MICROSCOPIC AND SPECTROSCOPIC ANALYSIS OF THE TOOTH-COMPOSITE INTERFACE

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Abstract: The purpose of this study is to evaluate the quality of the tooth-composite interface. *Material and Method*: Composition-based zirconia-silica commercially available as Valux Plus TM was used as repair material for a permanent premolar cavity dimensions of 4 mm long, 3 mm wide and 2 mm deep. After restoration was extracted premolar orthodontic reasons, and prepared a cross section containing the interface between composite and natural tissue being examined by SEM microscopy and FTIR spectroscopy. *Results and discussion:* Restoring anorganic is loaded with 71% by volume of zirconium-silica particle sizes between 3.5-0.01 microns. 10% silica content of Zn-Si composite were broadcast in the natural tissue, with good adhesion to biocomposite the natural tissue. *Conclusions:* Restoration of Zn-Si is successfully used to restore premolar, as demonstrated by FTIR spectroscopy and SEM microscopy.

Keywords: composite zirconia-silica based, tooth-composite interface, microscopy, spectroscopy.

INTRODUCTION

Resin based materials are widely used as direct restorative materials. Composite resins consist of a heterogeneous blend of organic resins and inorganic fillers with excellent esthetic and physical properties. These have enabled the dental profession to advance tremendously in cosmetic dentistry. The gap formation between filling materials and tooth structure has been a major dental problem that is responsible for cases of secondary or recurrent caries, discoloration as well as pulpal inflammation and re-infection. The dental practitioner must carefully evaluate the probable abrasive effects of using certain restorative materials like porcelain or resin composites to oppose existing tooth structure. The surface of the composite filling contains both resin matrix and filler particles. Therefore, if there is a mismatch between the filler hardness and surface roughness of the composite and that of enamel, there is a danger that the enamel will be worn down. During mastication, composite filler particles may scratch and abrade the antagonistic enamel. The vertical dimension will thus decrease if premolar and molar regions are treated with such a restorative material. Therefore, the surface texture and the particle size, shape, size distribution, and hardness play important roles in determining the biological strength of composite restorations [1]. A smooth surface provides reduced frictional wear at the occlusal contact areas. This smoothness will benefit the composite wear as well as the antagonistic enamel wear. To meet smoothness requirements, the intrinsic surface roughness of resin composites must be equal to or less than the average roughness value of enamel-to-enamel occlusal contact areas.

The objective of this study is to evaluate the quality of interface upon using a Zirconia-Silica based composite in premolar restoration, by SEM microscopy and FTIR spectroscopy. Scanning electron microscopy analyses combined with local quantitative chemical analyses based upon the X-rays characteristic spectrum (EDS) for the composition elements allow the observation of the details of the surfaces morphology concomitant with the elemental analyses in situ, at the interface between the composite and natural tissue (dentine). On the other hand, FTIR spectroscopy allowed for a quick differential identification of typical dental materials produced from organic compounds for inorganic restorations and of tooth structure-resembling hydroxyapatite and its contaminate forms with fluoride and carbonate ions. For the purpose of assessing mineral properties of calcified tissues i.e. bone and tooth, the vibrational spectroscopy, based on Fourier techniques is required [2, 3]. Thus, the researchers used these methods to carry out comparisons of dental tissues including enamel and dentin and to examine the hydroxyapatite single crystallites. The chemical composition of apatite is significant because of the consequences, which ensue from different chemical and physical properties [4]. Moreover, the application of near-infrared spectroscopy was also reported as a useful tool for the characterization of minerals, especially combined with hydroxyl groups or water bonded in the structure [5]. The study of sound enamel using X-ray microtomography (XMT) was performed to assess quantitative mineral distribution in occlusal pit and fissure of the teeth. These sites of the teeth are the most susceptible for primary carious lesions. Hence is essential meaning such of examinations for dentistry [6].

MATERIALS AND METHODS

Zirconia-Silica based composite commercially available as Valux TM Plus was used as restorative material in the case of a permanent premolar with the cavities dimension 4mm in length, 3 mm in width and 2 mm in depth. Upon restoration, the premolar was extracted for orthodontic reason, and a transversal section containing the interface between the composite and the natural tissue was prepared by polishing the surface using a water-cooled polishing machine at 10,000 rpm.

The surface morphology was investigated using a scanning electronic microscope type 5600 LV JEOL equipped with an X-rays spectrometer type Oxford Instrument, with the following characteristics: resolution 3.5 nm with secondary electrons; enlargement 300,000x; local quantitative chemical analyses based upon the X-rays characteristic spectrum (EDS) for the elements listed between boron and uranium, with the detection limit of 0.01%. The specimen surface was also investigated by FTIR spectroscopy using an Elmer-Perkin BXII spectrometer, equipped with an ATR Miracle device (single reflection ATR with ZnSe crystal). The spectra were collected in the range 400 to 4000 wave numbers [cm^{-1}], with the spectral resolution 2 cm^{-1}.

RESULTS AND DISCUSSIONS

Valux TM Plus composite is a visible-light activated, radiopaque material designed for used in both anterior and posterior restorations. The inorganic filler is zirconia-silica loaded 71% by volume with particle size range of 3.5-0.01 micron. It also contains BIS-GMA and TEGDMA resins. The SEM images of region corresponding to the interface between the composite and the natural tissue (dentine) is presented in Figure 1, along with the local elemental analysis in both sides of the junction.

A

B

C

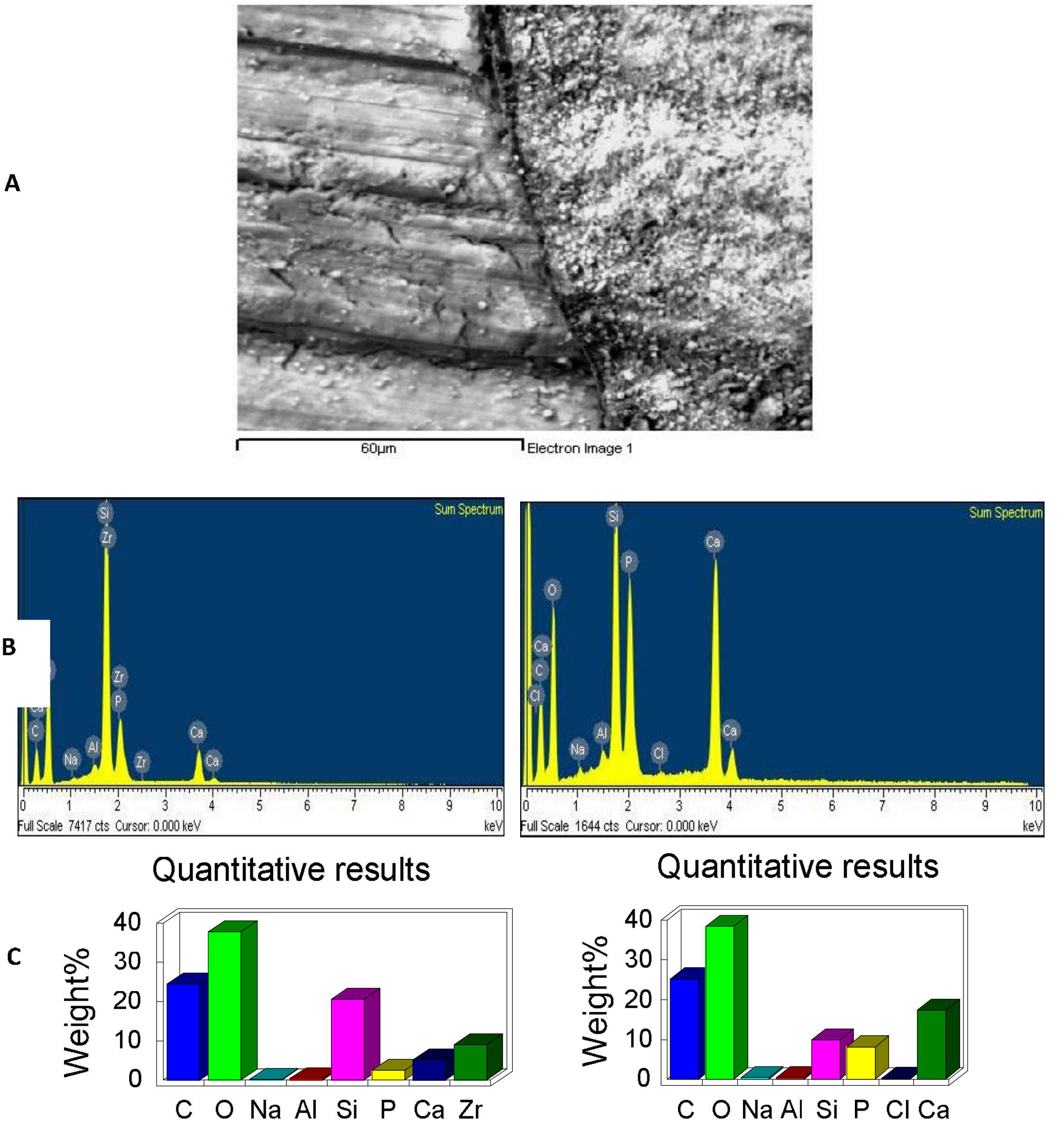

Fig. 1A. The surface morphology of the region corresponding to the interface between the composite and the natural tissue: right side- dentine, left side- Zn-Si composite. Magnification X300. 1B. X-rays characteristic spectrum (EDS) corresponding to dentine region (right) and Zn-Si composite (left side), 1C. local quantitative chemical analyses corresponding to left and right side of the junction, respectively.

Both the SEM image and the quantitative chemical analyses revealed a good biocompatibility of the composite and a successful medical procedure, as the micrograph indicate no gap between tooth structure and composite. Moreover, as a result of silanisation, 10% of silica content in Zn-Si composite was diffused in the natural tissue, revealing a good adherence of the biocomposite to the natural tissue. The placement of a low –viscosity and adhesive bonding resin before the restorative resin material readily wets tooth surface and allows flow of material into the micro pores created by the etching process.

In order to sustain the chemical and structural composition of the dental composite in contact with the natural tooth, the characteristics of the ATR-FTIR spectra of the same specimen was investigated and presented in Figure 2.

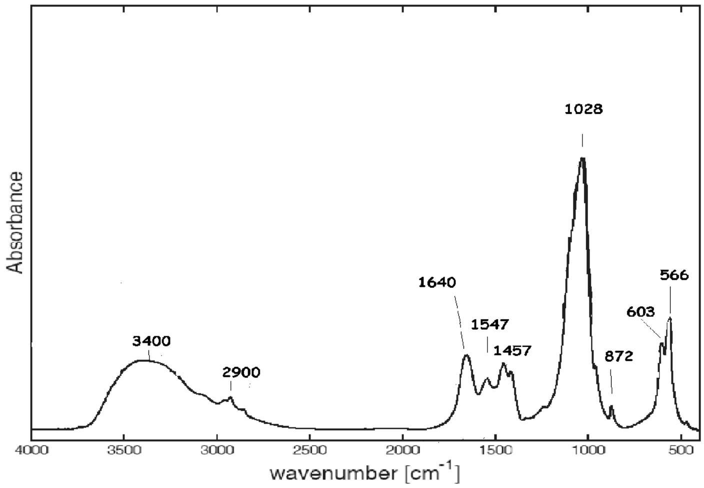

Fig. 2. ATR FTIR spectra of the specimen containing the Zn-Si filler in contact with the natural tooth (junction).

The typical peaks were found at around 566, 603 and 1028 cm^{-1} associated with phosphate ion PO_4^{3-} (very intensive, broad band). Moreover, within the broad absorption band (the peak at 1028 cm^{-1}) the characteristic dominant band at 872 cm^{-1} was exhibited which was attributed to carbonate ion (CO_3^{2-}) [7]. This IR spectra also included the most intense peaks at 1415 and 1457, 1547, 1640 cm^{-1} which can be linked to carbonate ion (1415 and 1457 cm^{-1}), next amide II (-CO-NH-) and amide I (-CO-NH2-) groups, respectively [7,8]. The broad band at 3400 cm^{-1} is related to Si-OH vibration group. Comparing the above results with the existing FTIR spectra of the natural dental tissue (dentin, enamel), this analysis confirmed the presence of the inorganic components of the filler along with the dentin organic components in the specimen.

CONCLUSIONS

We conclude from our study that Zn-Si filler are successfully used in premolar restoration, as demonstrated by SEM microscopy and FTIR spectroscopy. Elemental analysis confirms also a good adherence of the composite to the natural tissue. The FTIR spectroscopy was a useful tool concerning the identification of chemical functional groups of the filler material in contact with the natural tissue.

REFERENCES

1. SAM'AN M. MASUDI, ROSLIN N., SULAIMAN S., Microsc. Microanal. 9, **supl.3**, 364-366, 2003.
2. CARDEN A., MORRIS MD., J. Biomed. Opt. 5, 259 (2000).
3. PASCHALIS EP., DICARLO E., BETTS F., SHERMAN P., MENDELSOHN R., BOSKEY AL., Calcif. Tissue Int. **59,** 480 (1996).
4. DOWKER SEP., ELLIOTT JC., WILSON GR., CLOETENS P., Eur. J. Oral **Sci. 114**, 353,(2006).
5. FROST RL., ERICKSON KL., Spectrochim. Acta A Mol. Biomol. Spectrosc. **61,** 45 (2005).
6. FRANZ A., KÄONIG F., ANGLMAYER M., RAUSCH-FAN X., GILLE G., RAUSCH WD., LUCAS T., SPERR W., SCHEDLE A., Dent. Mater**.** **19,** 382 (2003).
7. KAKEI M., NAKAHARA H., KUMEGAWA M., YOSHIKAWA M., KUNII S., Biochim. Biophys. Acta **1524,** 189 (2000).
8. MARTIN N., JEDYNAKIEWICZ NM., FISHER AC., Dent. Mater. **19,** 77 (2003).