



Effect of Water Sorption and Solubility on Color Stability of Bulk-Fill Resin Composite

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ABSTRACT

Aim: The objective of this study is to compare the bulk-fill resin composite to a conventional one, as regards; water sorption, solubility, and their effect on color stability.

Materials and methods: This study was conducted using two types of composites: Bulk-fill composite (Filtek Bulk-Fill) and nanohybrid composite for control (Filtek Z250 XT). Specimens were prepared using a ring mold, 10 from each material. Specimens were desiccated, then weighed in a digital balance until a stable mass was acquired. For water sorption test, specimens were immersed in distilled water and placed in a lightproof incubator at $37 \pm 1^\circ\text{C}$ and the mass was measured weekly for 8 weeks. For solubility test, specimens were desiccated again in the desiccator until a stable mass was achieved. A spectrophotometer was used to record the baseline color measurements prior to water immersion and weekly for 8 weeks.

Results: Bulk-fill composite showed higher water sorption value and lower water solubility values compared with that of the conventional one. No statistically significant difference was found for water sorption ($p = 0.104$) or water solubility ($p = 0.098$) between groups. The mean ΔE was lower in bulk-fill than conventional composite, and results showed a statistically significant difference ($p = 0.009$).

Conclusion: Bulk-fill resin composite yielded better color stability and similar water sorption and solubility values.

Clinical significance: Bulk-fill resin composite can be used in esthetically concerned patients.

Keywords: Bulk-fill composite, Color stability, Solubility, Water sorption.

How to cite this article: Mansouri SA, Zidan AZ. Effect of Water Sorption and Solubility on Color Stability of Bulk-Fill Resin Composite. *J Contemp Dent Pract* 2018;19(9):1129-1134.

Source of support: Nil

Conflict of interest: None

INTRODUCTION

Resin composite is considered one of the most popular restorative materials nowadays, but it has a major drawback that it is subjected to dimensional changes leading to failure in the bond. The reason for these changes is inadequate polymerization. The polymerization can induce internal stresses and shrinkage to the material. This will result in internal microcracks and separation from the bonding agent to form a gap that will cause leakage into the dentin and produce postoperative sensitivity, recurrent caries, and marginal discoloration.¹

The introduction of bulk-fill resin composite promised to solve the problem of dimensional changes of the resin composite when it was first launched in February 2011. The main difference of this material is that it can be placed in bulk for up to 4 mm thickness instead of the conventional 2 mm thick incremental placement. This technique will reduce the chair time, leading to less chance for water and saliva contamination. This means that this technique is more cost-effective in the long run.²

When placing the increments there are high chances of voids forming between increments. These voids will prevent complete adaptation and can weaken the restoration, leading to bulk fracture, and the bulk-fill technique reduces these voids significantly.³

The bulk-fill resin composite differs from conventional composites by the organic resin structure that has double the thickness of the polymerized resin composite layer with decreased polymerization time.⁴

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The clinically relevant properties of the bulk-fill material have been investigated in several articles showing a lower rate of monomer elution compared with conventional materials. Czasch and Ilie² compared the degree of conversion between 2 and 4 mm samples and found no lowering in mechanical properties between the samples. These results correspond well with the results of Finan et al,⁵ who found no significant difference in the degree of convergence in depths of 1 to 4 mm, but the degree decreases linearly at depths of 5 to 8 mm, they also found a reduction in the released bisphenol A-glycidyl methacrylate (bis-GMA) and triethylene glycol dimethacrylate (TEGDMA) from bulk-fill materials compared with the conventional ones.

Water sorption plays a crucial role in deciding the dental material clinical success, although resin composite is considered impermeable to water and highly stable. The presence of polymer networks showed moisture absorption that accommodates several percentages of material total weight. Water sorption has a negative effect on the restorative material by contributing to discoloration, reduced wear resistance, deterioration of mechanical properties, the release of unreacted monomers, and hydrolytic degradation of bonds mostly at resin–filler interface.⁶

The effect of water sorption on the resin composite behavior is influenced by several features, such as the composition of the polymer matrix, the type and content of the filler, and the size and shape of filler particles.⁷

Janda et al⁸ conducted an experiment to investigate the water sorption and solubility differences between various types of dental resin composites and found that the correlation between water sorption and filler load was significant. The lowest water sorption values were found in the composite with the highest filler load, and the highest water sorption values were found in the composite with the lowest filler load.

Solubility can compromise the material biocompatibility and reduce its bulk, which weakens its mechanical properties.⁹

Previous studies showed that bulk-fill composite is considered stable when stored in water for a long time, but there are no reports about the effect of water sorption and solubility on color stability of the material.⁷

Several factors can affect resin composite color stability that include extrinsic and intrinsic factors, the size and configuration of the filler particles. These factors can affect the polished surface and make the composite susceptible to extrinsic stains. The intrinsic factor can also influence color stability; it includes incomplete polymerization and resin matrix composition. By having lower polymerization shrinkage, the bulk-fill composite can improve the color stability of the resin-based composites.¹⁰

Water solubility can attribute to discoloration by chemically degrading the filler–resin bond of the resin matrix. Light curing unit spectral distribution, exposure time, and intensity can also play their role in polymerization quality and therefore, discoloration.¹¹

Color stability is becoming very important because of more patients seeking color matching restorations. Therefore, color stability is one of the important criteria determining the success of resin composite restorations because it is one of the main reasons for replacing resin-based restorations.¹⁰

MATERIALS AND METHODS

This study was conducted using two commercial resin composite materials (Fig. 1): One was a bulk-fill composite (Filtek Bulk-Fill, 3M ESPE, GmbH, Germany) and the other was nanohybrid resin composite as a control (Filtek Z250 XT, 3M ESPE, GmbH, Germany). To standardize this study, Vita shade A2 was used for the two material types. The technical profile of each resin composite is shown in Table 1.

The prepared samples are used to measure: (1) Water sorption and solubility; (2) color stability.

A total of 20 disk-shaped specimens were prepared using a ring mold (Fig. 2), 10 from each material, with the dimensions of 2 thickness × 10 mm diameter (n = 20).

Fabrication of the samples was done for each material by applying it into the ring mold. A glass slab was used to support the ring mold. The material was slightly over-filled into the mold, and another glass slab was pressed firmly to extrude the excess material, and care was taken to minimize air entrapment.

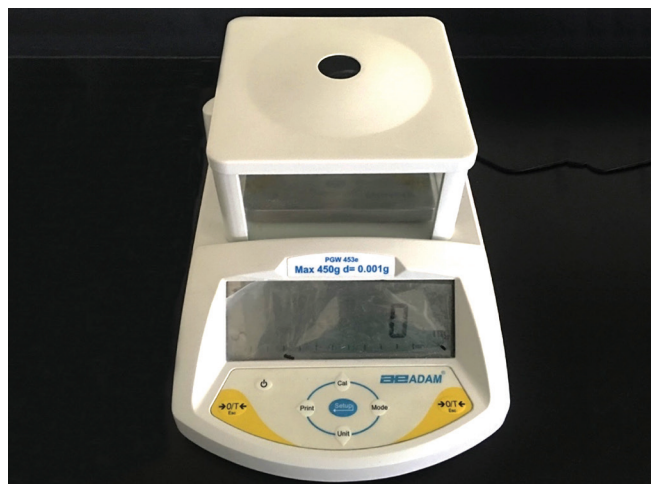
Curing of each sample was done for 40 seconds in the top surface and for 20 seconds in the bottom surface using light curing unit with LED lights (Ivoclar Vivadent, Schaan, Liechtenstein) under standard curing mode. The light curing unit output irradiance was circa 1,200 mW/cm² and the wavelength was 430 to 480 nm.



Fig. 1: Filtek Bulk-Fill posterior restorative and Filtek Z250 XT

Table 1: The technical profile of the tested resin composites

Material	Composition	Filler (wt%)	Filler (vol%)	Manufacturer
Filtek Z 250XT	The resin matrix: Bis-GMA, UDMA, bis-EMA, PEGDMA and TEGDMA The filler: Surface-modified zirconia/silica with a median particle size of approximately 3 µm or less. Nonagglomerated/nonaggregated 20 nm surface-modified silica particles	82	68	3M ESPE GmbH, Germany
Filtek Bulk-Fill posterior restorative	The resin matrix: ERGP-DMA, diurethane-DMA and 1,12-dodecane-DMA The filler: 20 nm silica filler, a nonagglomerated nonaggregated 4–11 nm zirconia filler, an aggregated zirconia/silica cluster filler (comprised of 20 nm silica and 4–11 nm zirconia particles), and a ytterbium trifluoride filler consisting of agglomerate 100 nm particles	76.5	58.4	3M ESPE GmbH, Germany

**Fig. 2:** Disk-shaped specimen**Fig. 3:** Digital balance

After curing, specimens were pushed out gently using a blunt instrument with cloth attached to it to prevent scratching that may alter shade results. Samples were stored in vials and labeled with the group name and sample number.

Water sorption and solubility were determined in this study, by the procedure described in ADA specification number 27.¹² Specimens were placed in a desiccator with anhydrous self-indicating silica gel for 48 hours and then weighed in a digital balance (Fig. 3), with an accuracy of 0.001 gm (Adam equipment, Oxford, Connecticut, USA) until a stable mass was acquired (m_1). After stabilization, specimens were subjected to distilled water immersion and placed in an incubator (ThermoFisher Scientific, Waltham, Massachusetts, USA) at $37 \pm 1^\circ\text{C}$.

To calculate water sorption values, specimens were removed individually and dried gently with an absorbent sheet and weighed to acquire (m_2). To calculate solubility values, specimens were reconditioned in the desiccator in the same manner as for m_1 , then weighed until a stable mass is acquired again (m_3). Minor differences in the weight may be attributed to mold size variations of ± 1 mm in the diameter measurements.

Measurements were taken after 24 hours and for the first 4 days and then weekly till saturation. Weight changes were monitored for 8 weeks.

Water sorption and Wsl values were calculated using the following equations for each specimen:⁷

$$W_{sp} = 100 \times \frac{m_2 - m_1}{m_1}$$

$$W_{sl} = 100 \times \frac{m_1 - m_3}{m_1}$$

A spectrophotometer (VITA Easyshade, Zahnfabrik, Bad Sackingen, Germany) (Fig. 4) was used to record the baseline color measurements of all specimens prior to distilled water storage according to the CIE $L^*C^*h^*$ system. A small mark was made with a graphite pencil to repeat the tested surface and measurement was taken away from that mark.

CIE $L^*C^*h^*$ scores were calculated against a white background relative to a standard illuminant, with L representing lightness (lighter or darker), C representing chroma (brighter or duller), and h representing hue angle. Specimens color was measured and the mean value of three measurements for L^* , C^* , and h^* was recorded. Color measurements were acquired again after 1, 4, and 8 weeks of distilled water storage at $37 \pm 1^\circ\text{C}$, and the following formula was used to calculate color changes (ΔE).¹³

$$\Delta E = [(L_1 - L_0)^2 + (C_1 - C_0)^2 + (h_1 - h_0)^2]^{1/2}$$



Fig. 4: Spectrophotometer

Statistical Package for the Social Sciences (SPSS) version 23.0 Windows software (SPSS Inc., Chicago, Illinois, USA) was used to analyze the data. The acquired data from water sorption, solubility, and color stability tests were analyzed using independent t-test ($p = 0.05$) to determine the possible correlation between water sorption, solubility, and color stability.

RESULTS

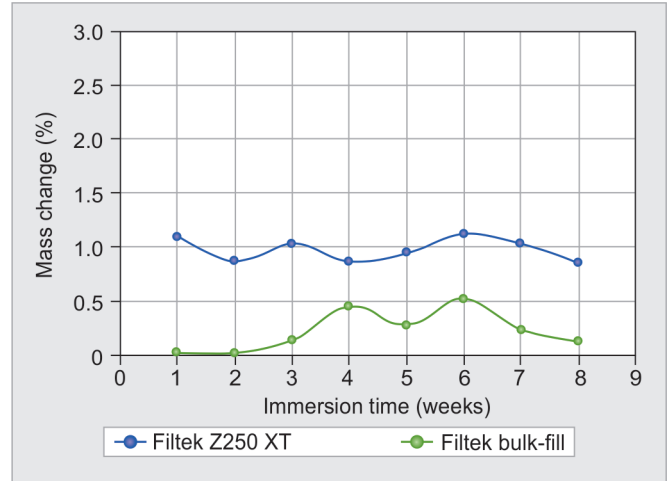
The results were transferred to Microsoft Excel 2016 spreadsheet (Microsoft Corporation, USA) and then the data were analyzed by Windows software (SPSS Inc., Chicago, Illinois, USA).

An independent t-test was performed at a significance level of $p < 0.05$ to compare the water sorption and solubility of the two materials and the mean ΔE . The results include mean and standard deviation and p-value for the water sorption, solubility, and ΔE of the study are presented in Table 2.

All materials show mass change with time upon water storage. The illustration in Graph 1 demonstrates the change of mass in the course of immersion time. After 8 weeks bulk-fill showed higher water sorption value (0.85 ± 0.73) than conventional composite (0.17 ± 1.08). Water solubility values, on the contrary, favored the bulk-fill composite with a mean of 0.28 ± 0.35 compared with 0.70 ± 0.67 for the conventional composite.

Table 2: The mean, standard deviation, and p-value of compared materials

Materials	n	Water sorption (%)	Solubility (%)	Color change (ΔE)
Filtek Z250 XT	10	0.17 ± 1.08	0.70 ± 0.67	2.48 ± 0.78
Filtek Bulk-Fill	10	0.85 ± 0.73	0.28 ± 0.35	1.43 ± 0.83
p-value		0.104	0.098	0.009



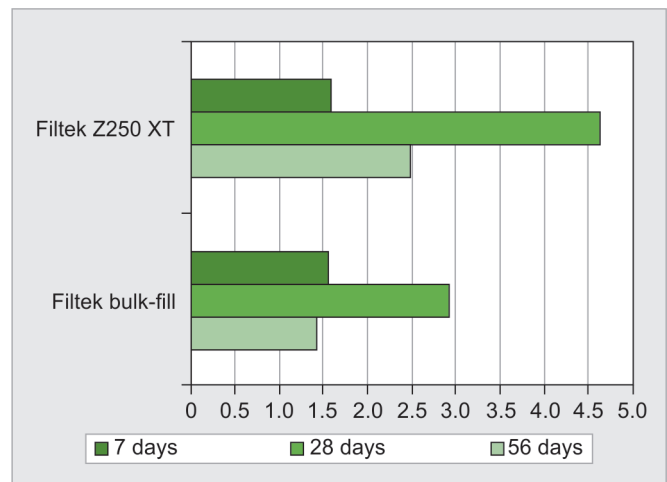
Graph 1: Means of changed mass over the immersion time

Table 3: The mean sorption and mean ΔE compared

Material	Time interval (days)	Mean sorption (%)	Mean color change (ΔE)
Filtek Z250 XT	7	0.0323	1.585
	28	0.445	4.637
	56	0.704	2.488
Filtek Bulk-Fill	7	0.802	1.563
	28	0.946	2.927
	56	0.283	1.434

No statistically significant difference was found using independent t-test for water sorption ($p = 0.104$) or water solubility ($p = 0.098$) between groups. A comparison of the mean sorption and mean ΔE is presented in Table 3. The mean ΔE was lower in bulk-fill (1.43 ± 0.83) than conventional composite (2.48 ± 0.78), and independent t-test showed a statistically significant difference between the two ($p = 0.009$).

The illustration in Graph 2 shows the changes in ΔE means over 1, 4, and 8 weeks.



Graph 2: Change of ΔE means over time

DISCUSSION

Water was used to evaluate weight changes because saliva is a diluted fluid composed of 99% water. It simulates the liquids that typically found in the mouth interacting with restorations and teeth. The concentration of dissolved solids (organic and inorganic) is widely variable from person to person and within the same person. Consequently, deionized distilled water was used as test standard.¹⁴

The packable composite was used in this study because they are recommended to use in stress-bearing areas as the higher filler content and distribution make the consistency of the material stiffer and improve handling properties.¹⁵

The results showed an increase in mass in the two types of resin composite. Water storage will cause expansion of the restoration that could be clinically favorable if it counteracts the effect of shrinkage, but if it exceeds the polymerization shrinkage the tooth will be introduced to further stress.¹⁴

The presence of hydrophilic resin matrices like bis-GMA, TEGDMA, and urethane dimethacrylate (UDMA) may cause absorption of water to a greater degree than hydrophobic resin. Nevertheless, determining the degree and rate of water sorption may not be relayed solely on monomer type. Rather, discrete zones presence within the restorative material could possibly produce an osmotic effect that may influence water sorption. Thus, the water sorption phenomenon is a multifactorial process and influenced by more than one factor not just by the media of immersion.¹⁴

Resin matrix capacity to absorb water is determined by the percentage of filler volume in relation to the volume of the restoration. Hence, when the material has lower filler content and high resin matrix content, it will yield higher water sorption values.¹⁶

The finding of the present study coincides with the finding mentioned earlier. The bulk-fill composite resin contains slightly lower filler volume than the conventional nanohybrid composite that leads to an increased water sorption result.

The accessibility of water will determine the rate of water sorption. This means the degree and rate of water sorption for active composite restoration is actually lower compared with the water sorption in the *in vitro* studies, because disks are free in water with large surface exposer, meaning it can absorb water from all surfaces; while in mouth, composite restoration that is confined within limited tooth surfaces will have smaller surface area to absorb water.¹⁷

The solubility of resin composite material is influenced by leaching monomers that did not react to curing. Therefore, increased conversion of monomers will lead to lower solubility as a higher percentage of C=C bonds means the leaching of unreacted monomer will be in a smaller amount. Other factors, such as particle size, filler

concentration, surface area, and coupling agent may be considered as it can influence material solubility.⁹

This study showed a slight decrease in solubility in the bulk-fill composite during testing compared with the conventional composite. This can be attributed to the more proper bonding of fillers to the matrix.

The composite material can be susceptible to color changes by two types of factors, endogenous and exogenous. Endogenous factors depend on material composition, including filler (size, content, hardness, ratio) and initiator systems, whereas exogenous factors can be from the accumulation of plaque and absorption of stains (dyes or pigments).¹⁸

Staining susceptibility of resin composite is determined by their water sorption and their degree of hydrophilicity. The composite that absorbs water can absorb other fluid, leading to discolorations. Resin matrix type is an important factor of discoloration potential as UDMA matrix exhibits less water sorption and higher resistance to stains than bis-GMA matrices as it is too viscous and used without thinning. It cannot be purified as it is a mixture of high molecular weight optical isomer. Susceptibility to staining can also be influenced by the surface roughness parameter that is linked to the content of the filler.¹⁹

The results of this study showed that bulk-fill resin composite has fewer color changes (ΔE). This means it is more color stable compared with conventional composite after immersion of water. This finding can be linked to the decreased solubility of the bulk-fill materials found in this study. Furthermore, the increased sorption could be the cause of increased susceptibility of the bulk-fill composite material to discolor with staining fluids like coffee as shown in other studies, because more sorption means more susceptibility to extrinsic stains.²⁰

The CIE L*C*h* color system was selected for the color assessment in the current study. The ΔE value presents relative color changes of dental materials or tooth surfaces before and after an intervention. According to reviews, ΔE values of more than 3.3 are considered noticeable by nonexperienced individuals and are not clinically acceptable. Hence, color changes above a ΔE value of 3.3 were considered clinically unacceptable.²⁰

In this study, significant differences in ΔE values were found among the two materials favoring the bulk-fill composite but both values were below 3.3 and thus considered clinically acceptable.

CONCLUSION

Bulk-fill resin composite showed higher water sorption and less solubility with fewer color changes. However, there is no statistical difference between bulk-fill and conventional resin composite regarding water sorption and solubility after water storage.

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