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Summary and Conclusions

Besides restoring the biological functions of defective tooth tissues, modern restorative dentistry also aims to restore the esthetics of the tooth. While tooth colored materials such as ceramics and resin composites can produce esthetically satisfying restorations, adhesive materials and techniques through their ability to integrate the restorative interfaces can achieve the functional and the biological objectives. Currently, hybrid resin composites are the material of choice for restoring small and medium sized cavities. However, the shrinkage stress development during setting in direct resin composite restorations is still one of the major drawbacks of this material. 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. Therefore, in case of restoring large sized cavities where also re-establishment of the anatomical form of the tooth can be a demanding task, the indirectly fabricated tooth-colored restorations made of composites or all-ceramics appear feasible alternatives. The indirect restorative approach offers better surety to construct an appropriate tooth form and anatomy. Moreover, the shrinkage stresses generated during setting are limited only to the thin resin-cement layer, which is used to bond the restoration. The key success of such restorations relies to a great extent on the mechanical, biological, esthetical and adhesive characteristics of the luting cements.

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 restorations made by indirect techniques through *in vivo* and *in vitro* studies. The study presented in this thesis is part of this project and had two main objectives:

- 1) to improve the esthetics of restorations produced by machining prefabricated restorative blocks.
- 2) to enhance the bond strength of these restorative materials to dentin by adhesive resin cements.

In **Chapter 1**, an introduction is presented about materials and techniques used for fabrication of tooth colored indirect restorations and their advantages over the metallic and direct resin based composite restorations. The use of monocolored ceramic blocks has provided satisfactory esthetics for posterior inlays fabricated by the CEREC system. However, the use of these blocks to produce onlays, which are readily visible in the mouth, may lead to esthetically displeasing

results. Ultimately, only porcelain layering can optimize the esthetics of the restoration. In addition, individual effects in the adjacent teeth, such as decalcifications and opalescent effects can be imitated by stains to offer a lifelike appearance to the restoration. Apart from improving esthetics, the adjustment of the occlusal table with porcelain veneering will also improve the occlusion and strength of the final restoration. **Chapter 2** described a restorative approach to achieve a satisfactory esthetic all-ceramic restoration fabricated by CEREC-technology in 27 patients. This approach included an experimental onlay preparation design with shoulder finish lines; modification of the digital drawing to reduce the restoration occlusal table leaving room for porcelain veneering; and laboratory porcelain veneering and staining of the milled ceramic onlay to optimize its esthetic. An observation period varying between 1 and 4 years after placement showed no fractured restorations. Therefore, it was concluded that the described technique for porcelain-veneered CAD/CAM onlays was able to create a layered esthetic, functional, and strong all ceramic restoration.

In **Chapter 3**, the microtensile bond strength test was used to evaluate the bond strength of resin cements to prefabricated ceramic and composite block surfaces after various surface pretreatments. Three surface pretreatments were evaluated:

1. application of adhesive resin prior to the application of the cement,
2. etching with hydrofluoric acid (HF) and silanization, and
3. a combination of the previous two treatments.

For the ceramic blocks, it was found that treatment of the surface with HF followed by silanization is necessary to provide an adequate resin-ceramic bond. In addition application of adhesives to HF etched and silanized ceramic block surfaces, prior to the application of the cement, only increased the bond strength, if the adhesive contained fillers. For the prefabricated composite blocks application of an adhesive, prior to cement application, improved the bond strength in all cases, whether the surfaces were etched or non-etched. It was also observed that bond strength values obtained with composite blocks were superior to those obtained with ceramic blocks. This indicated that a more brittle material like a ceramic tends to fracture at the adhesive interface at lower values than the more resilient composite. In addition, composite as a substrate of bonding may better yield to the shrinkage stresses generated during the resin cement polymerization.

The durability of the resin-ceramic bond is crucial for the longevity of all-ceramic restorations. Deterioration of the bond by time could result in loss of support for the brittle ceramic material with subsequent fracture of the restoration, leakage, colour alteration or dislodgment of the restoration. In **Chapter 4**, the influence of two main parameters on bond

strength and bond durability of two resin cements bonded to prefabricated ceramic blocks was investigated. The first parameter was the type of acidic conditioner used, which was either hydrofluoric acid (HF) or phosphoric acid (H_3PO_4) and the second was the nature of the intermediate bonding agent (hydrophilic or hydrophobic) applied prior to cement application. Two different hydrophilic adhesives were tested (OptiBond Solo Plus, and Syntac Single Component) and one hydrophobic adhesive (Visio Bond). Visio Bond, in contrast to Syntac Single Component and OptiBond Solo Plus, has marked hydrophobic properties, as it solely consists of a tricyclic aliphatic diacrylate monomer, a saturated hydrocarbon with two acrylate groups. The resin-ceramic bond durability was tested on resin-ceramic microbars used in the microtensile bond strength test. The microbars had a cross-sectional area of 1mm^2 and were stored in water at 37°C for 1 day, 7 days, or 28 days. It was found that acid treatment with HF followed by silanization provided a stronger and more durable resin-ceramic bond in comparison to acid treatment with H_3PO_4 followed by silanization. In addition, when using an intermediate bonding agent between the cement and the ceramic, the degree of hydrophilicity of these adhesives had a significant influence on the resin-ceramic bond durability. Initially the bond strength was improved by the application of an intermediate bonding agent, however in water the hydrophilic bonding agents rapidly lost strength due to water sorption. On the other hand, the hydrophobic bonding agent showed a stable bond in water, while as suggested in this study, the bond strength of the particular hydrophobic bonding agent investigated can probably be improved by a small change of its molecular structure by attaching the C=C functional groups to longer chains.

A decline in bond strength after long-term water storage was also recorded for resin-dentin bonds by previous investigations [Chapter 1 ref # 66-69]. Hydrolysis of the collagen fibrils just below the hybrid layer and/or hydrolytic breakdown of the adhesive due to incomplete conversion of monomers by the action of moist dentin were considered to be major causes. However, in the present study (Chapter 4) the substrate was a ceramic material, which in contrast to dentin is hydrolytically stable. The decline in cement-ceramic bond strength of hydrophilic bonding agents on water storage as found in this study indicated that instability of the hydrophilic adhesives due to water sorption may also play an important role in the deterioration of the resin-dentin bonds. These findings make it attractive to evaluate bond durability of various types of dentin adhesives by applying the methodology as described in Chapter 4 on microbar specimens with the stable ceramic material as substrate instead of dentin as a substrate.

Adhesion to dentin as the second substrate involved in bonding indirect restorative materials by adhesive resin cements was investigated in **Chapter 5**. The dentin bond strength of three different contemporary adhesive resin cements that use self-etching primers with different acidity were studied. Two of these cements were dual-cured resin cements while the third one was chemically cured PMMA based resin cement. The study evaluated the influence of increasing the conditioning time of the self-etching primers (30, 60 or 180 s) on dentin bond strength of these cements. It was hypothesized that unlike the total etch adhesive systems, prolonged conditioning times of the dentin surface with self-etching primers can improve the bond strength. To explain and support the results also a scanning electron microscopy (SEM) study was performed on fractured surfaces of specimens from the bond strength test as well as on intact resin-dentin interfaces and dentin surfaces that were treated according to the various treatment times. The results for the two resin cements that utilize aggressive self-etching primers (low pH) supported the hypothesis. However, this was not the case with the resin cement with a less acidic primer (higher pH), where the bond strength to dentin did not improve when the treatment time with the primer was extended. SEM showed that the less acidic self-etching primer was not able to completely remove the smear layer and plugs. These results suggested that in order to create a strong resin-dentin bond the self-etching primer should be able to completely dissolve the smear layer and plugs to allow appropriate infiltration of the highly filled resin cement. An interesting observation from the SEM study was the presence of a porous structure at the fractured resin-dentin interface, when the dentin was treated with one of the primers with the lowest pH. As the voids were so great in number, they were probably generated by carbon dioxide gas production from the reaction between the acidic primer and the carbonate in the dentin apatite and captured in the interface during setting.

Bond strength testing is one of the methods often used to evaluate the bonding capacity of adhesive systems to tooth tissues and other substrates such as ceramic or composite. The microtensile bond strength test as an alternative to the conventional tensile or shear bond strength tests has been used by many laboratory research groups. However, knowledge about stress distribution and concentration within the specimens during tensile loading has not been fully developed yet. Such knowledge can facilitate standardization of the microtensile test method to obtain more reliable results. In **Chapter 6**, an experimental study combined with a Finite Element Analysis (FEA) study was conducted to investigate the influence of specimen attachment and specimen dimension on the microtensile strength (μ TS). It was hypothesized that the inverse relationship between specimen size and the microtensile bond strength (μ TBS), as has been reported in the literature [Chapter 1 ref # 139, 144] is mainly caused by the lateral way

of attachment of the specimens to the testing device. To test the hypothesis three different dimensions (1x1, 1x2 and 1x3 mm) of composite bars were cut from composite blocks and attached at their 1 mm wide side to the μ TS test device for determination of the μ TS. FEA was carried out to determine the patterns of stress distribution involved. The results of this study demonstrated a clear dependence of the μ TS on the thickness of rectangular composite bars. The thinner the specimens became (3mm \rightarrow 2mm \rightarrow 1mm) the higher were the values for the μ TS. This outcome arose, as the loads at fracture for the three different specimen sizes were of the same magnitude, while the cross-sectional area decreased (3mm² \rightarrow 2mm² \rightarrow 1mm²). The rationale for this inverse relationship between the μ TS and thickness was obtained from the FEA. This showed that with lateral attachment the resultant stresses were not uniformly distributed and that stress concentration near the points of specimen fixation to the test set-up occurred with approximately the same magnitude. The hypothesis that the μ TS is dependent on the thickness of the specimens when the attachment is at the sides of the specimens was therefore accepted. The results of this study explain the sensitivity of the μ TBS to minor changes in specimen size when these are attached at their lateral sides and indicate that the smallest possible specimen thickness would provide the most favorable stress distribution pattern during the microtensile bond strength testing.

General Conclusions:

- 1) Laboratory porcelain veneering of the monochromatic machined ceramic restorations could serve to improve esthetics, function, and strength.
- 2) Restoration surfaces milled from prefabricated composite blocks have a higher bond strength to resin cements than surfaces milled from ceramic blocks.
- 3) Hydrofluoric acid etching followed by silanization of the prefabricated ceramic material is necessary to create strong and durable resin-ceramic bonds.
- 4) Adhesives used as an intermediate between cement and ceramic that contain hydrophilic monomers have a negative influence on the resin-ceramic bond durability when stored in water.
- 5) Dentin bond strength of resin cements that use more aggressive (low pH) self-etching primers can be improved by increasing the primer conditioning time.
- 6) A more favorable stress distribution during tensile loading in specimens attached with their lateral side to the test device for microtensile (bond) strength testing can be obtained when the specimens are cut with the smallest possible thickness.

Future Prospects

Although the technique described in **Chapter 1** can provide a satisfying esthetic and functional all-ceramic restoration, it will lack one of the main advantages of a restoration made by the CEREC-technology, which is the possibility to deliver a chair side restoration during a single appointment. However, if preprocessed composite blocks are used, in-office staining and characterization of the final restoration may be feasible.

In **Chapter 4**, it was speculated that the bond strength of adhesives with monomers like that of Visio Bond could be improved if the two functional C=C-groups would be placed on longer chains, further away from the tricyclic aliphatic group for more effective coupling with the silane-C=C. Combined with the high water stability, such monomers would be promising bonding agents in porcelain repair. Also composites based on this monomer when applied directly to HF-etched and silanized porcelain surfaces could be an option for strong and durable repairs.