Physical characterization of unfilled and nanofilled dental resins: Static versus dynamic mechanical properties

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A B S T R A C T

Objective. The aim of this study was to evaluate the mechanical properties of dental resins and dental nanocomposites by means of 3-point bending, 4-point bending and piston-on-three ball biaxial static tests and also to investigate their dynamic mechanical properties. The obtained results from the static 3-point bending test also were compared with the corresponding mode in DMTA.

Methods. The hydrophilic surface of the inorganic OX-50 particles was treated with γ-MPS and the resulting silanized OX-50 was characterized using FTIR and TGA. The test specimens with a specified shape and dimensions for each type of flexural static and dynamic tests were prepared for Bis-GMA/TEGDMA (70/30 wt%/wt%) and the corresponding nanocomposite containing 50 wt% of silanized OX-50. The specimens were stored in distilled water at 37 °C for 24 h prior to the mechanical tests and then they were subjected to different types of static mode and also to dynamic mode of flexural test. The effect of test speed, type of the test and the presence of silanized nano-particles were investigated for specimens under static flexural tests. The results were then statistically analyzed and compared using one-way ANOVA and the Tukey's post hoc test (significance level = 0.05). Fractured surfaces were observed by means of scanning electron microscopy. Finite element analyses were also performed to compare the static tests. On the other hand, the effect of frequency, temperature and the presence of silanized nano particles on the viscoelastic properties were investigated for dynamic mechanical tests.

Results. The grafting of γ-MPS onto the OX-50 nano particles was confirmed. The results of 3-point bending and 4-point bending uniaxial and biaxial flexural tests of resin and composite showed the highest strength values for biaxial test specimens and the lowest strength value for 4-point bending specimens in accordance with finite element analysis results. Also, an increasing trend was observed for flexural strength of all resin samples with increasing the test speed up a critical speed which decreased beyond it. The nanocomposite specimens showed higher modulus and lower tan δ than the unfilled resin. The storage modulus measured in the dynamic tests approached the static elastic modulus values with decreasing the frequency.

Significance. Different static and dynamic test methods are used to evaluate the mechanical properties of dental resins and composites. This study provides an insight into the tests and presents more details on the effect of test conditions.

1.   Introduction

The development of direct restorative materials based on synthetic polymers has been initiated due to their advantages over the traditional metallic/nonmetallic restorations. The materials could overcome the defects of the silicate materials, namely, erosion, brittleness, acidity and the moisture sensitivity and are readily cured at mouth temperature with excellent esthetic properties [1–3]. The composite materials are commonly based on methacrylate monomers. The direct restorative materials, especially those that are used for posterior restorations, are filled with a high percentage of silica/silicate micro/nano-particle reinforcing fillers in order to improve the mechanical properties and withstand the forces induced during function [1–6].

Strength is an important mechanical property which has a significant effect on the successful function of the restorative materials. It is dependent upon various parameters such as the materials microstructure, composition, testing method, test conditions and failure mechanisms [5,7]. Measurement the strength of dental materials is often performed through flexural tests. The International Organization for Standards (ISO) introduces the use of uniaxial three-point bending as a valid test for strength measurement of the materials [8]. The other test methods for evaluating the mechanical properties of dental materials such as diametral tensile strength, 4-point bending and biaxial tests, have also been reported in the literature and the effect of different factors such as test method, the material composition and photopolymerization procedures have been investigated [5,9–11]. The mechanical properties of dental materials have mostly been evaluated in low strain rates by static flexural test methods. As the materials experience a wide range of loading conditions during mastication cycle or unexpected high speed impacts during various activities, it is worth investigating the mechanical properties of dental restoratives at different strain rates. Dynamic-mechanical thermal analysis (DMTA) is a suitable method to evaluate mechanical properties at high strain rates that is enables the effect of frequency and temperature to be studied [2,12–14].

The aim of the present work was to study the flexural properties of dental resins and dental composites containing silanized silica nano-particles, more precisely, by means of 3-point bending, 4-point bending and piston-on-three ball biaxial tests at different test speeds. In this part, the effect of the test method and the test speed on the static flexural properties were evaluated. Finite element analysis was performed to estimate the effect of the test method on the flexural strength. The dynamic-mechanical thermal analysis in the 3-point bending mode was also performed and the effect of frequency on the viscoelastic properties such as storage modulus (E'), loss tangent ($\tan \delta$), and different transitions was

investigated. Furthermore, the effect of the incorporation of silica nano-particles to the resin matrix on the static and dynamic properties was evaluated.

2.   Experimental

2.1.   Materials

2,2-Bis-[4-(methacryloxypropoxy)-phenyl]-propane (Bis-GMA), triethyleneglycoldimethacrylate (TEGDMA) and Aerosil OX-50 amorphous fumed silica were supplied by Evonik (Germany). The average particle diameter of Aerosil OX-50 (nanosized silica) was 40 nm with a specific surface area of $50\,m^2\,g^{-1}$. Camphorquinone (CQ) and 2-(dimethylamino)ethyl methacrylate (DMAEMA) were obtained from Sigma–Aldrich Chemical Co. (Germany). 3-Methacryloxypropyltrimethoxy-silane (γ-MPS, Dynasylan® MEMO) was kindly provided by Evonik (Germany). Dibutylamine was purchased from Merck (Hohenbrunn, Germany). Cyclohexane was supplied by Daejung Chemicals & Metals Co., Ltd. (South Korea).

2.2.   Methods

2.2.1.   Surface modification of nano-silica particles with γ-MPS

In order to enhance the interfacial adhesion between resin matrix and inorganic particles, the hydrophilic surface of the inorganic particles was treated with γ-MPS. The silica powder 25.0 g, γ-MPS 2.5 g (the amount of γ-MPS was kept constant at 10% (wt/wt) with respect to silica), and cyclohexane 250 ml stirred at room temperature for 24 h. Then, the mixture was sonicated with a high-power probe-type ultrasonicator (SONOPULS HD 2200, BANDELIN electronic GmbH & Co. KG, Germany) for 5 min in order to improve the dispersion of nano particles in the mixture and to reduce their agglomeration. Afterwards, dibutylamine 2.5 g was added to the mixture. The mixture was heated in a rotary evaporator (Rotavapor, R-3000, Heidolph, Germany) at 60 °C for 3 h and then for 1 h at 90 °C in order to remove the solvent. The suspension was then centrifuged at 7500 rpm (Sigma, 2-16, Germany) repeatedly with fresh cyclohexane for three times to remove catalyst and unreacted silane coupling agent, followed by heating the washed powder at 80 °C in a vacuum oven for 24 h.

2.2.2.   Characterization of the silanized silica nano-particles

The pristine and silanized OX-50 were identified by FTIR spectroscopy (EQUINOX 55, Bruker, Germany, resolution of $4\,cm^{-1}$ and 32 scans) in the range of 4000–400 cm^{-1} using KBr disk technique. Thermogravimetric analysis of the pristine and silanized OX-50 was also performed (TGA-1500, Polymer