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# Ultra-high-molecular-weight polyethylene fiber reinforced dental composites: Effect of fiber surface treatment on mechanical properties of the composites

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## ABSTRACT

**Objectives.** Poor interfacial adhesion between the fibers and resin matrix in the ultra high molecular weight polyethylene (UHMWPE) fiber reinforced composites (FRCs) is the main drawback of the composites. This study aims to evaluate the effect of corona and silane surface treatment of the fibers on the mechanical properties of the UHMWPE FRCs.

**Methods.** UHMWPE fibers were exposed to corona discharges for different periods of time (0 s, 5 s, 7 s). The surface characteristics of the UHMWPE fibers were investigated by attenuated total reflectance Fourier transformed infrared spectroscopy (ATR-FTIR), atomic force microscopy (AFM), scanning electron microscopy (SEM) and nanoindentation technique. The flexural strength and flexural modulus of the FRCs made of the treated fibers were determined on 2 mm × 2 mm × 25 mm specimens. The fracture toughness (the critical stress intensity factor,  $K_{IC}$ ) of the composites was also evaluated using a three-point single edge notch beam (SENB) bending technique. Statistical analysis of the data was performed with ANOVA and the Tukey's post-hoc test. The fiber-resin interface and the fracture surface were investigated using SEM.

**Results.** The change in the surface mechanical properties and chemistry of the corona treated UHMWPE fibers were monitored. The fibers exposed to corona for 5 s showed higher surface nanohardness. In the FRCs, the specimens reinforced with 5 s corona treated silanized fibers showed higher mechanical properties (flexural modulus, flexural strength, and fracture toughness), SEM images revealed a better adhesion between the resin and fibers after 5 s fiber corona treatment and silanization.

**Significance.** Corona and silane treatment of UHMWPE fibers provide dental FRCs with improved mechanical properties.

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## 1. Introduction

Development of fiber-reinforced composites (FRCs) have offered dental researchers the possibility of making resin-bonded, mechanically and esthetically good, and metal-free restorations for teeth replacement [1,2]. Variety of dental applications, such as bridges, splints, posts, space-maintainers, orthodontic retainers, denture bases, clasps and connectors, and implant prostheses made of FRCs have successfully been used in dental practice providing acceptable mechanical properties [3,4]. In general, in a FRC, fibers are the main load-carrying members, while the surrounding matrix acts as a load transfer medium and protects the fibers from environmental harms [5]. The mechanical properties of FRCs are dependent upon the mechanical properties of each component, quality of impregnation of fiber with resin, adhesion between the fiber and matrix, quantity of fibers in the matrix resin, the fiber volume fraction, fiber architecture, and the fiber type [6,7]. UHMWPE fibers in comparison with other plastic materials are found to have high impact resistance, toughness, and tensile strength. The fibers are also light weight, corrosion and wear resistant [8,9]. Adhesion of UHMWPE fibers to the matrix resin is, however, difficult because the UHMWPE is non-polar in nature with low surface energy [10]. To provide a good adhesion between the UHMWPE fibers and the polymer matrix, the surface of the fibers has been modified using different methods such as chemical modification [11], chemical grafting [12], corona discharging [13,14], oxygen plasma [15–17], high energy laser [18], UV [19], and gamma irradiation [20]. Improving the adhesion performance of UHMWPE may result in improved dental FRCs. Corona discharge, is a process by which ionized gas species are formed around an active high potential electrode due to the electrical current [21]. In corona treatments of polymers the ionized species transfer their charge to the polymer surface resulting in increase in surface energy by introducing polar groups on the surface. The corona discharge may also change the surface roughness [22]. The process can impart hydrophilicity to polymer surfaces which may improve the surface affinity and sticking strength with some hydrophilic polymers [13]. It can also improve the bonding characteristics of materials by raising the surface energy, wettability, oxygen to carbon ratio, number of functional polar groups on the surface, and specific surface area leading to enhanced adhesive capabilities, increase in the delamination force [23,24] and improvement in the fracture toughness and flexural properties [25]. Silane treatment, on the other side, is a well-known method for improvement of surface wettability and interfacial adhesion between matrix resin and OH-covered substrates [26–28].

Therefore, the combination of these two surface modification methods may result in FRCs with improved properties. This study, consequently, designed to test the hypothesis that the double surface treatment of UHMWPE fibers improves the mechanical properties of an experimental dental FRC containing the fibers as reinforcing phase and Bis-GMA/TEGDMA as matrix.

## 2. Materials and methods

### 2.1. Materials

Materials used in this study are presented in [Table 1](#).

### 2.2. Methods

#### 2.2.1. Surface corona treatments of UHMWPE fibers

UHMWPE fibers were treated for 5 s and 7 s under atmospheric pressure air corona discharge using SpotTEC corona power Generator (Tantec, Lunderskov, Denmark, 6.5 kV/25 kHz and 550 W). The distance between electrode and UHMWPE fiber surface was 5 cm.

#### 2.2.2. AFM and nanoindentation analysis

AFM imaging analysis was performed (SPM Dualscope/Rasterscope C26, DME, Copenhagen/Herlev, Denmark) to study the surface topography and roughness of the fibers before and after corona treatment. Hardness ( $H_i$ ) of the fibers was determined using nanoindentation technique (CSM, Peseux, Switzerland, indenter type: Berkovich, indenter material: diamond, max depth: 500 nm, loading and unloading rates:  $1000 \text{ nm min}^{-1}$ ). The elastic modulus of the specimen in depth-sensing indentation techniques, used in nanoindentation test, is determined from the slope of the unloading of the load-displacement response,  $E_i$ . Vickers hardness was also calculated from the  $H_i$ .

#### 2.2.3. Fourier-transform infrared spectrometer (FT-IR)

A FTIR spectrometer (EQUINOX55, Specac golden gate, ZnSe prism, Bruker, Germany) was used in order to identify functional groups formed on the fibers after corona and silane treatments. The spectra were collected in the wavenumber range of  $4000\text{--}400 \text{ cm}^{-1}$ , using attenuated total reflectance technique (ATR-FTIR). In this technique the infrared beam is directed into a crystal of relatively high refractive index (ZnSe) which is in contact with the fibers. Multiple reflections of the beam in the crystal collects the spectrum of the intimate fibers.

#### 2.2.4. Silanization of UHMWPE fibers

The chemical surface modification of UHMWPE fibers was carried out using a 2 (wt/vol)%  $\gamma$ -MPS in ethanol/distilled-water (70/30 wt/wt, pH adjusted at 3.8 using acetic acid). Having the silane pre-hydrolyzed for 1 h, the fibers were immersed in the solution and left for 1 week at room temperature to dry.

#### 2.2.5. Composite preparation and mechanical measurements

The composites were classified in 5 groups ([Table 2](#)). Group 1, group 2 and group 3 were prepared to evaluate the effect of corona exposure time. All these three groups were made of 10 bundles for flexural test and 20 bundles for fracture toughness test. As the volume of the fracture toughness test mold was two times of the flexural test, twice fibers were inserted in the fracture toughness molds to keep the fiber content constant

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