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# Zirconia enriched dental adhesive: A solution for OCT contrast enhancement. Demonstrative study by synchrotron radiation microtomography

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## ABSTRACT

**Objective.** The major aim of this study was to prove the capability of the optical coherence tomography (OCT) method in visualizing the integrity of the adhesive fillings and of the interfaces between the adhesive, tooth structures and composite resin. As zirconium dioxide was added to the composition of the adhesive layer in order to strengthen the backscattered light in the OCT investigation, for a better visualization of the interfaces, the determination of a proper zirconia concentration was another aim of our study.

**Method.** Several class II cavities were prepared in human premolars and were filled with dental adhesive containing different zirconia concentrations and light-curing composite resin. Both OCT and synchrotron radiation microtomography (micro-CT) were used to analyse the morphology of the tooth–adhesive–composite interfaces and to investigate the adhesive layer.

**Results.** The pore distribution, both at the interfaces level and in the resin, and the analysis of the adhesive layer integrity were obtained. A good agreement between OCT and micro-CT analyses was observed in terms of detecting discontinuities in the adhesive layer. Furthermore, micro-CT showed that zirconia percentages in the adhesive higher than 20 vol.% lead to conglomerates formation, which can negatively influence mechanical properties. Meanwhile, OCT confirmed a factor of 3 for the contrast enhancement when 20% of zirconia was included in the adhesive composition.

**Significance.** The present study proved the capability of the OCT method in visualizing the morphology and integrity of zirconia doped tooth adhesive fillings, to be used for a further in vivo tool development.

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## 1. Introduction

The treatment of carious lesions requires removal of affected dental tissue, thus creating cavities that need to be filled with dedicated materials. The restoration of such cavities consists in several steps, including etching of enamel and dentin with 37% orthophosphoric acid, rinsing and drying, followed by application of an adhesive and insertion of a light-cured composite resin. One of the problems that arise when using light-cured resins is the marginal gap formation due to the polymerization shrinkage. The polymerization process induces tensions at interfaces that may lead to cracks and, consequently to marginal leakage. Another factor leading to marginal gap formation is represented by differences in thermal expansion between tooth structure and resin [1,2], which can in average reach 2–3%, with values of up to 3.5% when using flowable materials. Such gaps may worsen and even new gaps can develop due to the temperature variations or due to the occlusal load the restorations are subjected to [3]. Marginal gap formation in turn leads to microleakage which is defined as the penetration of fluids from the oral environment at the resin tooth interface [4]. Microleakage has been investigated through a series of methods that are however invasive and can only be performed under laboratory conditions on extracted teeth. Scanning electron microscopy is widely used in the quantitative analysis of the margins [5], light, confocal microscopy and transmission electron microscopy are also applied for the examination of hard dental tissues/adhesive interfaces [6,7]. The main disadvantage of these methods is their invasiveness that renders them unsuitable for clinical practice [8]. Another disadvantage of the above mentioned investigation methods is that they all require specimen preparation such as sectioning, staining or decalcification, accompanied by loss of important information during the experimental protocol [9].

A relatively recent investigation method of the tooth–restoration interface is the optical coherence tomography (OCT). This is a non-destructive, non-invasive imaging technique that has proved its diagnostic usefulness in ophthalmology [10], art [11], cardiology [12], dermatology [13], gastroenterology [14] and many other medical and non-medical fields.

An important advantage of the OCT method is its potential for *in vivo* evaluation of the integrity of the fillings and of the interfaces between them and the teeth structures. Due to its non-invasiveness, some of the dental fillings could be optimized during the same dentist appointment. For instance, the dentist can notice via OCT that the filling interfaces could lead to secondary cavities due to absence of the adhesive or material defects situated at that level. OCT allows fast diagnosis and 3D reconstructions of interfaces of interest. Studies conducted up to now have shown the effectiveness of this method in the examination of structural quality of direct and indirect dental restorations [15].

When specifically used for the investigation of interface between the hard dental tissue and composite resin, OCT is limited due to the appearance of the adhesive layer, which sometimes cannot be told apart from air. Interfaces between different materials are detected in OCT based on the

difference between their indices of refraction [16]. The normal tooth adhesive has a quite low refractive index. In order to better visualize such challenging interfaces, materials doping the adhesive and enhancing its contrast can be used to strengthen backscattered light [17]. Indeed, the adhesive layer appears as a transparent zone in OCT images, therefore aeric inclusions present low contrast and are hard to be confirmed. Therefore, it would be appropriate to increase the contrast of the adhesive layer by altering its optical properties. This should be done in such a way as not to affect its mechanical and esthetic properties.

Zirconium dioxide appears to be suitable for such study due to its biocompatibility and lack of adverse effects to dental structures and due to the outcome of the esthetic restoration [18]. In this work, zirconia particles were added to the adhesive in three different concentrations in order to evaluate the most suitable solution for the fillings to enhance the contrast of the adhesive.

Another non-destructive and non-invasive investigation method is the synchrotron radiation-based microtomography (micro-CT). This is a powerful tool for a better understanding of the morphological characteristics of the site of interest, offering the possibility to obtain a high resolution 3D bulk reconstruction of extracted teeth in a non-destructive way.

Micro-CT was successfully applied for advanced characterization in bone [19,20] and bone tissue engineering [21,22], cardiology [23], muscular dystrophy research [24] and other medical and technological fields [25,26]. Micro-CT was employed also for tooth and relative problematic non-destructive investigations [27]. Micro-CT is similar to conventional computer tomography (CT) usually employed in medical diagnosis and in industrial applied research. Unlike these systems, which typically have a maximum spatial resolution of about 0.5 mm, micro-CT is capable of achieving a spatial resolution of up to 0.3 microns, i.e. about three orders of magnitude higher. For non-destructive teeth investigations by micro-CT, a resolution of a few microns is normally used in order to keep the entire tooth volume in the field of view. The use of X-rays delivered by Synchrotron Facilities has several advantages compared to X-rays produced by Laboratory sources. It includes the possibility to take advantage of the high photon flux, which guarantees the achievement of high spatial resolution with good signal-to-noise ratio. Furthermore, the Synchrotron-produced X-ray beam is tuneable. This allows performing measurements at different energies. The use of monochromatic X-ray radiation also eliminates the beam hardening effects.

In this work, micro-CT was used to study the morphology of the tooth–adhesive–resin interfaces and evaluate the best zirconia concentration in the adhesive layer in order to optimize the OCT contrast on the same specimens. Furthermore, micro-CT experiments aimed at the localization of discontinuities in the adhesive layer, the results being in good agreement with those obtained by the OCT investigations.

## 2. Materials and methods

Human premolars (nr=20, crack and caries free) extracted for orthodontic purposes at the Faculty of Dental Medicine,

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