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Investigation of different bleaching conditions on the amount of elutable substances from nano-hybrid composites

Jürgen Durner^{a,b,*}, Julia Obermaier^a, Nicoleta Ilie^a

^a Department of Operative/Restorative Dentistry, Periodontology and Pedodontics, Ludwig-Maximilians-University of Munich, Goethestr. 70, 80336 Munich, Germany

^b Walther-Straub-Institute of Pharmacology und Toxicology, Nussbaumstr. 26, 80336 Munich, Germany

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ABSTRACT

Objectives. To analyse the influence of irradiation time, aging before bleaching, and bleaching technique (home bleaching vs. in-office bleaching) on the amount of elutable substances from modern nano-hybrid resin-based composites (RBCs).

Methods. Specimens ($n=5$) of three nano-hybrid RBCs (Venus[®] Diamond, Tetric EvoCeram[®] and Filtek[™] Supreme XTE) were irradiated for 5, 10, 20 and 40 s. The degree of conversion (DC) was measured in real-time with Fourier transform infrared spectroscopy (FTIR). Specimens were bleached either directly after irradiation or after aging (1.5 or 6 month in distilled water at 37 °C) with Opalescence[®] PF15% for 6 h (simulation of home bleaching) or PF35% for 0.5 h (simulation of in-office bleaching) and incubated in ethanol/water (3:1) at 37 °C for 7 d. The eluates were analyzed by gas chromatography/mass spectrometry. Unbleached specimens at the above mentioned irradiation times were used as controls.

Results. Bleaching increases the amount of elutable substances. This amount is generally stronger influenced by aging than by polymerization time or concentration of the gel. 2-Hydroxyethyl methacrylate was found in amounts up to 334.14 (106.91) $\mu\text{mol/l}$ (Tetric EvoCeram[®], irradiation time 5 s; bleaching with 15% CP) as a destruction product. Diethoxydimethylsilane was found in all eluates from bleached specimens, but not in the control groups. This substance may be formed by oxidation of 3-methacryloxypropyltrimethoxysilane, indicating that the bond between inorganic filler and organic matrix might be weakened after bleaching.

Significance. Bleaching gels might alter the physical properties of resin-based composites, especially at low irradiation times and fresh placed restorations.

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1. Introduction

During the last decades the desire for white teeth has become more and more important all around the world. Besides

discoloration of the tooth caused by food, smoking, tea, coffee or red wine, poor oral hygiene, diseases of enamel or dentin, pulpal necrosis, the use of pharmaceuticals like tetracycline or doxycycline, aging and genetic considerations, the main reason for bleaching teeth are of cosmetic matter [1–3].

* Corresponding author at: Walther-Straub-Institute of Pharmacology und Toxicology, Faculty of Medicine, Ludwig-Maximilians-University of Munich, Nussbaumstr. 26, 80336 Munich, Germany. Tel.: +49 89 2180 73801; fax: +49 89 7095 73842.

E-mail address: juergen.durner@med.uni-muenchen.de (J. Durner).

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Hydrogen peroxide (HP) in the form of its precursor, carbamide peroxide (CP), an HP releasing agent, is widely used for tooth whitening (bleaching) both, in professionally and self-administered products [1]. Various bleaching techniques are offered to patients including in-office bleaching, with usually 25–35% HP, and home bleaching, usually with 6.5–22% CP [4,5].

Different studies confirmed that a predictable tooth whitening can be achieved by using a 10% CP gel in a bleaching tray at night (the night-guard vital bleaching technique) [6–9], HP strips [10] or 35% HP with or without light and/or heat activation ('power bleaching') [1,11,12]. It could be shown that HP diffusion through dental tissues and its cytotoxic effects was proportional to the contact time of the bleaching gel with enamel, although shorter bleaching times reduced the results of bleaching [13]. While many studies and case reports focus on the influence of HP/CP on enamel, dentin or pulp tissue, less data is available regarding the reactions of HP with the three-dimensional polymer network of resin-based composites (RBCs). Nevertheless few studies pointed out the need to enlighten patients with dental restorations that changes may occur during whitening, and their bleached restorations may need to be polished or replaced at the end of the treatment [3]. One reason therefore is that the polymer network of RBCs consists of carbon-carbon (C–C)-single or C–C-double bonds, which may react with oxidants like HP [14]. Durner et al. [15] supposed that bleaching with HP could degrade the three-dimensional polymer network of RBCs, leading to an increase in unpolymerized monomers, additives and unspecific oxidative products release, when compared to unbleached controls.

It is common knowledge that elutable substances like monomers, co-monomers, initiators, stabilizers, decomposition-products or contaminants can lead *in vitro* to cytotoxic, teratogenic, estrogenic, mutagenic and/or genotoxic effects as well as to allergic reactions *in vivo*, like allergic contact dermatitis and asthma in humans [16–21]. The release of these substances are shown in a lot of studies [22,23].

The oxidative interaction with the polymer network of RBCs during bleaching influences not only the amount of elutable substances, but also the physical properties of the filling. It could be proved that the color of RBCs changes significantly after bleaching, however with no clinical relevance [24]. Another research group showed that the color-stability depends on the volume of resin matrices and filler type [25]. A significant decrease in surface hardness (Knoop hardness test) of bleached RBCs, on superficial surfaces and in deeper layers was reported as well [26,27]. It was also demonstrated that treating RBCs with HP/CP increased their surface roughness, however without clinical relevance [28,29].

Material properties as well as the amount of elutable substances can be influenced by several parameters like the irradiation time, the degree of conversion (DC), the storing condition of the specimens or the concentration of CP. Therefore the aim of our study was to investigate if 5, 10, 20 or 40 s polymerized specimens (while measuring the DC at the same time) of three different modern nano-hybrid RBCs aged under three different storing conditions before bleaching (no storage; 1.5 months; 6 months) and bleached by using two different bleaching techniques (0.5 h with 35% CP-gel simulating in-office bleaching and 6 h with 15% CP-gel simulating

home bleaching) release higher amount of eluted substances compared to unbleached samples, polymerized under same conditions (control group).

The null hypothesis tested was that the amount of elutable substances would not be influenced by the parameters irradiation time and DC, nor by the parameters bleaching technique and storing time of the RBCs samples before bleaching. Furthermore the effect strength of the analyzed parameter on the amount of elutable substances was calculated.

2. Materials and methods

All solvents and reagent products were obtained from Merck, Darmstadt, Germany and of highest purity available.

2.1. Preparation of samples and irradiation time

From the light-curable nano-hybrid dental restorative materials Tetric EvoCeram[®], Venus[®] Diamond, and Filtek[™] Supreme XTE (Table 1) specimens of approximately 30 mg (thickness of 2 mm, diameter of 3 mm; color A3, with a resulting surface of 32.99 mm² volume of the cylinder of 14.13 mm³) were prepared under photolaboratory conditions. The specimens ($n = 5$) were covered with plastic matrix strips (Frasaco, Tettngang, Germany) and irradiated for 5, 10, 20 and 40 s by using an LED light source (Freelight2, 3M-ESPE, 1241 mW/cm²). The curing unit was directly applied on sample's surface. The irradiance of the curing unit was measured by means of a calibrated fiber optic spectrally resolving radiometer equipped with an integrating sphere (S2000, Ocean Optics, USA).

2.2. Degree of conversion (DC)

The measurements of the DC ($n = 5$) were made in real time with an FTIR-Spectrometer with an attenuated total reflectance (ATR) accessory (Nexus, Thermo Nicolet, Madison, USA). Therefore, the un-polymerized composite paste was put directly on the diamond ATR crystal in a mold 2 mm high and with a diameter of 3 mm. The mold was filled in one increment and the curing unit was applied directly on the sample surface. The FTIR spectra were recorded in real time for 5 min on the lower surface of the samples with irradiation for 5, 10, 20 and 40 s. The diameter of the measured surface was 800 μm , the wave number range of the spectrum was 4000–650 cm^{-1} and the FTIR spectra were recorded with two scans per second at a resolution of 8 cm^{-1} . The FTIR-spectrometry based on an idea from A. Michelson. Two rays were brought to interference. By variation of the wavelength of the rays, the resulting intensity of the interference was shown in an interferogram. The Fourier Transformation, a mathematical algorithm, is making an IR spectrum from the interferogram. Different chemical groups show a special sign (transmission) at a defined wavelength.

To determine the percentage of the remained unreacted double bonds, the DC was assessed as the variation of the absorbance intensities peak height ratio of the methacrylate carbon double bond (peak at 1634 cm^{-1}) and those of an internal standard (aromatic carbon double bond; peak at 1608 cm^{-1}) during polymerization, in

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