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Contact angle and surface free energy of experimental resin-based dental restorative materials after chewing simulation

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ABSTRACT

Objective. To investigate contact angle and surface free energy of experimental dental resin composites containing novel delivery systems of polymeric hollow beads and low-surface tension agents after chewing simulation test.

Methods. A delivery system of novel polymeric hollow beads differently loaded with two low-surface tension agents was used in different amounts to modify commonly formulated experimental dental resin composites. The non-modified resin was used as standard. Surface roughness R_a , contact angle θ , total surface free energy γ_s , its apolar γ_s^{LW} , polar γ_s^{AB} , Lewis acid γ_s^+ and base γ_s^- terms were determined and the results prior to and after chewing simulation test were compared. Significance was $p < 0.05$.

Results. After chewing simulation R_a increased, θ decreased, R_a increased for two test materials and γ_s decreased or remained constant for the standard or the test materials after chewing simulation. R_a of one test material was higher than of the standard, θ and γ_s of the test materials remained lower than of the standard and, indicating their highly hydrophobic character ($\theta \approx 60\text{--}75^\circ$, $\gamma_s \approx 30 \text{ mJ m}^{-2}$). γ_s^{LW} , and γ_s^- of the test materials were lower than of the standard. Some of the test materials had lower γ_s^{AB} and γ_s^+ than of the standard.

Significance. Delivery systems based on novel polymeric hollow beads highly loaded with low-surface tension agents were found to significantly increase contact angle and thus to reduce surface free energy of experimental dental resin composites prior to and after chewing simulation test.

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1. Introduction

Contact angle θ and surface free energy (SFE) are discussed to influence plaque formation on modern dental filling composites [1–5]. Since there is a positive correlation between SFE and

early plaque formation [5,6], dental resin composites differing in contact angle θ were broadly investigated and bacterial adherence of materials with high θ and low SFE was found to be significantly less [2,7–11]. But it was also reported that no correlation occurred between streptococcal adhesion and

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substratum surface roughness or SFE [12–14] and it was realized that the influence of SFE on bacterial adhesion decreased significantly after saliva coating [14,15].

The present work is related to earlier investigations reporting an entirely new approach to obtain resin materials with low SFE but acceptable physical properties [16] which also proved diminished bacterial adherence [10]. Both papers reported about a new delivery system of polymer hollow beads (Poly-Pore, AMCOL Health & Beauty Solutions, Arlington Heights, IL, USA) as carrier material, highly loaded with two different low-surface tension agents (Tego Protect 5000, Evonik Tego Chemie GmbH Essen, Germany and Dimethicone, Dow Corning Corp., Midland, MI, USA) which were added in small quantities of 5–6 wt.% to experimental dental resin composites. It was found that due to abrasion processes, simulated by polishing, the carrier hollow beads were destroyed and released the low-surface tension agents which flushed the surface and thus reduced the resin's SFE [16]. Now the effect of the exposition of the low-surface tension agents on SFE was re-investigated under more clinically relevant conditions with a chewing simulation device. To simplify the comparison with the previous papers [10,16] the selected experimental resins were coded accordingly. The null hypothesis was that the SFE of the delivery system containing material did not differ from the standard after performing the chewing simulation test.

2. Materials and methods

2.1. Test material preparation (analog [16])

Four experimental light-curing resin-based restorative materials (A, B, C and E) were prepared (Tables 1 and 2) using a laboratory vacuum planet kneader (Herbst Maschinenfabrik GmbH, Buxtehude, Germany). Material ST, representing a common formulation for dental resin composites, was used as the standard. The preparation process of all test materials and ST was done under vacuum at 50 °C. Preparation of the test materials: Poly-Pore was loaded with two different active agents (Tego Protect 5000, Evonik Tego Chemie GmbH or Dimethicone, Dow Corning Corp.) in varying quantities of the agents resulting in four different delivery systems (Table 2). The delivery systems of materials A and B contained Tego Protect 5000, and materials C and E contained Dimethicone as the active agent. ST was modified by replacing 5% or 6% of the glass filler with the respective delivery system. At first the monomers were mixed, next all other ingredients but the glass filler and the Poly-Pore delivery systems were added and dissolved in the monomer mix. Then the respective Poly-Pore delivery system and finally the glass filler were homogeneously dispersed in the mixture.

2.2. Preparation of the delivery systems (analog [10,16])

To obtain the four delivery systems (Tables 1 and 2) 80 g or 85.7 g, respectively, Dimethicone or Tego Protect 5000 were dissolved in 400 g butanone (Lot 244238, Brenntag GmbH, Mülheim, Germany), then 20 g or 14.3 g, respectively, Poly-Pore hollow beads were added. The mixture was stirred

for approximately 10 minutes to optimally wet the sorption particles. The mixture was slightly warmed to evaporate the solvent, and stirring was stopped when it became too stiff. Then it was put in a drying closet at 50 °C until a constant weight was obtained (generally 24 h). After this treatment, the loaded sorption materials, representing the delivery systems, appeared totally dry and powdery.

2.3. Specimen preparation and chewing simulation

Ten disks (thickness: 1 ± 0.1 mm, diameter: 10 ± 0.1 mm) of each material were made and cured for 40 s on each side (Spectrum 800, Dentsply deTrey GmbH, Constance, Germany). The output of the curing device was routinely checked (bluephase meter, Ivoclar Vivadent AG, Schaan, Liechtenstein). Irradiances of 931 ± 90 mW cm⁻² were measured, and no significant decrease in the output was observed. The cured specimens were stored for 14 days in water at 37 °C and then one side was wet-polished (sterile water) with fine and superfine polishing discs (Super-Snap mini, Shofu Inc., Kyoto, Japan) for 1 min, with a grinding pressure of 40–50 g and 10,000 rpm (Endo-Mate TC, Nakanishi Inc., Tochigi, Japan). The degree of polymerization was not measured but the high irradiance of the light curing device ensures an optimal polymerization. Next the specimens were fixed with the polished sides up in the mounts of a chewing simulator (CS-4, SD Mechatronik GmbH, Feldkirchen-Westerham, Germany) with a MMA/PMMA resin (Technovit 4000, Heraeus Kulzer GmbH, Hanau, Germany). They were stressed for 650 cycles in water at 37 °C to simulate a 24 h chewing period according to the literature, which correlates the number of cycles to a realistic period of time [17,18]. The 24 h were chosen to test the daily availability of the active agents. The test specimens were sliding under contact to a spherical antagonist (aluminum oxide, 5 mm diameter, Quick-Ohm Küpper & Co. GmbH, Wuppertal, Germany) for a linear distance of 8 mm (speed: 40 mm s⁻¹) and a vertical load of 50 N resulting in a contact area of 0.6 mm × 8 mm and an average wear of 5.4 μm (± 2.5 μm). This was repeated five times resulting in a worn area of 3 mm × 8 mm = 24 mm² to obtain sufficient surface to place a drop.

2.4. Contact angle and surface free energy (analog [10,16])

Contact angles were determined with the sessile drop method (Phoenix-Alpha contact angle goniometer, Surface Electro Optics – SEO – Corporation, Suwon-City, Korea). Immediately after placing the drop on the specimen's surface ten pictures of each drop were photographed (magnification: 25-folds) in one second (CCD-camera: FireDragon, Toshiba-Teli Corporation, Tokyo, Japan) and analyzed (Image XP-software, Version 6.0 FW 012108, SEO Corporation). The drops were formed by a standardized dosing tube and, therefore, had always the same volume. Contact angle θ was measured one time on each of the ten disks. SFE was calculated from the θ measured after water storage. Apolar Lifshitz-van der Waals γ_L^{LW} , polar Lewis acid γ_L^+ and Lewis base γ_L^- terms of the test liquids were taken from the literature [19] and the specimens' total SFE γ_S , their apolar term γ_S^{LW} , polar term γ_S^{AB} , acid term γ_S^+ and base term γ_S^- were calculated according to the equation of van Oss et al.

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