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Dynamic mechanical thermal properties of the dental light-cured nanohybrid composite Kalore, GC: Effect of various food/oral simulating liquids

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ABSTRACT

Objective. The aim of this work is the study of the dynamic mechanical thermal properties (viscoelastic properties) of a current dental commercial light-cured nanohybrid resin composite, Kalore, GC (GC Corporation, Tokyo, Japan) along with the study of the effect of some food/oral simulating liquids (FSLs) on these properties.

Methods. Dynamic mechanical thermal analysis (DMTA) tests were performed on a Diamond Dynamic Mechanical Thermal Analyzer in bending mode. A frequency of 1Hz and a temperature range of 25–185 °C were applied, while the heating rate of 2 °C/min was selected to cover mouth temperature and the material's likely T_g . The properties were determined after storage in air, distilled water, heptane, ethanol/water solution (75% v/v) or absolute ethanol at 37 °C for up to 1 h, 1, 7 or 30 days.

Results. Storage modulus, loss modulus and tangent delta (tan δ) were plotted against temperature. The glass transition temperatures are taken from the peak of the tangent tan δ versus temperature curves. Moreover, some factors indicating the heterogeneity of the polymer matrix, such as the width (Δ T) at the half of tan δ peak and the " ζ " parameter were determined. All samples analyzed after storage for 1 h or 1 day in the aging media showed two T_g values. All samples analyzed after storage for 7 or 30 days in the ageing media showed a unique T_g value.

Significance. Storage of Kalore GC in dry air, water or heptane at 37 °C for 7 days caused postcuring reactions. Storage in air or water for 30 days did not seem to cause further effects. Storage in heptane for 30 days may cause plasticization and probably some degradation of the filler–silane bond and polymer matrix. Storage in ethanol/water solution (75% v/v) or ethanol for 7 days seems to cause post-curing reactions and degradation reactions of the matrix–filler bonds. Storage in ethanol for 30 days caused a strong change of the sample morphology and the DMTA results were not reliable.

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1. Introduction

The composition of resin-based composite materials has evolved significantly since they were firstly introduced in Dentistry in the mid-1960s. Current changes are more focused on the polymeric matrix of the material, mainly to develop systems with reduced polymerisation shrinkage and, more importantly, reduced polymerisation shrinkage stress or to make them self-adhesive to tooth structure as well [1]. Recently, a low-shrinkage resin composite has developed and launched in the market (Kalore, GC, Tokyo, Japan). Kalore GC is a visible-light curable radiopaque nanohybrid resin composite. Its unique composition enables the creation of anterior and posterior direct composite restorations (i.e. Class I, II, II, IV and V cavities) with high polish, high wear resistance, low polymerisation shrinkage (stress) and durability. It can be also used for direct restorations for wedge-shaped defects and root surface cavities or direct restorations for veneers and diastema closure [2].

Dental resin-composites are characterized generally as viscoelastic materials, since they behave neither as perfectly elastic solids, nor as completely viscous materials, due to the presence of the resin matrix (polymer). The degree to which a material behaves either viscously or elastically depends on environmental temperature, vibrations' frequency, dynamic strain rate, time effects (creep, relaxation), aging and other irreversible effects [3]. Since denture-based polymers display viscous and elastic properties, both storage (E') and loss moduli (E") are necessary to completely describe deformation response [4]. Storage modulus, which is measured by the slope of the elastic region of the stress-strain diagram, describes the relative stiffness or rigidity of the material [5]. Dental composites with low storage modulus will more readily deform elastically under functional stresses, which may result in catastrophic fracture of the surrounding tooth structure. Ideally, the storage modulus should fit to those of the dental tissues, which are supposed to be replaced [6,7].

Dynamic mechanical tests, such as Dynamic Mechanical Thermal Analysis (DMTA) are particularly well-suited for viscoelastic materials, since they determine both the storage and the viscous response of the materials [3]. Also, dynamic tests mimic better the cyclic masticatory loading to which dental composites are clinically subjected [6,8]. This might be extremely valuable to predict the clinical performance of bio-materials when working under the cyclic oscillations generated by the human body's physiological movements [6]. In our previous works we have studied the dynamic thermomechanical properties of several commercial dental light-cured resin-based composites [9–12].

In oral environment, it can be stated that saliva, food components, beverages and interactions among these materials can degrade and age dental restorations. The food simulating liquids used for the aging of the materials in this study were chosen according to FDA guidelines. Water simulates the oral environment provided by saliva; heptane simulates vegetable oils, butter and fatty meats whereas the aqueous ethanol solution and ethanol simulate beverages, including alcoholic drinks, vegetables, fruits and candy [13]. This work is concerned with the study of viscoelastic properties and parameters, such as the storage modulus (E'), the loss modulus (E''), the tangent delta (tan δ) and the glass transition temperature (Tg) of the Kalore, GC using DMTA. Moreover, some factors indicating the heterogeneity of the polymer matrix, such as the width (Δ T) at the half of tan δ peak, the " ζ " parameter were determined. These properties were determined after storage of the composite in air, water, heptane, ethanol/water solution (75% v/v) or ethanol for several time intervals. It was found interesting to examine the viscoelastic properties of this composite, because of its unique composition of the organic resin matrix, filler and interphase between them.

2. Materials and methods

2.1. Material composition

The light-cured nanohybrid composite studied was the Kalore, GC (GC Corporation, Tokyo, Japan), shade A₃, Lot. 1107201.

2.1.1. Organic resin matrix

Kalore consists of an organic matrix, fillers, photoinitiation system and pigments. The matrix contains a mixture of dimethacrylates [2,14]. It contains a new dimethacrylate monomer the DX-511, (CAS: 1026782-73-9, 5-10% by weight) which is recently developed by DuPont (DuPont monomer) and is based on urethane dimethacrylate chemistry (Fig. 1). This monomer has a long rigid molecular core and flexible arms in the structure. The long rigid core prevents monomer's deformation and reduces polymerisation shrinkage. On the other hand, if the molecular core is flexible, the monomer may fold and will occupy less space, causing a loss in dimension. The molecular weight of this monomer is 895 which is twice that of Bis-GMA or UDMA. Generally, the short chain monomers with lower molecular weight have the greatest polymerisation shrinkage and inferior physical characteristics than the long chain monomers. A high molecular weight monomer reduces polymerisation shrinkage because it contains only a small number of double bonds C=C, which is a factor in polymerisation shrinkage. However, if the monomer chain becomes too long, then reactivity decreases. To overcome this challenge, flexible arms were created on the new DuPont monomer, thus increasing the potential for reactivity. The manufacturer has reported volumetric shrinkage values of 1.72%, claiming that the shrinkage stress values are the lowest of any composite resin system [15]. The matrix of Kalore GC also contains Urethane Dimethacrylate (UDMA) (CAS: 72869-86-4, 5-10% by weight) and Bisphenol A polyethoxymethacrylate (CAS: 41637-38-1, 1-5% by weight). The latter is also called Bisphenol A ethoxylate dimethacrylate and its form has 2-4 units of ethoxylation, sometimes referred as Bis-EMA.

2.1.2. Filler

At the core of the Kalore GC filler system the newly developed high-density radiopaque *prepolymerised* fillers exist (30–35% by weight) [2]. These prepolymerised fillers (average: $17 \mu m$) contain 60 wt% of 400 nm modified strontium glass and

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