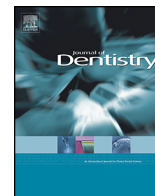




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Long-term sorption and solubility of bulk-fill and conventional resin-composites in water and artificial saliva

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ABSTRACT

Objectives: To assess sorption and solubility of several bulk-fill and conventional resin-composites after one-year storage in water and artificial saliva (AS).

Methods: Six bulk-fill (SureFil SDR, Venus Bulk Fill, X-tra base, Filtek Bulk Fill flowable, Sonic Fill, and Tetric EvoCeram Bulk Fill) and eight conventional resin-composites (Grandioso Flow, Venus Diamond Flow, XFlow, Filtek Supreme XTE, Grandioso, Venus Diamond, TPH Spectrum, and Filtek Z250) were tested. Disc shaped samples ($n=5$) were randomly immersed into distilled water and AS for one-year period and weighed at different time intervals. Data were analysed using repeated measures ANOVA, one-way ANOVA, and Tukey's post hoc test ($\alpha=0.05$).

Results: In water, all materials (with the exception of X-Flow) reached a stable mass within three months with a slow increase observed in AS up to one year. Sorption values in water and AS for most materials were not significantly different ($p \geq 0.2$). Sorption and solubility values in water ranged from ($6.5 \mu\text{g}/\text{mm}^3$ and $-1.77 \mu\text{g}/\text{mm}^3$ respectively) for X-tra base to ($78.8 \mu\text{g}/\text{mm}^3$ and $44.77 \mu\text{g}/\text{mm}^3$ respectively) for X-Flow ($p < 0.005$). Sorption of the polymer matrix in water ranged from 1.18% for XB to 9.95 % for XF.

Conclusions: Water sorption and solubility of resin-composites are material-dependent and highly affected by the filler loading and hydrophilicity of the resin matrix. BisEMA and UDMA-BisEMA based resins appeared to be more hydrophobic than BisGMA based systems. Water and AS, are generally comparable as storage media in terms of water sorption.

Clinical significance: Bulk-fill materials and conventional resin-composites tested varied in terms of sorption and solubility but both were considered stable in longterm water storage. The composition of each material is critical and can affect the long-term clinical performance of either type of resincomposites.

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1. Introduction

Resin-composites today are considered the material of choice for restoring load bearing surfaces of posterior teeth using the minimally invasive approach [1]. Their properties have improved over time to increase their stability in the challenging oral environment. Resin-composites should ideally be highly stable and impermeable to water, however, dental polymer networks based on dimethacrylate monomers have been shown to absorb moisture to the extent of several percent of their total weight [2,3]. Water sorption is considered a critical property in determining the

clinical success of a dental restorative material. It has a negative effect on a resin-composite hydrolytic stability which contributes to material's discolouration [4], deterioration of mechanical properties [5,6], reduced wear resistance [7], and hydrolytic degradation of bonds especially at resin-filler interface [8]. Water sorption also contributes to material's hygroscopic expansion [9] and hygroscopic stress which could result in micro-cracks or even cracked cusps in restored teeth [10].

Moisture absorption into a resin composite occurs mainly by direct absorption into its polymeric resin matrix, and its extent is directly related to the amount and properties of this phase [11,12]. Glass fillers do not contribute to the sorption process but water may get adsorbed onto their surface depending on the quality of the interface between the glass fillers and the polymeric resin matrix [13]. Sorption into a polymeric material is a diffusion

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controlled phenomenon that can be explained by two theories: the free volume theory, and the interaction theory [14]. The free volume theory involves solvent absorption through voids in the polymer, while in the interaction theory; water binds to specific ionic groups of the polymer chain depending on the water affinity of these groups [15]. In general, the amount of water sorption into a polymeric structure is influenced by the degree of conversion [16], the cross-link density [8], and hydrophilicity of the polymeric network [6]. The absorbed water may result in swelling and widening of space between polymer chains depending on the degree of cross-link density of its structure. This allows free unreacted monomers trapped in the polymer network to diffuse out into the storage solvent depending on their molecular size and their affinity to the aqueous solvent [11]. Solubility compromises the biocompatibility of the material and reduces its bulk which weakens its mechanical properties [14].

Sorption and solubility studies of conventional resin-composites and neat dental resins have been mainly conducted in water and for limited time periods [11,12] with fewer studies conducted in other media such as ethanol [17,18], artificial saliva [18–20], and different pH solutions [21]. Recently, a new class of resin-composites, so called bulk-fill, has been introduced into the market including several materials, some of them are based on new monomer technology [22]. Bulk-fill composites may contain polymerization modulator chemical groups or plasticizers in their resin matrix to reduce the effect of polymerization shrinkage stress when these materials are applied in bulk [23]. Such chemical alterations may affect the quality of the polymer network of these materials and their resistance to moisture compared to conventional resin-composites. Although many studies have been conducted on bulk-fill resin-composites to assess their polymerization [24], depth of cure [25], and their short-term physico-mechanical performance [26,27], the literature is still lacking data regarding their long-term stability in aqueous media compared to conventional materials.

The aim of this study is to assess sorption and solubility of several bulk-fill and conventional resin-composites after one-year storage in water and artificial saliva. The null hypotheses to be tested were: (i) there would be no significant effect of material's type on sorption and solubility, (ii) there would be no significant effect of storage medium on sorption and solubility after one-year storage, and (iii) there would be no significant long-term mass change during the one-year storage period for all materials in both storage media.

2. Materials and methods

2.1. Preparation of samples

Fourteen commercial resin-composite materials including six bulk-fill materials and eight conventional resin-composite materials were tested. A list of the resin-composites studied is given in Table 1. Samples were prepared ($n=5$) using a brass ring mould (15 mm diameter \times 1 mm thickness) according to the dimensions specified by ISO FDIS 4049:2009 [28]. Samples were fabricated by applying each material into the mould placed against a polyester matrix strip and a glass slab. The mould was slightly overfilled with the material and the excess was then extruded by applying another polyester matrix strip and a glass slab and pressed firmly. Care was taken to minimize entrapped air while uncured materials were applied into the mould. Each sample was then cured for 20 s at five overlapping sections of the top and bottom surfaces using an LED light curing unit (EliparTM, 3M ESPE, USA), with a 10 mm diameter light exit, under standard curing mode. The light curing unit had an output irradiance of *circa* 1200 mW/cm² and wavelength range 430–480 nm as stated by the manufacturer. A calibrated radiometer system (MARC Blue-light Analytics Inc., Halifax, NS, Canada) was used to ensure consistent irradiance at each use of the light curing unit. After curing, each sample was gently pushed out from the mould and excess flash at the periphery was removed using either a sharp blade or a grit abrasive paper.

Samples were then transferred into separate glass vials and stored in a lightproof desiccator with anhydrous self-indicating silica gel at $(37 \pm 1)^\circ\text{C}$. After 22 h, the samples were moved into another desiccator maintained at room temperature $(23 \pm 1)^\circ\text{C}$ for 2 h and then weighed to an accuracy of 0.01 mg using a calibrated electronic analytical balance (Ohaus Analytical Plus, Ohaus Corporation, USA). This cycle was repeated until the mass loss of each specimen was not more than 0.1 mg in any 24 h period to ensure the completion of post-irradiation polymerisation and dehydration. This constant mass m_1 was the initial mass of the specimen.

After final drying, the samples dimensions were measured using an electronic digital calliper (Powerfix, OWIM GmbH & Co., KG, Germany) with an accuracy of 0.01 mm. Two measurements of the diameter of each sample were taken at right angle to each other. The thickness was measured at the centre of the sample and at four equally spaced points on the circumference. Using the average mean diameter and thickness, the volume (V) of each sample was calculated in cm³.

Table 1

Test materials and manufacturer information [bulk-fill (light grey) and conventional composite (dark grey)].

Material	Code	Organic matrix	Filler (wt%)	Filler (vol%)	Lot number	Manufacturer
SureFil [®] SDR [®] flow	SDR	Modified UDMA, BisEMA, TEGDMA	68	44	10211	DENTSPLY Caulk, USA
Venus Bulk Fill	VBF	UDMA, BisEMA, TEGDMA	65	38	010101	Heraeus Kulzer GmbH, Germany
X-tra base	XB	BisEMA, EBPADA	75	NA	1208392	VOCO GmbH, Germany
Filtek Bulk Fill Flowable	FBF	BISGMA, UDMA, BisEMA, TEGDMA, Procrlyat resin	64	42.5	N370958	3 M ESPE GmbH, Germany
TetricEvoCeram [®] Bulk Fill	TEC	BISGMA, UDMA, BisEMA	77	60–61	R04686	Ivoclar Vivadent
SonicFill [™]	SF	BISGMA, TEGDMA, BisEMA	83.5	NA	4964921	Kerr Corporation, USA
Grandioso Flow	GRF	BISGMA, TEGDMA, HDDMA	81	NA	1305362	VOCO GmbH, Germany
Venus [®] Diamond Flow	VDF	UDMA, BisEMA, TEGDMA	65	41	010104	Heraeus Kulzer GmbH, Germany
X-Flow	XF	DEGDMA, BisEMA	60	NA	1267	DENTSPLY Caulk, USA
Filtek [™] Supreme XTE	FF	BISGMA, TEGDMA, BisEMA, Procrlyat resin	65	46	N522058	3 M ESPE GmbH, Germany
Grandioso	GR	BISGMA, TEGDMA	89	73	1304304	VOCO GmbH, Germany
Venus Diamond	VD	UDMA, TCD-DI-HEA, BisEMA	64	63.5–65.1	010046	Heraeus Kulzer GmbH, Germany
TPH [®] 3 Spectrum	TPH	BISGMA, BisEMA, TEGDMA	75	57	1301000713	DENTSPLY Caulk, USA
Filtek [™] Z250	Z250	UDMA, BisGMA, BisEMA, TEGDMA	82	NA	N458477	3 M ESPE GmbH, Germany

NA stands for not available.

UDMA: urethane dimethacrylate, BisEMA: Bisphenol A ethoxylate dimethacrylate, TEGDMA: triethylene glycol dimethacrylate, DEGDMA: diethylene glycol dimethacrylate, BisGMA: Bisphenol A glycidyl dimethacrylate, HDDMA: 1,6-hexanediol dimethacrylate, TCD-DI-HEA: bis-(acryloyloxyethyl) tricyclo[5.2.1.0_{2,6}]decane.

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