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Surface and bulk properties of dental resin-composites after solvent storage

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ARTICLE INFO

Article history:

Received 10 September 2015

Received in revised form

11 May 2016

Accepted 13 May 2016

Keywords:

Resin-composite

Bulk-fill

Solvents

Micro-hardness

Diametral tensile strength

ABSTRACT

Objectives. To investigate the surface micro-hardness and the diametral tensile strength (DTS) of bulk-fill and conventional resin-composites after storage in food simulating solvents.

Methods. Eight materials were investigated. For the micro-hardness measurement, Teflon mould with an internal dimensions of 10 mm and 2 mm ($n=15$). For the DTS measurement, Split stainless steel moulds were used to make disk-shaped specimens of 6 mm diameter and 2 mm thickness ($n=15$). Materials were subdivided in to three groups (water, 75% ethanol/water and MEK). Micro-hardness measurements were made under a load of 300 gm with a dwell time of 15 s at 7, 30, and 90ds after storage. DTS was measured after 30ds at a cross head speed of 0.5 mm/min.

Results. The storage time and type of solvent had a significant influence on the micro-hardness. MEK showed more drastic reduction in the material micro-hardness with an exception of G-aenial universal flo (GA-F) which showed similar results in water/ethanol and MEK. DTS values of materials stored in water ranged from 48.7 MPa for the GA-F and 30.6 MPa for Ever X posterior (EXP). Generally, the results are observed to decrease with increasing solvent power, except for GA-F.

Significance. Bulk-fill materials showed no superior results compared with the other materials. For the bulk-fill materials that are designed to be used as a base, their penetration by the solvents may be shielded and thus the changes observed in this study may not be of clinical importance.

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1. Introduction

The continuous development of resin-composite materials aims for long lasting aesthetic dental restorations. There is a large range of available resin-composites, generalisation about their behaviour and performance should be made

cautiously, many resin-composite materials still have their own shortcoming in clinical performance [1]. Resin-composite restorations must survive an aggressive environment that varies from patient to patient as do the masticatory forces, occlusal habits, abrasive foods, chemically active foods and liquids, temperature fluctuations, humidity variations, bacterial products, and salivary enzymes. These factors, separately

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<http://dx.doi.org/10.1016/j.dental.2016.05.007>

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or collectively, determine the longevity of the restoration [1–6]. From a material aspect, the performance of the resin-composite restoration depends on several factors including the monomer system, the filler type, filler loading and the extent of cure [7].

Degradation of resin-composite restorations in the oral environment can be simulated by the use of different food simulating solvents which are known to cause different effects on the mechanical properties of the restoration and its longevity [4,8–10]. Similar polarity of a particular solvent and a substance will tend to make them mutually soluble, different polarity on the other hand will make the solubility difficult [11,12]. The effect of solvents on resin-composites is determined by their solubility parameter and the nature of their monomer. The solubility parameter is important in terms of its similarity to that of other substance [10].

The solubility parameter is defined as the square root of the cohesive energy density of the solvent; it is a mean of predicting the ability of a solvent to dissolve a substance (Eq. 5-1). It provides a clear numerical way of predicting the extent of interaction between materials. Solubility parameter is expressed as $\delta/\text{MPa}^{1/2}$ [11,12]. Water is known to be used as an aging media in several studies [10,13–15], ethanol and Methyl ethyl ketone (MEK) are known to represent food simulating solvents and cause an extreme dietary effect.

$$\delta = \sqrt{c} = \left[\frac{\Delta H - RT}{V_m} \right]^{1/2} \quad (1)$$

c: cohesive energy density, ΔH : heat of vaporisation, V_m : molar volume, R: gas constant and T: temperature.

In the chemical degradation theory, the chemical components of food and saliva may be absorbed by the resin matrix, resulting in softening and surface destruction [16]. Degradation of fillers could also be a potential problem regarding durability and marginal integrity as it may cause de-bonding from the material [17–19]. Clinical performance of resin-composite materials is determined by their mechanical properties including their fracture toughness, flexural strength, compressive strength, diametral tensile strength, surface hardness and wear resistance [5,20]. Failure of resin-composite restorations is commonly represented by fracture of the restoration, tooth fracture, marginal fracture, discolorations, marginal staining and secondary caries [1].

Surface hardness is a surface property that is defined as the resistance of the material surface to indentation [21]. Measuring the surface hardness can give an indication of the degree of conversion and consequently the clinical performance of resin-composite material after aging in food simulating solvents [22,23]. Furthermore, Dental restorations are expected to withstand tensile stresses from oblique or transverse masticatory functional loading, therefore tensile strength is an important property for any dental material [24]. Since resin-composites are relatively brittle under conventional loading rates, they would be expected to fail under tensile stresses during mastication. For this reason, the tensile strength of these materials may be considered to have more clinical relevance than the compressive strength [25].

Recently available resin-composites incorporate a range of monomer systems that may respond differently to aqueous

and non-aqueous solvents as present in food materials. For a given matrix, the most likely effect of a solvent is to decrease its mechanical properties [18,26] nevertheless, hardness of resin-composites have been shown to progressively increase over a period of time of at least 30 ds. Therefore, it is possible that some opposing trends could be operating during the period of solvent storage: first, the softening effect of the solvent and secondly the hardening effect due to the elevated degree of conversion [27–30].

The effect of the food simulating solvents is one aspect of simulation of the oral environment. Therefore, the objectives of the present study was to investigate the effect of three solvents (de-ionized water, 75% Ethanol/water and MEK) on the surface micro-hardness and diametral tensile strength of eight commercially available resin-composite materials. In this study, the null hypotheses were

- 1) There is no significant difference between the examined materials with the regards to their surface micro-hardness and DTS,
- 2) Different solvents provoke the same effect on different materials (on surface micro-hardness and DTS) and
- 3) There is no significant difference between the aging durations on the surface micro-hardness of the examined materials.

2. Materials and method

Eight resin-composite materials were investigated (Table 1). There were four bulk-fill, two nano-hybrid and two micro-hybrid resin-composites.

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2.2. Surface micro-hardness

120 disk-shaped specimens were produced according the manufacturers' instructions. 15 specimens were made for each material using a Teflon mould with an internal diameter of 10 mm and 2 mm thickness. The specimens were made carefully, to avoid any air entrapment during placement of the uncured material. The specimens were fabricated at a room temperature of $23 \pm 1^\circ\text{C}$ and a relative humidity of $50 \pm 2\%$. The mould was sandwiched between two polyester films and microscopic slides (1 mm thickness) on each side. The curing was made for 20 s at an output irradiance of 1200 mW/cm^2 using an Elipar S10 LED curing light from 3M ESPE. The light output was measured using a laboratory grade NIST-referenced USB-4000 spectrometer (MARC Resin Calibrator v.3, Blue-light analytics Inc, Halifax, NS, Canada). The specimens were irradiated from top and bottom surfaces. The specimens were finished to remove any irregularities using 1000 grit abrasive papers.

Immediately after polymerization, the specimens were stored in an oven at a $37 \pm 1^\circ\text{C}$ for 24 h. The baseline

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