and **III**.

Statistically significant differences were found on the load-bearing capacities of the tested FDPs between the different composite resins used as filling materials. The highest load-bearing capacities were found in the FRC FDPs made using a short fiber-reinforced composite resin as a filling material tested at 90˚. The increased filler content, in addition to the short fibers immersed in this composite resin might be an explanation for the improvement seen in the fracture resistance of the tested FDPs. Between the specimens tested at 30˚, the most durable FRC FDPs were found in the group where a flowable composite was used as filling material. The dissolving capabilities of flowable composite on acrylic surfaces may be an explanation for this phenomenon. This assumption is based on the results obtained in **Studies II** and **III**, where it was assessed. These dissolving capabilities may have provided an adhesive interphase strong enough to withstand the non-vertical forces applied at an angulation of 30˚. On the contrary, the flowability of this composite makes it a non-ideal material to withstand the vertical load in testing performed at 90˚.

The clinical performance of FRC FDPs has been previously evaluated, providing some reports on their suitability (Freilich *et al*., 2002; van Heumen *et al*., 2010; van Heumen *et al*., 2009; Vallittu, 2004). In this fourth study, a mean fracture strength value of 1700 N demonstrated the high fracture resistance of these prostheses and their potential to be used in clinical applications. Fracture of the veneering composite has been reported as the main shortcoming of FRC FDPs (van Heumen *et al*., 2010). This is consistent with the findings in this fourth study where delamination of the veneering material was observed. However, this event was manifested during a stress application that exceeded a mean value of 1500 N. This value was the lowest obtained from the final fracture in the FRC FDPs tested dry at 90˚. It has been recommended to include additional bundles of fibers at the pontic area to act as an additional reinforcement that potentially could counteract the fracture seen on the veneering material (van Heumen *et al*., 2010; Freilich *et al*., 2002). This fourth study included additional fiber reinforcement in the pontic area, which could be a possible explanation of the high fracture strength values obtained.

Short discontinuous and long continuous fiber-reinforced composites have different mechanical properties. When continuous unidirectional fibers are changed to longitudinally oriented discontinuous short fibers, a detrimental effect on the ultimate tensile strength of the composite is seen. This is manifested as a reduction of the tensile strength. When short fibers are randomly oriented, the tensile strength reduces even more, and the FRC changes from anisotropic to isotropic (Vallittu 2015).

In **Study IV** of this thesis the FRC frameworks were made including two bundles of FRC between the inlays of the abutments. An additional FRC bundle was placed transversally in the pontic area. A variation of the conventional FRC framework was made, where the additional transversal fiber was not included. This variation of the framework was used in one experimental group. A short fiber-reinforced composite was used as a filling material in this experimental group. This filling material was used to complete the contour of the shell-shaped acrylic teeth used as pontics. The goal was to evaluate the influence of the short fibers of this composite resin in the final behavior of the FRC FDPs. The intention was also to evaluate the fracture resistance of the prostheses that did not have a transversal bundle of fibers. Similar mean values were obtained with and without the transversal fiber reinforcement in the groups made with this short fiber-reinforced composite resin. The conclusive results in the load-bearing capacities of this experimental group might be due to adequate bonding properties and improved toughness of the polymer matrix of semi-IPN. This refers to the matrix of short fiber-reinforced composite used in this experimental group.

Restorative composites with microfibers suffer extensive wear and fracture. This happens due to the use of fibers with a length below the critical fiber length. The critical fiber length of E-glass with Bis-GMA polymer matrix varies between 0.5 and 1.6 mm (Cheng *et al*., 1993). In order for fibers to effectively reinforce polymers, stress transfer from the polymer matrix to the fibers is crucial (Garoushi *et al*., 2007; Garoushi *et al*., 2012). This is obtained by having a fiber length equal or greater than the critical fiber length (Garoushi *et al*., 2007; Garoushi *et al*., 2012). The short fiber-reinforced composite used in this study had fiber length between 1 and 2 mm, exceeding the critical fiber length. That might explained how the short fiber inclusion with semi-IPN resin matrix provided improved mechanical properties in this study.

The reinforcing effect of the fiber fillers is based on stress transfer from the polymer matrix to the fibers, and also the behavior of individual fiber as a crack stopper. A study (Garoushi *et al*., 2007) showed how short fiber fillers could increase the fracture resistance of a restoration by stopping the crack propagation. However, another report shows that the use of fibers with a low aspect ratio can weaken the composite as fiber ends create discontinuity in the matrix and act as stress concentrators or flaws (Shouha *et al*., 2014). After the static fracture test was performed in the current study, the

fractured pontics were cross-sectioned and analyzed using SEM. It was demonstrated that the fracture initiated at the lower surface of the main FRC framework, continuing the crack propagation towards the occlusal surface until it reached the acrylic resin denture teeth. Delamination at the interface acrylic teeth-composite resin exhibited insufficient adhesion between these two materials.

Water storage and mechanical loading are used as aging methods of polymerized FRC. These have a detrimental effect on the mechanical properties by reducing strength by almost 30% (Bouillaguet *et al*., 2006). In this study, specimens stored in water during one month were evaluated. No statistically significant differences compared with the dry specimens were found. However, the delamination seen at the acrylic teeth-composite resin interface shows that adhesive failures may exist in stress conditions.

Other factors that influence the mechanical properties of FRCs are volume, orientation and location of the fibers, and the chemical bonds between the components (Tezvergil *et al*., 2006). It has been reported that loss of interfacial bonds between fibers and matrix is the main cause of reduction in mechanical properties (Bouillaguet *et al*., 2006). In clinical conditions FRCs are subject to water sorption. Water sorption is a mechanism of water penetrating into the resin matrix itself (Behr *et al*., 2002; Lassila *et al*., 2002), and between matrix and fibers. This event is developed due to the existence of voids along the fibers, caused by incomplete fiber-matrix impregnation (Lassila *et al*., 2002; Lastumäki *et al*., 2003). Water sorption creates small increases in volume and induces plasticization of the resin matrix. It also deteriorates the fiber-matrix interphase by hydrolytic degradation of the polysiloxane network between fibers and matrix (Lassila *et al*., 2002). The aforementioned events weaken the structure, leading to a decrease in dynamic failure load. Semi-IPN matrix-based FRCs, which were used in this study, are prone to water sorption possibly explained by the hydrophilic properties of the resin matrix (Lassila *et al*., 2002). The fact that no statistically significant differences were found between the dry and water stored specimens on this study may indicate that improved adhesion was achieved between the veneering composite and the FRC framework, which could have reduced water sorption. However, this is an assumption and not a proof of improved adhesion.

Reports of many *in vitro* studies, as well as the results described on this thesis suggest the suitability of FRC FDPs to be used in clinical applications. Long-term clinical studies are needed to confirm these findings, which may provide a better understanding of how advisable the use of these kinds of prostheses as a permanent restorative solution would be. This thesis showed improved mechanical and physical properties of FRC FDPs. Improvements that might provide long lasting prosthetic solutions and may contribute to the increased use of FRCs in clinical dentistry.

7. CONCLUSIONS

Based on the studies included in this thesis, the following can be concluded:

1. The thickness of pontics of FRC FDPs, directly related to the vertical positioning of the FRC framework, influenced the load-bearing capacities of prostheses of these kinds. Thicker pontics provided the most durable FDPs, being the ceramic pontics the ones that showed the best performance when evaluated at the maximum thickness.

2. The debonding stress, so-called shear bond strength of acrylic resin denture teeth to a composite resin was influenced by the chemical pre-treatment of their ridge-lap surfaces. Decreasing values of the surface hardness of acrylic teeth according to the monomers' exposure time showed the dissolving possibilities of the bonding surface of acrylic teeth.

3. The penetration depth of monomers systems into acrylic resin denture teeth is influenced by the type of monomer used. This should be considered when designing an FRC FDP with a polymer pontic due to the possibilities of establishing more durable FDPs with a FRC framework.

4. The composite filling material used in combination with pre-shaped acrylic resin denture teeth as pontics of FRC FDPs has an influence on their load-bearing capacities. These kinds of acrylic teeth are able to withstand the masticatory forces that are normally seen in clinical applications. FRC FDPs whit a short-fiber composite resin used as filling material showed the highest load-bearing capacities when tested at 90˚ to the direction of the occlusal surface.

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