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Are linear elastic material properties relevant predictors of the cyclic fatigue resistance of dental resin composites?

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ABSTRACT

Objective. The aim of this study was to measure the linear elastic material properties of direct dental resin composites and correlate them with their fatigue strength under cyclic loading. **Methods.** Bar specimens of twelve resin composites were produced according to ISO 4049 and tested for elastic modulus (Emod) in 3-point bending ($n=10$), flexural strength (FS) ($n=15$) and single-edge-notch-beam fracture toughness (FT) ($n=15$), both in 4-point bending. Using the same specimen geometry, the flexural fatigue strength (FFS) was determined using the staircase approach after 10^4 cycles at 0.5 Hz in 4-point bending ($n=25$). The observation of the fracture surface and fracture profiles was conducted using a scanning electron microscope in order to evaluate the respective fracture mechanisms according to the two different loading conditions.

Results. Materials were ranked differently according to the tested parameters. Only weak correlations were found between any of the initial properties and FFS or strength loss. The best correlation to FFS was found to be the Emod ($r^2=0.679$), although only slightly. Crack path in both loading conditions was mainly interparticle, with the crack propagating mainly within the matrix phase for fatigued specimens and eventually through the filler/matrix interface for statically loaded specimens. Fracture of large particles or prepolymerized fillers was only observed in specimens of FS and FT. Initial properties were better associated with microstructural features, whereas the fatigue resistance showed to be more dependent on aspects relating to the matrix phase.

Significance. Our results show that linear elastic properties such as elastic modulus, flexural strength and fracture toughness are not good descriptors of the fatigue resistance of dental resin composite under cyclic bending, and may therefore have limited clinical relevance.

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1. Introduction

Predictions of material performance based on mechanical properties are anchored in the theory of correlation, a statistical tool that describes the degree to which two sets of data

show any association. Depending on the data distribution, high correlation coefficients may characterize a high functional dependency, express susceptibility or causality between independent variables. Mechanical properties such as fracture toughness, surface hardness and elastic modulus were found to have a defined relationship to the wear resistance of

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resin-based composites [1]. This theoretically enables the prediction of clinical wear rates of composites based on experimentally obtained parameters. By increasing the predicability of *in vitro* data, costly and time-consuming clinical studies for material performance loose urgency and material development processes become more dynamic.

Traditionally, the characterization of the mechanical behavior of resin composites starts with the measurement of intrinsic properties under a condition of uniform increase in load (static loading). The most relevant of these properties concern the stiffness (elastic modulus) and those involving the resistance to fracture (i.e., strength and fracture toughness). For the measurement of both strength and toughness a high amount of energy is applied to cause fast fracture, resulting in parameters that define the state of a material in a point in time. Under these conditions, material behaviors liable to dynamic or cyclic loading are poorly described. That is especially true for materials that present a high susceptibility to fatigue, where the mechanical properties are ever-changing by influence of environmental and stress-related phenomena. For fatigue-resistant materials, measurements of strength and fracture toughness serve to characterize their mechanical state at different periods even after long loading challenges.

Relationships between clinical performance and mechanical parameters in dental resin composites are complex, since both derive from scenarios of very different loading conditions. Under function resin composite restorations are loaded periodically, in which the dynamic mechanical behavior of the polymer matrix and water-/stress-degradation processes play a significant role. The viscoelastic effects occurring in the resin during *in vivo* cyclic fatigue are absent in specimens tested under high strain rates, where the release of high elastic energy levels results in brittle-like fracture patterns [2]. Also *in vitro*, factors related to test design and specimen geometry add to the apparent discrepancy to *in vivo* data [3–5]. Strength measurements for example, naturally amplify the role of surface flaws on fracture initiation, whereas clinical wear tends to reshape the surface and introduce new flaw populations to the loaded restoration. Such complexities are in part disregarded in the literature, and although no concrete correlations between mechanical properties measured under static loading and the fatigue resistance of resin composites have been ever established [6], inferences on the latter based on the former have become commonplace. For other restoratives such as glass-ceramics, the *in vivo* clinical performance has shown to be better characterized by *in vitro* fatigue parameters that consider time-dependent processes involved in their failure [7].

Conversely to the measurement of strength and fracture toughness, where failure results from the unstable propagation of a crack of critical length, the mechanical degradation, or “fatigue”, of a material involves the growth of subcritical defects at subcritical loads. The fatigue susceptibility can be assessed using parameters defined by fracture mechanics, such as the stress intensity factor, by measuring its development during crack extension at consecutive cycle intervals. Outside the fracture mechanics realm, phenomenological approaches base their evaluation on the residual strength after fatigue loading, where endurance ranges can be extracted and compared to clinically relevant stress amplitudes.

In the present study we selected for testing twelve commercial direct resin composites that represent the current state of the art regarding matrix composition and filler technology. These materials were tested for inherent mechanical properties (e.g., elastic modulus, fracture toughness and flexural strength) and fatigue resistance after 10^4 cycles using the staircase approach. The tested null-hypotheses were that (i) there is no correlation between the inherent properties and the fatigue resistance or strength loss (in %) in resin composites and (ii) there is no correlation between elastic modulus, fracture toughness and strength in these materials.

2. Materials and methods

2.1. Materials

Twelve currently commercially available resin composite materials for direct restorations were selected for testing. In Table 1 details about their filler content, filler configuration and matrix composition can be found. This selection of composites included those with novel monomer formulations (Filtek Silorane, Venus Diamond and Kalore), those containing prepolymerized fillers (Kalore, Tetric EvoCeram, Miris 2, Clearfil Majesty Esthetic) and a mixture of glass particles and long glass fibers for the use as a base material (Xenius Base). Apart from the nanofilled Filtek Supreme XT, most are nanohybrid materials, with an inorganic filler content ranging from 54 vol% (Tetric EvoCeram) to highly filled systems, such as Grandio SO (73 vol%) and Clearfil Majesty Posterior (82 vol%).

2.2. Specimen preparation

Sixty-five bending bars (2 mm × 2 mm × 25 mm) were produced for each material using a tungsten carbide/glass mold under calibrated conditions of 23 °C and 50% humidity. The materials were inserted in the mold in one previously weighted increment, and light-cured with five overlapping spots of $\varnothing = 8$ mm on both upper and lower sides with a commercial light-curing unit (Elipar® Trilight, 3M ESPE, Germany) with an output intensity of 800 mW/cm² for the time recommended by the manufacturers (20 s per spot for all materials for the curing intensity range used). For Xenius Base, the material was inserted in the mold out of the cannula so that the parallel glass fibers were oriented in the long axis of the specimen. The specimen fabrication procedures followed the manufacturers' recommendations and ISO 4049 standard [8]. After removal from the mold, the specimens were ground in the lower side with silicon carbide paper of down to 4000 grit to remove surface flaws that could influence strength values [9]. The specimens were stored in distilled water at 37 °C for 24 h before the fracture toughness and elastic modulus testing and for 14 days before testing the initial flexural strength and the flexural fatigue resistance.

2.3. Elastic modulus (*E_{mod}*) testing

To determine the elastic modulus (*E_{mod}*) ($n=10$), the stress–strain curves were obtained between 10 and 40 MPa for each specimen by loading them in a 3-point bending test

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