Esthetic and bonding enhancements of tooth colored indirect restorations
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CHAPTER 1

General Introduction
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During the last few decades, the approach to restorative dentistry has dramatically changed from the drill and fill concept to replace the defective tooth tissues, to a treatment with minimal sacrificing the sound tooth tissues. Nowadays, restorative dentistry is moving towards more extensive caries diagnosis and assessment, non-invasive caries management, minimal invasive restorations, and adhesive tooth colored restorations.

The requirements for a satisfactory restorative material are no longer high strength and low wear only, but a complex set of interrelated properties dominated by biocompatibility and esthetics.[1] Therefore, the research and development invested to address this aspect have brought numerous adhesive tooth-colored restorative materials onto the market as alternatives to the widely used high strength metallic restorations.

1.1 Esthetic alternatives to metallic restorative materials

Due to their high mechanical properties, metallic restorations such as gold alloys and amalgam have provided satisfactory results for many decades with respect to the preservation of tooth anatomy. However, there is an increasing concern about the possible hazardous effect of mercury to the patient and to the environment when using amalgam as restoratives. Besides, the increased esthetic needs of the patients, have urged the dental professional to find a material, which has a tooth like color. Currently, hybrid resin composites are the material of choice for restoring small and medium sized cavities.[2] However, setting shrinkage stress development in direct resin composite restorations is still one of the major drawbacks of this material.[3-5] Excessive shrinkage stresses being placed on the tooth cusps due to wall-to-wall contraction may lead to cuspal distortion, marginal discrepancies, postoperative hypersensitivity and microleakage.[6-8]

Factors that influence the magnitude of polymerization stresses, are the E-modulus of the material and the cavity geometry, which determines the height of the so-called configuration-factor (C-factor), which is the ratio of the bonded surface to the free non-bonded surface of the restoration.[4] Therefore, in case of restoring large defective tooth tissues with an unfavorable C-factor, the use of direct resin composite restoratives could place the marginal integrity of the restoration at risk. In addition, re-establishing the anatomical form of the tooth for such large defects can be a demanding task for a direct approach in particular in difficult accessible areas.[9] For such unfavorable conditions, the indirectly fabricated tooth-colored restorations made of composites or all-ceramics appear feasible alternatives.[10] The indirect restorative
approach offers better surety to construct an appropriate tooth form and anatomy. Moreover, the shrinkage stresses generated during setting of the material can be limited only to the thin resincement layer, which is used to bond the restoration.[11,12]

1.2 Tooth-colored indirect restorations

Indirect tooth-colored restorations (inlays and onlays) represent an interesting option to esthetic restorative treatment of the posterior dentition and can be made from:[13]

- preprocessed composite (direct, semidirect, indirect, or milled), or
- feldspathic ceramic (sintered or milled) or glass ceramic (cast, heat-pressed or milled).

The choice between processed resin composites and all-ceramic indirect restorations has become increasingly complex. Laboratory-processed composites have reached a high technological level, providing high level of esthetics and physical properties. Despite these improvements, it has been reported that indirect ceramic restorations tend to leak less and adapt better with superior clinical success than composite inlays and onlays.[14,15] On the other hand, another study has demonstrated deterioration of marginal quality of ceramic inlays under load, particularly at the gingivo-proximal enamel margin.[16] This was attributed to the inability of the stiff ceramic restoration to follow the tooth deformation caused by mechanical loading. Due to the large number of variations in techniques and systems, which are available for fabrication of composite or all-ceramic indirect restorations, no consensus is reached about which material performs better clinically. Although the esthetic results and survival rates of both systems are quite successful,[17] each system has its own advantages and disadvantages.[16,18]

The CEREC CAD/CAM system (Sirona, A.G., Bensheim, Germany) developed by Mormann [19] allows the dentist to take an optical impression of the tooth preparation and with the aid of a computer to design the restoration. Once the design is completed the information is processed by the computer for direct milling of prefabricated blanks of ceramic material into the final restoration (Figure 1.1a).
The advantage of the ceramic machining systems is that it allows the use of materials with improved mechanical properties that cannot be obtained by the conventional restorative procedures. Accurately controlled industrial ceramic processing can produce materials with increased micro structural uniformity, higher density, lower porosity, and decreased residual stresses. Fabrication of the restoration from materials without internal defects will result in stronger restorations. However, ceramic blocks, which are used by ceramic machining systems, are monochrome and the milled restoration lacks the presence of any surface characterization. This may limit the esthetic result of the final restoration.

Also preprocessed composite blocks have been introduced for fabrication composite inlays and onlays by the CEREC system (Paradigm MZ100, 3M ESPE, St. Paul, MN, USA). The manufacturer claims as advantages over restorations milled from ceramic blocks, easier finishing and polishing, kindness to the natural dentition with regard to wear and easier to make add-on adjustment.

### 1.3 Adhesion: The key to success of the tooth-colored restorations

To fulfill the requirements of a successful dental restoration, the restorative material used should be able to restore simultaneously the biological, mechanical and esthetical functions of the defective tooth tissues. These requirements could be achieved if the material is biocompatible, environmental friendly and able to biointegrate with the hard tooth tissues. A strong and durable bond due to material bio-integration will seal the tooth-restoration interface, preventing microleakage and subsequent ingress of microorganisms. Microleakage and ingress of microorganisms are considered to be the main etiological factors for pulpal damage.[22] The
formation of continuity between the tooth and restoration by a strong bond will increase the strength of both the restoration and tooth structure through uniform transformation and distribution of the functional stresses. Thus a brittle but high esthetic material such as ceramic can be used as restoration without metal substructure. In addition, a bonded restoration would offer more conservation of the tooth tissues, as excessive removal of sound tooth structure to mechanically retain the restoration is no longer required.

Figure 1.2 Schematic illustrations showing the interfaces of an adhesively luted indirect restoration.

Statement of the problem

Despite the great success that has been achieved regarding bonding of the direct restoration to the tooth tissues, bonding of the indirect restorations is still a challenging matter. This is because the indirect restorative procedure leads to an increase of interfaces for bonding. One interface is located at the tooth structure and the other at the fitting surface of the restoration (Figure 1.2). Therefore, in order to establish a strong and durable bond, which is necessary for the biomechanical aspect of the tooth-restoration system, appropriate knowledge about the respective surface treatments and the cementing materials is crucial.

1.4 Dental luting cements

With the introduction of adhesive cements the function of dental cements has been changed from simply filling the gap between the restoration and tooth structure to actively bonding the two substrates together.[23] Early extracoronal or intracoronal restorations completely relied on macromechanical retention of which the effectiveness mainly depended on the geometry of the preparation. Supplementary means of retention were also used by preparing grooves, skirts, slots,
pins or dowels, but these required unnecessary removal of sound tooth structure, which increased
the chance for pulpal irritation. It has been shown that the thickness of the remaining dentin is
inversely proportional to the pulpal response to an irritant.[24] Moreover, dentin crazing or root
fracture could be developed from using retentive means such as pins and radicular posts.[25,26]

<table>
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<th>Glass/Ceramic</th>
<th>Zn O</th>
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<td>Polyalkenoic acid</td>
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<td>Eugenol</td>
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<td>Resin and polyalkenoic acid</td>
<td>Resin-modified glass-ionomer</td>
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<td>MMA monomer b</td>
<td>Unfilled Resin</td>
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a Polymethyl-methacrylate polymer
b Methyl-methacrylate monomer

Originally, the term cement was used in dentistry for luting materials that are composed of a
powder and a liquid to be mixed. Later when other delivery systems became popular, to-paste
materials with equal compositions became available too. Nevertheless, dental cements can be
characterized by the powders and liquids of which they are composed of. The powder is used as
the reinforcing component and consists mainly of metal oxides (glass, ceramics, zinc oxide), or
prepolymerized resin (polymethyl-methacrylate). The liquid forms the matrix during the setting
reaction that embeds and bonds the filler particles together (Table 1.1). Generally, two types of
setting reactions can be distinguished. First, a polymerization reaction of the resin matrix,
initiated either by mixing a catalyst with an initiator or by exposing a photo-initiator to light. The
second is an acid-base reaction that takes place between the basic glass and the acidic liquid,
where polyalkenoic acid chains are cross-linked to form the matrix. Most of the dental luting
cements can be characterized this way as a composite consisting of a matrix filled with
reinforcing filler. The filler particle size and distribution influence the viscosity of the unset
cement as well as the cement film thickness.

Dental cements can be also classified according to their ability to bond to the tooth structure,
into conventional and adhesive cements. The conventional or non-adhesive cements are those
that do not have the ability to bond to tooth structure, while the adhesive luting cements do bond
to the tooth structure. The latter can be divided into two main groups; cements that have the intrinsic property to create chemical bonding to the tooth tissues through an ionic exchange process, and cements that bonds by micro-mechanical interlocking with the conditioned tooth structure. The first group consists mainly of glass ionomer cements and resin modified glass ionomer cements, while the second group forms the resin-based cements, which include unfilled adhesive resin cements and resin composite cements (Figure 1.3). The resin composite cements are generally composed of dimethacrylate-based monomers, such as Bis-GMA and/or Urethane dimethacrylate, and inorganic filler particles.[2] Their composition is basically that of resin composite restorative materials, but with a lower filler loading and/or higher percentage of diluting monomer such as triethylene glycol dimethacrylate (TEGDMA). Actually, the adhesive properties of the latter group usually are determined by the type of the combined adhesive system and not primarily by the choice of the cement per se.
Figure 1.3 Classification of dental luting cements based on their adhesive potentiality.

* Self-etching primer
1.4.1 Mechanical properties of the cements

An ideal luting cement should have sufficient mechanical strength to resist functional forces and degradation in the oral environment over the lifetime of the restoration.

Cement solubility and wear

In the clinical situation, solubility, water sorption, and frictional forces can cause degradation of the cement, leading to failure of the restoration. Conventional cements, such as zinc phosphate, polycarboxylate, and glass-ionomer cements are more soluble than resin based luting cements.[27] For that reason a thin cement film thickness at the margin was required for the conventional cements. In spite of the fact that resin based cements lose some of their contents when subjected to water, they are hardly soluble.[28] Milleding et al. have used the microhardness test to indicate the ability of resin cements to resist degradation in an aqueous environment.[29] They found that cements perform better with a higher filler content and higher viscosity.

Resin cement wear was shown to be an occlusal problem caused by direct contact with the opposing tooth (2-body wear) and by the food bolus (3-body wear). However, this problem is thought to be self-limiting due to the sheltering effect of the inlay and enamel.[30] Ideally, a cement should exhibit a wear behavior similar to enamel, but several studies have shown that the wear resistance of resin cements is still much lower than that of enamel and inlays.[16,31,32]

Resin cement filler content, filler type, size, and viscosity are among the factors that influence cement wear. Due to the smoother surface of microfilled luting composites these cements are more wear resistant than hybrid luting composites.[33-35] The choice of the type of the cement and its filler loading becomes more important when the marginal gap distance between the restoration and the tooth structures increases.[36] Torii et al. found that cements with a higher filler content had an increased wear resistance and were remarkably better in wider gaps.[37] Therefore, with regard to resistance to degradation and wear, resin cements with higher filler content and higher viscosity are preferable.

Since high-viscous cements interfere with inlay cementation, the ultrasonic insertion technique was introduced, which takes the advantage of the thixotropic properties of a composite.[38] When subjected to ultrasonic vibrations, a high-viscous composite is better able to flow.[13]
**Water sorption**

Water sorption of resin cement may compensate for its initial polymerization shrinkage [39], but an adverse effect could follow by over-compensation, which can result in expansion stresses.[40] It has been shown that water sorption is more pronounced and excessive for resin-modified glass-ionomers and unfilled PMMA based cements than for glass-ionomer or resin cements.[28] Therefore, an all-ceramic restoration cemented with these cements may be exposed to expansion stresses, which increases the risk for crack initiation and failure of the restoration.[41,42]

**Stability and Stiffness**

All cements in dentistry undergo dimensional changes during and after setting. Shrinkage occurs during setting [43], while exposure of the set cement to the oral environment may results in water sorption and swelling. Also temperature changes in the oral environment contribute to dimensional instability [44] being different for cement, tooth, and restoration, because of differences in their thermal expansion coefficients. All the dimensional changes mentioned can cause stresses in the cement layer. Setting shrinkage stresses and thermally induced stresses may place the adhesion at risk, while the hygroscopic expansion may affect the integrity of all-ceramic restorations. Interestingly, finite element analysis showed that setting shrinkage stresses in the cement layer of a flexible porcelain veneer restoration can protect this restoration from (opposite) destructive stresses induced during function.[45] In other cases, if little or no elastic yielding of the surrounding materials (tooth and restoration) is allowed, shrinkage stresses can be detrimental for the bonded interfaces or even the cohesive integrity of the cement layer.[46] Theoretically for a complete rigid surrounding the shrinkage stress is related to the C-factor (ratio of bonded surfaces to free non-bonded surfaces), which takes a very high value for cement layers. Because flow is not possible along the surfaces, all shrinkage will be directed uniaxially from wall-to-wall approaching the volumetric shrinkage, which results in a stress that is three times higher than from pure linear shrinkage.[47] Thin cement layers however, only need a small amount of elastic yielding from their surrounding to lower the stress, just enough for the adhesion or cohesive strength to survive.

Elastic moduli intermediate between those of the restoration and tooth structure are desirable because this can reduce interfacial stress concentrations without causing excessive strain.[48] A cement with a low modulus of elasticity such as the unfilled PMMA based adhesive resin cements may offer resiliency and flexibility to the bond with higher resistance to occlusal impact stresses. However, this is more relevant for splinting loose teeth, or cementing metal based adhesive bridges. Due to the low critical strain values of ceramic materials a stiffer supporting
cement is needed to minimize elastic deformation during occlusal loading as to avoid catastrophic failure.[49]

**Curing strategies**

Resin based cements can be divided by their polymerization method into auto-cure, light-cure, and dual-cure cements. For facial veneers, light-cure resin cements can be used, as they transmit sufficient light for polymerizing the cement. When access of the curing light is limited for example for large ceramic or resin composite indirect restorations, dual-cure resin-based cements are recommended to compensate for the light attenuation caused by the thickness and shade of the restoration.[50] However, self-curing alone is insufficient for dual-cure cements to achieve maximum hardening.[51-53] In the absence of light poor mechanical properties of the cement result and consequently inadequate bonding to the restoration.[54] Therefore, it is recommended to light-cure the restoration from several directions to do as much as possible to initiate the dual-cure cement.[55] The use of light curing devices that generate light with a high intensity could be of further help in this respect.

**1.4.2 Biological properties**

*Biocompatibility* is formally defined as the ability of a material to elicit an appropriate biological response to a given application in the body.[56] The biocompatibility of a resin based luting agent is related, among others, to its degree of conversion. Complaints of sensitivity may arise from the incomplete polymerization of the resin cement.[57] The degree of conversion of polymer based dental materials is influenced by many factors, including the addition of polymerization promoters and inhibitors [58], the chemical structure of the monomer [59], the filler composition of the material [60], the shade of the material, and the chemical or light energy impaired to activate the reaction.[61,62] It has been found that polymerization of light-activated composite luting agents can not be accomplished predictably through a preprocessed resin restoration exceeding 2 mm in thickness.[63]

An important biological aspect of luting cements is its ability to seal the tooth-restoration interface. At present, the biological seal of cut tooth tissues can be achieved either with auto-adhesion mechanisms by ionic bond formation using glass-ionomer based cements or with micromechanical interlocking of adhesive resin based cements with the tooth tissues by forming an impermeable resin infiltrated hybrid layer.[64] Adhesively retained restorations yield profit in terms of the conservation of sound tooth structure especially with teeth of compromised retention.
due to short, over convergent, or insufficient remaining tooth tissues to retain a restoration. Additionally, by relying on the bonding capability of the adhesive cements it is more feasible to end the preparation line above the gingival margin, which ensures minimal periodontal response.

It is important for the cement-tooth interface to form a strong and durable adhesive bond, not only to resist the shrinkage stresses during setting, but also for the long-term to resist bacterial acidic attack and other detrimental factors from the oral environment. However, an absolute leak proof and durable strong resin-dentin bond, so far could not be reached.[65-69] Therefore, luting cements, which are able to release fluoride, may have an advantageous effect on caries inhibition. For this to realize, a strong initial fluoride release ‘burst effect’, and a less strong but stable and constant release by the material are required.[70] It has been reported that conventional glass-ionomer cements and resin-modified glass-ionomer cements showed an initial higher fluoride burst effect and higher fluoride uptake in comparison to polyacid-modified and fluoride containing composites.[71-74] However, the low mechanical strength and the moisture sensitivity of the conventional glass-ionomer cements and the high water sorption of the resin-modified glass-ionomer cements limit their use as adhesive cements, particularly for retaining all-ceramic restorations.

![Antibacterial monomer MDPB](image)

**Figure 1.4** Antibacterial monomer MDPB

Recently a two-step self-etching adhesive system with antibacterial properties has been introduced (Clearfil Protect Bond, Kuraray, Osaka, Japan). A new monomer, 12-methacryloxydodecylpyridinium bromide (MDPB), was developed by combining the antibacterial agent quaternary ammonium and a methacryloyl group (Figure 1.4), incorporated into the resin of the self-etching primer.[75,76] The bactericide-immobilized agent does not leach out from the material, but acts as a contact inhibitor against bacteria. *In vitro* studies have reported that the incorporation of MDPB is effective in providing dentin-bonding systems with antibacterial activity before and after curing the resin.[77,78] It is assumed that application of MDPB-containing adhesives can inactivate residual bacteria in the cavity in the unpolymerized stage[79] and have an inhibitory effect on invading bacteria, if microleakage would occur.
However, this anti-microbial effect and its clinical relevance still need to be confirmed in clinical trials.

1.4.3 Adhesive characteristic of the cements

**Bonding to ceramics**

Metal and porcelain-fused-to-metal restorations have for along time been successfully luted with non-adhesive cements. However, the brittle nature of a restoration made of all-ceramic material requires the use of strong stable substructure to support the restoration against the destructive tensile stresses. Ceramic material start to fail when a flow or a crack in the material propagate under an applied tensile stresses.

There are several factors which can be associated with crack initiation and propagation in dental ceramic restoration, including:[80]

1) shape of the restoration.
2) microstructural inhomogeneities.
3) size and distribution of surface flaws.
4) residual stresses and stress gradients, induced by grinding, polishing, or thermal processing.
5) the environment in contact with the restoration.
6) ceramic-cement interfacial features.
7) thickness and thickness variation of the restoration.
8) elastic moduli of restoration components.
9) magnitude and orientation of applied loads.

Ceramic support can be reached either by a strong adhesion of the ceramic to metal substructure or adhesive bonding the ceramic with the underlying tooth structure. Various investigations have shown that all-ceramic restorations cemented with adhesive resin based cements have higher fracture resistance when compared with non-adhesive cemented restorations.[81-83] It is believed that the bonding capability of the adhesive resin cement is able to repair the surface flaws located at the fitting surface of the all-ceramic restoration, hence it might reduce the potential for crack propagation. Moreover, the higher fracture toughness and improved mechanical properties of resin based cement in comparison to conventional cements.
provide an effective load transfer of the functional stresses through the brittle ceramic restoration to the underlying tooth structure.[84]

Bonding of adhesive resin cement to ceramics can be achieved through micro-mechanical and/or chemical bonding mechanisms. There are several methods to condition ceramic surfaces to enhance bonding to resin luting cements, though, the effects of different surface treatments on bonding are strongly dependent on the type and the microstructure of the ceramic surface to bond to.[47,85,86]

Surface pretreatment for mechanical bonding

Mechanical bonding to ceramic surfaces can be obtained by preparing the surface by grinding, abrasion with a diamond rotary instrument, sandblasting with alumina, and conditioning using different types of acids. Hydrofluoric acid (HF) is commonly used to etch porcelain for indirect restorations.[87,88] As alternatives, to avoid the hazardous HF, acidulated phosphate fluoride [85] or phosphoric acid (H₃PO₄) can be used. However, their effectiveness for the enhancement of the bond strength is still doubtful.[89] Phosphoric acid is used in industry to etch glass at high temperature. At room temperature the action of H₃PO₄ is limited to clean the ceramic surface without producing an apparent etching pattern. Therefore, this treatment may not contribute to strong resin-ceramic bond by micromechanical retention. However, it was reported that H₃PO₄ would improve the resin-ceramic bond by chemical alteration of the ceramic surface.[90] The acidity of the H₃PO₄ may increase the concentration of H⁺ ions on the ceramic surface resulting in chemical activation of the subsequently applied silane primer.

The capability of HF to alter the ceramic surface depends on the ceramic microstructure and composition. Ceramic that contain a glass-phase (leucite, silica-based feldspatic or glass ceramics) can be etched with HF while all-ceramic restorations made of alumina cores cannot be etched sufficiently. HF creates a surface pattern for micromechanical attachment by preferential dissolution of the glass phase from the ceramic matrix which increases the surface area and enhances the micro-mechanical retention of the resin cement.[91] The micro-undercuts formed on the ceramic surface by HF etching allow the penetration of both the resin and fillers components of the luting composite cement to form particle-reinforced resin tags that contribute for strong resin-ceramic bond.[92] On the other hand, over-etching with high HF concentrations or extended etching times may lead to a reduced bond strength.[93] HF can be so aggressive to the surface of some ceramic materials that can affect its mechanical properties, which would in turn reduce the resin-ceramic bond strength.[86,94] Consequently, one should take into account the type of ceramic being used before HF etching.
Sand blasting with Alumina particles is used to remove refractory investment material during the laboratory procedures of the fired ceramic restorations when the hot-press technology is used, for these cases the ceramic surface is always gently roughened. It was found that the bond strength of porcelain laminate veneer was greater when etched than when lightly sandblasted.[88] Conversely, excessive sandblasting to improve the bond can induce chipping and adversely affect the fit of all-ceramic restorations without significantly improving bond strength.[95,96]

Surface pretreatment for chemical bonding

For effective and durable resin-ceramic bond, not only micromechanical bonding but also a chemical bonding should be aimed. The most common and effective way to achieve a chemical resin-ceramic bond is through using silane coupling agents. Silane coupling agents are bifunctional molecules that improve the wettability of the ceramic surface and form a covalent bond with both the ceramic and the resin cement.[97] Silane agents commonly contain \( \gamma \)-methacryloxypropyl trimethoxysilane (\( \gamma \)-MPTS) as an active molecule. The reaction between methoxy silane groups of \( \gamma \)-MPTS and OH groups of the porcelain surface that formed siloxane bonds can be initiated and accelerated by using acid catalysis.[98]

Nowadays contemporary ceramic primers utilize separate acidic catalyst liquids, such as the 10-methacryloyloxydecyl dihydrogen phospha t (MDP) monomer, or carboxylic acid compounds.[99] When the acidic catalyst is mixed with the silane coupling agent, the methoxyl groups hydrolyze to form siloxane bonds (Si-O-Si) with the ceramic surface (Figure 1.5).

Accordingly ceramic primers can be classified as:[100]

1) unhydrolyzed single liquid silane primers.
2) prehydrolyzed single liquid silane primers.
3) two-or three-liquid primers with separate silane coupler and acid activator.

The single liquid prehydrolyzed silane primers have shown a better bonding performance than the unhydrolyzed form. However, the stability of prehydrolyzed silane primers appears to be insufficient and their shelf life is limited compared to the multicomponent liquid primers.[100]
Improvement in ceramic-resin bond strength can also be accomplished through heat treatment of the silanized ceramic. It is believed that during heating (100 °C for 60 s) water and other contaminants such as alcohol or acetic acid are eliminated from the silane treated surface, which drives the silane/silica surface condensation reaction towards completion and promotes silane silica bond formation.[96]

The try-in procedure is an important step for all-ceramic restoration to optimize fitting and color match. Etching and silane treatment are best to be accomplished after the try-in procedure to prevent contamination of the conditioned ceramic surface. Nevertheless, for the convenience of the dental practitioner, to save time at the chair side, many commercial dental laboratories etch and silanize the fitting surface of the ceramic restoration. When this pretreated surface is contaminated, during the try-in procedure, with saliva or blood, the surface has to be cleaned and silanized again before the application of the adhesive cement.[101] Cleaning can be carried out with phosphoric acid or acetone, where after the silane treatment has to be repeated.[102]

The silane solution can be affected by its storage condition. An improperly sealed container will permit evaporation of solvents, which increases the concentration of the coupling agent. The highly concentrated coupling agent can then act as a separating medium and adversely affect the resin-ceramic bond strength.[103]
Although, many laboratory studies have shown that only silane treatment without additional micromechanical bonding could provide sufficient resin-ceramic bond strength [98,104], the technical sensitivity of silane treatment and the complicated multi-step cementing procedure favor employing the chemo-mechanical bonding mechanism to insure a strong and durable bond.

HF etching followed by silanization, which enhances the resin bond to conventional silica-based ceramics, does not improve the resin bond strength to alumina or zirconia based ceramics. This is probably due to the inherent microstructure that is more resistant to HF.[105] Alumina based ceramics only have a small percentage of silica and zirconia ceramics contain no silica. This makes it is less likely that silane treatment of alumina and zirconia based ceramic surfaces can initiate effective chemical bonding. Tribochemical application of a silica layer by means of sandblasting (Rocatec system, 3M ESPE, Seefeld, Germany) followed by silane application provide long term durable bonds with BIS-GMA based resin composite cements to alumina based ceramics.[106] In addition, the ability of phosphate ester groups contained in some adhesive resin based cements (e.g. Panavia, Kuraray, Osaka, Japan) can also offer an alternative bonding mechanism to sandblasted alumina or zirconia based ceramics as it directly bonds to metal oxides.[107,108]

**Bonding to pre-processed composites**

The first composite inlays were made from a microfilled material, which was heat cured under pressure [109], followed-up in 1987 by a system based on hybrid composites, which were simultaneously light and heat cured (DI system, Coltène AG, Switzerland). The exposure to heat was to increase monomer conversion and enhance cross-linking to obtain improved physical and mechanical properties.[110,111] However, bonding of resin cements to preprocessed heat cured composite restorations was challenged by the reduced number of reactive sites due to the high degree of double bond conversion. For this reason, the surface of preprocessed composites has to be chemically and mechanically modified to improve bonding with resin cements. The capability of HF to alter the surface of preprocessed composites to enhance bonding is generally influenced by the nature of the reinforcing filler, like it is for all-ceramic surfaces. HF improves the bond strength to microfilled composite inlays, as it has a roughening effect by preferentially attacking the SiO₂ glass filler.[112] Additional silane treatment of the surface further enhances the bond strength, as the filler particles at the surface are potential sites for silanization. In contrast, HF has an aggressive etching effect towards glass filled hybrid composites. It causes total dissolution of the exposed glass filler particles and partially degrades the resin matrix, which has a negative effect on the bond strength.[113,114] Another approach to increase the resin-
composite bond strength is to roughen the surface by sandblasting or with diamond burs and then increase the wettability by silanizing the silica-containing filler.[115-117]

**Bonding to tooth structure**

Buonocore in 1955, showed that etching of enamel with phosphoric acid leads to preferential dissolution of inter prismatic enamel leading to a specific micro-pitched etch pattern.[118] By applying unfilled bonding resins to etched enamel, the resin penetrates these micro porosities by capillary attraction to form a resin-enamel interlocked layer.

From that time adhesive dentistry has evolved rapidly and numerous types of adhesive systems have been brought to the market for enamel as well as for dentin. Traditionally, dentin adhesive systems involve three separate components for each step in the process to produce micro mechanical bonding of directly placed restorations. In the first step tooth tissues are conditioned using strong acidic etchants to decalcify the smear layer and the superficial portion of the surface, followed by water rinsing. In the second step the conditioned surface is primed with a hydrophilic monomer that penetrates and wets the surface and increases the surface energy. In the last step a bonding agent is applied to couple the primed hydrophilic surface with the hydrophobic restorative material.

Since 1995, developments are in progress to reduce the number of components of bonding systems from three (etchant, primer, and adhesive) to two (etchant and primer-adhesive or self etching-primer and adhesive) and finally to one (self-etching adhesive).[119]

Beside the development of adhesive systems for bonding direct restorations, several adhesive resin cements that utilize self-etching primers for bonding indirect fabricated restorations have been recently introduced. The formulation of the self-etching primers to combine with these cements generally includes an aqueous mixture of acidic monomers, such as a phosphate ester or a carboxylic acid and hydrophilic monomers such as hydroxyethyl methacrylate (HEMA).[120,121] Due to their intrinsic acidity, these primers can simultaneously condition and prime the hard tooth tissues, integrating the smear layer in the bonding process.[122] The mechanism of simultaneous conditioning and resin penetration allows complete resin infiltration up to the same depth of dentin demineralization.[121] In addition, by omitting the acid etching and rinsing steps the sensitivity of the bonding procedure to the degree of dentin humidity [123] becomes irrelevant for these kind of adhesives.

With the self-etching primers, adhesive cements are applied directly to the primed tooth surfaces without an intermediate bonding agent. The advantage of this approach is that it reduces problems with properly placing indirect restorations. Resin cements that use separate bonding
agents that are pre-cured prior to cementation can decrease the space available for the luting cement, which can result in incomplete seating of the restoration.[124]

However, self-etching primers may induce an adverse chemical interaction with chemical-cure resin cements by their acidity. It has been reported that the amine activator in the chemical-cure composite could be inhibited by the action of protons (H⁺) from the acidic dentin adhesives, which resulting in low resin-dentin bond strength.[125,126]

More recently adhesive resin based cement was developed (Rely X Unicem, 3M ESPE), which does not need a pretreatment of the tooth surfaces, as it depends on the inherent acidity of the resin matrix to condition the tooth surface. The organic matrix of this cement consists of multifunctional phosphoric acid modified (metha)-acrylates and resembles silicate cements by the incorporation of basic inorganic filler within the matrix, which reacts with the acidic groups of the monomer.

The difficulty, however, with the self-etching systems is to obtain simultaneously appropriate etching of both the enamel and dentin. Although these adhesive systems are more user's friendly, the efficiency to bond to enamel has been a controversial issue. Hara et al.[127] reported that bonding of self-etching adhesives to ground enamel was inferior when compared with single-bottle and multiple step, total-etch systems that utilize phosphoric acid as a separate conditioner. On the other hand, another study showed that self-etching systems are satisfactory alternatives to phosphoric acid conditioning of ground enamel.[128]

Bonding to dentin is considered to be more technique sensitive than bonding to enamel, as it is affected by several variables. After preparation dentin is always covered with a smear layer, which consists of organic and inorganic material adhering to the underlying dentin. The bonding performance of the self-etching systems is assumed to be affected by the quantity and quality of the smear layer.[129] It was found that the bond strength to dentin prepared with diamond burs (thick smear layer) was lower than the bond strength to dentin prepared with fine fissure steel burs (thin smear layer) after conditioning with self-etching primers. This difference in bond strength was not observed for adhesive systems that use phosphoric acid to condition the smear.[130]

However, adhesives with the self-etching approach vary in their acidity by differences in composition and concentration of polymerizable acids and/or acidic resin monomers.[131] Consequently the interaction with the smear layer and the underlying dentin could vary. Strong self-etching adhesives with low pH values have shown to produce a demineralization effect similar to that of the total-etch approach [132], but resulted in rather low bond strength values.[133] On the other hand, mild self-etching adhesives that partially demineralize dentin can
use residual hydroxyapatite as receptors for additional chemical bonding with the carboxylic acid-based or the phosphate based monomers.[121]

1.4.4 Esthetic properties

The esthetic properties of luting cements (color, translucency, opaqueness, and color stability) are of considerable significance due to the increasing use of translucent ceramic restorative materials, especially for anterior restorations.[134]

Color

Mainly for veneers the color of the cement can influence the final shade of the restoration. Ideally, the adhesive resin cement should bring out the color of the underlying dentin and not serve as an opaque screen to mask this tissue. The final color of the restoration will then be the result of the light reflected and absorbed by the ceramic, the cement and the tooth as a whole.[135] However, one has to recognize that luting cements can hardly change a wrongly chosen color of an esthetic restoration into a matching color. Only a slight change of the shade can be reached with luting cements. Mainly the opaqueness of the cement is of importance for cases in which the dentist wants to hide discolored dentin, but also for these cases a thin cement layer will not be able to hide discolorations totally. Because they are much thicker than veneers, inlays and onlays will be affected mainly at their margins with a wrong choice of the cement. If the cement is opaque or of high chroma, the esthetic properties of the inlay decline and the margins become visible.[136]

The photo-initiator in resin based cement can influence the color selection. The color of the camphorquinone photo-initiator changes during curing from yellow to colorless; before curing the color of resin based cements may be more yellow than after curing. Some cements contain bis-acylphosphine-oxide (BAPO) as photo-initiator to avoid this effect. However wavelength of the light to initiate BAPO to start the setting reaction is 415 nm instead of 470 nm to initiate camphorquinone. Most curing lights based on LED technology do not cover the 415 nm wavelength can not be used to cure the BAPO-containing cements.

Color stability

Cements may discolor during lifetime due to absorption of dyes present in food. Two factors of importance for the sensitivity of cements to this discoloration are the degree of cure and the
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hydrophilicity of the cement. An insufficient degree of cure makes resin based cements prone to
discoloration and hydrophilicity allows water to transport dyes into the resin. The more
hydrophilic the resin is the less is the color stability of the cement. This was the main drawback
of the earliest resin modified glass-ionomer cements that contains HEMA, which is a very
hydrophilic acrylate.

Although also intrinsic discoloration from chemical accelerators, necessary for dual
polymerization, can cause the color of the luting cement to change over time, a study by Noie et
al. showed that this color change is visually imperceptible.[137]

1.5 Bond strength test

Sealing of the cavity is recognized as an important factor for the longevity of dental restorations.
As a consequence much research is carried out to improve the adhesion of dental restorative
materials to the dental hard tissues. During the last decade many new adhesive systems has been
introduced for which good clinical performance is claimed. Such claims have to be proven in
laboratory studies as well as clinically. In vitro, the evaluation of adhesion of dentin adhesives
has been performed by various test methods, including bond strength measurements,
microleakage, and contraction gap size measurements. Yet no relation between sealing capacity
and bond strength, making it hard to predict the clinical efficacy of adhesive systems.

Several factors can influence the results of bond strength tests, such as testing method, test
conditions, substrate properties, and storage conditions.[138,139] With regard to the test
methodology, many investigators have questioned the reliability of the conventional shear and
tensile bond strength tests. There are numerous reports on bond strength evaluation in which
tests are applied, not only with different designs, but also showing varying incomparable results
between studies using the same design. Furthermore, it has been shown that the size of the
specimens has a relatively large influence on the test results.[140,141]

A crucial factor in evaluating the usefulness of a specific bond strength test is a thorough
awareness of the stress patterns, which are involved in bond failure. For all tests the average
bond strength is calculated by dividing the load at fracture by the area of the bonded surface
(nominal bond strength). However, the calculated nominal bond strength values are only valid, if
the applied load is equally distributed throughout the entire bonded interface. One way of
demonstrating the validity of this assumption is by establishing repetitive adhesive mode of
failures during bond strength testing. Nevertheless, this is not the condition with the traditional
bond strength tests.
Finite element analysis (FEA) studies demonstrated that the manner in which loads are generally applied in common shear or tensile bond strength tests, results in non-uniform stress patterns.[142] Conventional bond strength tests also suffer from frequent cohesive failures within the bonded substrate. The presence of cohesive failures in the substrate could be more related to the geometry of test than to increased adhesion of the material. Using FEA, Della Bona and van Noort [143] found that the shear test would measure the strength of the substrate rather than the strength of the adhesive interface. In another study, Versluis et al. [144] confirmed that the dentin pull-out in shear tests was due in part to the biomechanics of the test and did not necessarily mean superior adhesive strength or that the cohesive strength of the dentin was reduced.

A way to avoid stress inhomogeneities at the interface is by bonding two-rod specimens of uniform cross-sections together and pulling them apart by applying the tensile loads at the free ends of the specimens.[143]

Sano et al. in 1994 have introduced the microtensile bond strength test (μTBS).[145] By cutting a large bonded sample into a series of smaller slabs, they found that the tensile bond strength was inversely related to the cross sectional area of the bonded surface. They also found that reducing the cross-sectional area of bonded area, the number of cohesive failures of dentin also reduced and became zero at about 2 mm². These findings were explained by the reduction in number and size of specimen flaws, as a result of the diminution in the specimen size.[146]

The original design of the μTBS test utilized dumbbell-shaped specimens that were trimmed from slabs by using high-speed rotary instruments. The rational for this design was to allow the tensile stress to be more uniformly directed towards the weakest interfacial region.[147] In another modified version of the μTBS test the tensile load is applied to composite-substrate microbars that have a small but uniform cross-sectional area throughout the entire length of the bar.[148] This approach, known as the “non-trimming version”, (Figure 1.6) places less stress on the adhesive interface during specimen preparation and allows to study materials producing low bond strength.[146] A disadvantage of this test version is that cohesive failures in the substrates occur more frequently when bond strengths become higher at the expense of adhesive failures.[149]

Pashley et al. [138] have stated a number of potential advantages for the μTBS test:
1) the test shows more adhesive failures and fewer cohesive failures.
2) higher interfacial bond strengths can be measured.
3) the test offers the possibility to measure regional bond strengths.
4) means and variances can be calculated for a single tooth.
5) bond strengths to irregular surfaces can be measured.
6) the test permits testing of very small areas.
7) the small cross sectional area of the specimens facilitates SEM examination of fractures.

The test can also be performed on teeth prepared exactly as they are restored clinically, because the bonded surface area is determined after bonding by the trimming process.[146,150]

The μTBS test was originally designed to test the resin bond to dentin as a substrate, however it can also be used to test the bond to enamel, ceramics or resin composite as substrates.[86]

**Figure 1.6** Schematic illustration of the non-trimming version of the microtensile bond strength testing technique. The bonded ceramic composite block (A) is vertically sectioned (B) by slow speed cutting saw into 1mm thick slabs and other vertical cuts perpendicular to the first cuts (C) are prepared to again 1 X 1 mm microbars (D).

### 1.6 Aim and outline of investigations

The increased use of tooth-colored restorative materials and techniques as alternatives for the traditional metallic restorative materials has been documented by several studies. However, with the introduction of these new materials and technologies to the profession, comprehensive scientific validation of their efficacy is often not available. This leads for obvious reasons to confusion with respect to clinical decision making regarding the best choice of these materials for a particular restoration and the expected clinical success. Therefore, a combined project between the Department of Dental Material Science and the Department of Oral Function (Academic Center of Dentistry, Amsterdam) was started to study the different parameters that
can influence all-ceramic restorations through *in vivo* and *in vitro* studies. The parameters were, tooth preparation design, mechanical properties of ceramic materials, color determination and optimization, fitting accuracy, resin cement adhesion, and clinical longevity of the final restoration. The studies reported in this thesis make part of this combined project.

The aim of this thesis was to study different approaches that can be used to:

1) improve the esthetic result of restorations fabricated by machining prefabricated restorative blocks and
2) enhance bonding of these restorative materials to dentin with adhesive resin cements.

In Chapter 2, through clinical case reports, a laboratory veneering porcelain was used to study whether a thin veneering layer can improve the aesthetics of all-ceramic onlays fabricated by the CEREC CAD/CAM system from homogeneous, monochromatic ceramic blocks.

A strong and durable bond of these restorations to the tooth tissues by adhesive resin cements is crucial for their clinical success. In Chapter 3, the influence of surface pretreatments on the bond strength to prefabricated CAD/CAM ceramic and composite blocks was investigated for three different resin composite cements, and in Chapter 4 the durability of the resin-ceramic bond was tested. Durability was evaluated after conditioning the ceramic surface with two different acids. This study was also designed to address the effect on the resin-ceramic bond durability by using adhesives of various degrees of hydrophilicity.

With regard to bonding to dentin, adhesive resin cements that use the self-etching approach are more user's friendly than those using the etching and rinsing approach. These self-etching adhesives vary in their acidity by differences in composition and concentration of acidic resin monomers. In Chapter 5, the effect on the cement-dentin bond strength was evaluated by varying the conditioning time with self-etching primers of different acidity of three contemporary adhesive resin cements.

Bond strength determinations performed in Chapters 3, 4 and 5 were according to the methodology of the microtensile bond strength test. In Chapter 6, factors that can influence the results of this test methodology, such as the way of specimen attachment to the testing device and specimen dimension were investigated.

1.7 References


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[23] Zidan O, Ferguson GC. The retention of complete crowns prepared with three different tapers and luted with four different cements. J Prosthet Dent 2003; 89: 565-571.
Chapter 1


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Chapter 1


