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Atomic Force Microscopy study for assessing the characteristics of news materials sealants

KEYWORDS: biomaterial, atomic force microscopy, sealants.

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ABSTRACT *The degree of finishing for a surface depends on the structural features of the materials used. We proposed the comparative assessment "in vitro" of the topography and roughness for 4 sealing resin composite materials. The materials used were: Fissurit® FX (VOCO, Germany), DEFENSE CHROMA (ANGELUS®, Brasil), WAVE (SDI, Australia), PermaFlow™ (Ultradent, USA). Topography and roughness were analyzed by Atomic Force Microscopy (Park SYSTEMS XE -100) for a 5µm × 5µm with scan rate of 0.5 Hz and a resolution of 256 × 256 pixels. Tridimensional topographic analysis was performed by analysis software (XEI - Image-Processing and Analysis). Surface roughness was measured for each material in nanometers. There were differences in surface roughness among the sealants but their values were significantly different in favor of PermaFlow™ (Ultradent, USA).*

INTRODUCTION

The analysis of the morphological characteristics of sealing materials used in the primary prevention of dental caries is a theme by strict actuality.The morphologies, microstructures and compositions of some bioadhesive materials were systematically investigated by scaning electron microscopy (SEM), optical microscopy (MO), Energy-dispersive X-ray spectroscopy (EDS) and atomic force microscopy (AFM) analysis. (Saveanu (2011), Khalichi and Singh (2008), Oong, et al. (2008)).

De asemenea degradarea răşinii compozite favorizează retenţia de placă bacteriană cu favorizarea creşterii nivelului de *Streptococcus mutans*.(Khalichi and Singh (2008), Oong, et al. (2008))The structural characteristics of the sealing material offers the possibility to obtain a smooth surface which does not favor the formation, accumulation and plaque development. (Saveanu (2011)) It was found that the bacterial biofilm would accelerate the degradation of composite resin, causing the elimination by monomers in the oral environment. Also, the degradation of composite resin of favors the retention of plaque and increasing levels for Streptococcus mutans. (Khalichi and Singh (2008), Oong, et al. (2008))The ability of sealing material to prevent tooth decay depends by the technique of cleaning and by the technique of dental surface preparation and also by the physical, chemical, mechanical, rheological and biological characteristics of the materials.(Gray and Griffin (2009), Agrawal and Shigli (2012)The wear resistance of a material is strongly related to structure of the material used and of course with the degree of surface roughness. Also if the surface is smooth the wear resistance is higher. Because the degree of finish of a surface depends by the structural features of the materials used, we decided to analyze several types of resin composite materials used for sealing.

The purpose of this study *in vitro* was to evaluate by atomic force microscopy the morphology and the roughness for four sealing material resin composites.

METHODS

For AFM analysis were prepared samples of four different materials composite resin according to the table 1.

Table 1. Lists of investigated materials

Code	Brand Name	Manufacturer	Filler %	SM
F	Fissurit® FX	VOCO, Germany	-	L
D	Defense Chroma	ANGELUS®, Brasil	50	L
W	Wave	SDI, Australia	63	L
P	PermaFlow™	Ultradent, USA	68	L
SM - Setting Mechanism L- Light curing				

The materials were applied in conformity with manufacturer indication between two matrix strips of celluloid (Nr. 437 Alfred Becht GmbH-D 7600 Offenburg, Germany) and then between two glasses plates to obtain a flat surface. The materials samples with photo initiator were light-cured for 40 seconds through the glass slide with a conventional quartz halogen lamp (QTH), power density 570mW/cm^2 (3MESPE). After polymerization the materials were removed and analyzed. The microstructures and the composition of biomaterials were characterized by SEM (JEOLJSM 6390^a Japan). The roughness and the topography of the surface were characterized by AFM (Park SYSTEMS XE -100). AFM was used in non-contact mode using single crystal silicon tip (with nominal radius < 10 nm), which was connected to a fixed substrate on a canti-lever. The images were recorded with a scan rate of 0.5 Hz and a resolution of 256 × 256 pixels. For each specimen, two scans were carried out at each specimen surface quadrant at a scanning area of 5 µm × 5 µm. The collected 3D topographical data was analyzed with data analysis software (XEI - Image Processing and Analysis). For each group, the surface roughness was calculated in nm. (Saveanu (2011) Saveanu, Dragos and Chiriac (2012))

RESULTS

Surface roughness

Assessment through AFM (atomic force microscopy) of the degree of roughness of the materials showed that the level of roughness varies significantly between samples.After specimens were activated, the lowest surface roughness was measured for the Perma Flow. The results are listed in Table 2.

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Table 2.Analysis of the surface roughness [nm] for 5 µm ²obtained from AFM images.

	F	D	W	P
Min	-28.08	-143.06	-32.32	-20.12
Max	39.34	25.57	90.49	27.05
Md	5.65	-58.74	29.13	.3.46
Mean	0.00	.3.04	0.53	0.00
Rpv	67.34	168.63	122.71	47.18
Rq	7.13	19.38	9.25	6.81
Ra	5.40	11.85	6.66	5.15
Rzl	65.63	164.76	119.87	44.10
Rsk	-0.55	3.51	-1.77	-0.13
Rkm	4.66	18.08	13.81	.3.51

The material Fissurit® FX (VOCO, Germany) presented the highest degree of roughness for a range of 5µm2 compared to other samples. The material with the lowest degree of roughness was PermaFlow™ (Ultradent, USA). There were differences in surface roughness among the sealants but their values were significantly different in favor of PermaFlow™ (Ultradent, USA).

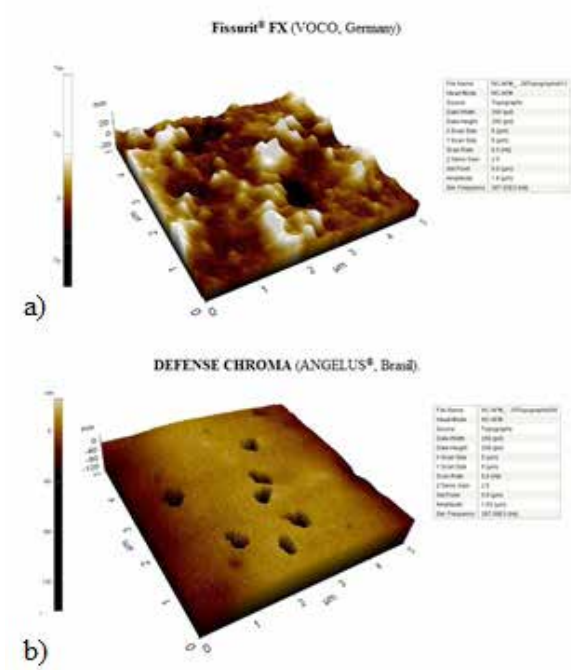
Surface topography characterization

The images obtained by tridimensional topographic analysis (performed by analysis software, XEI-Image Processing and Analysis) make it possible to observe the difference in surface morphology (Fig. 1-a, 1-b,1-c, 1-d).

Morphologically, by AFM, the particle of the F materials appeared to be exposed to a higher degree than the particle of the P. Selected AFM images of the surfaces of materials are presented in (Fig. 1-a, 1-b,1-c, 1-d). AFM images show a relatively smooth natural surface at D material (Figure 1-b). Increased surface irregularities are observed in the AFM images of other materials (Fig. 1-a and 1-c)).

DISCUSSIONS

Low viscosity for sealing material allows a good moistening of the tooth surface. The four resin composite material had a different viscosity depending on the percentage of inorganic fillers. Thus differences in the surface roughness might be due to the different degree of filling.



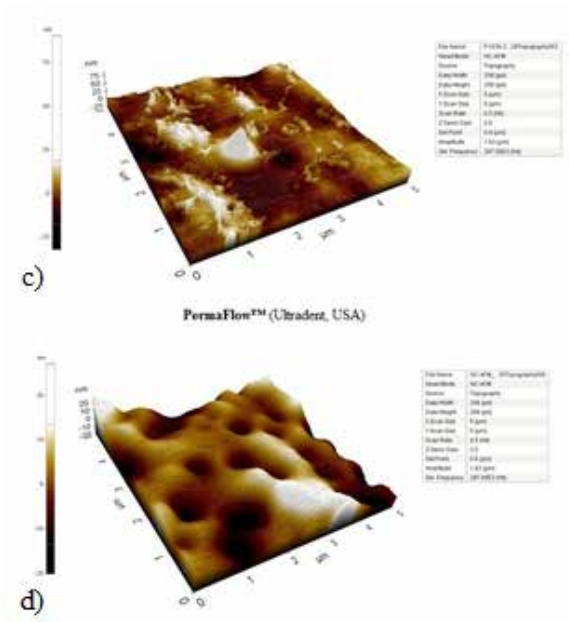


Figure 1. AFM - 5 µm2 image 3D (XEI-Image Processing and Analysis software) for a) Fissurit® FX (VOCO, Germany), b) DEFENSE CHROMA (ANGELUS®, Brasil), c) WAVE (SDI, Australia), d) PermaFlow™ (Ultradent, USA).

Of the four subjects, Fissurit® FX - without inorganic filler sealer, presented the highest roughness (624,62nm), followed by DEFENSE CHROMA(164,76nm), WAVE (119,87nm) and PermaFlow™- with the lowest surface roughness (44,104nm).

It was observed that the degree of roughness varied in the proportion of inorganic load, the lowest roughness material having the highest percentage of filler - 63% PermaFlow™. This characteristic does not allow accumulation, formation and development for dental plaque. A large percentage of inorganic filler particles will increase the viscosity excessively, and will not penetrate into micro-retentions previously created through etching or laser, which will affect the retention of the material.

AFM analysis of samples obtained by inserting the resinous material between 2 sheets of celluloid and then pressed between two glass plates, allowing perfectly flat and smooth surfaces. In vivo surface sealant remains intact after polymerization, but in the absence of premature contacts, otherwise, it is processed with the finished diamond cutters, then polished with gums and polishing paste. Also, as the surface is subject to mechanical stresses and physical contact with food mastication and the action of various chemicals, which are found in the oral cavity, thus contributing to the degradation of the material. However, these data on surface characteristics in vitro combined with those of microleakage and strength of materials, can guide the clinician in selecting a sealant performance.

CONCLUSIONS

Given the results of the current study, further investigations on the surface roughness of sealants materials are warranted. Within the limitations of this study it was concluded that:

The quality and morphology of the particles of the filler varies from one material to another.The use of roughest surface of resin compositesealants materials, in descending order, was achieved subsequent to the use of sealant with 50% filler, 63% filler, without filler and68% filler.

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