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Water Sorption, Solubility and Color Stability of Different Bulk-Fill Restorative Materials

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ABSTRACT

Objectives: The aim of this study was to evaluate the color stability, water sorption and solubility of recent bulk-fill restorative materials.

Materials and Methods Seventy disc shaped specimens (height:1mm, diameter:15mm) were prepared with a micro-hybrid composite resin (Z250; Filtek Z250, 3M ESPE), two resin-based bulk-fill (Filtek One Bulk Fill, 3M ESPE and EBF; Estelite Bulk-Fill Flow, Tokuyama Dental), glass ionomer-based restorative (Equia FF; Equia Forte Fil, GC Dental), self-cure and light-cure alkasite (CN; Cention N, Ivoclar Vivadent) and indirect composite resin (Gradia P; Gradia Plus, GC Dental) (n=10). Baseline color measurements were performed and mass of the samples (m1) were noted. Color measurements were repeated after 24 h and 28 days immersion of the specimens in water. After the 28-days immersion, mass was noted as m2. Specimens were kept in desiccators and final mass (m3) were recorded. The color change (ΔE_{00}) water absorption (WSP) and water solubility (WSL) of the specimens were calculated.

Results: After 28-days immersion, Gradia P showed significantly lower water sorption than EBF and Equia FF groups ($p<0.05$). Light-cured and self-cured CN groups showed similar sorption with resin composite groups ($p>0.05$). The lowest solubility was observed in Z250and Gradia P. The lowest (ΔE_{00}) was observed in self-cured CN group ($p<0.05$) and Gradia P showed the highest ΔE_{00}.

Conclusions: Light-cured and self-cured CN groups demonstrated similar sorption to the conventional composite resin and indirect composite resin. Self-cured CN demonstrated higher water solubility than composite resins. Restorative materials showed moderately unacceptable color change after 28-day immersion in distilled water.

Keywords: Water Sorption, Solubility, Color Change.

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Introduction

Along with the advances in restorative materials, composite resins have been the material of choice in clinical practice for restoring anterior and posterior teeth. Maintenance of esthetic, mechanical and physical properties over time, that could be the reason for the restoration replacement, is required for the longevity.[1] However, long-term stability of restorative materials is a major concern due to the ambient conditions of oral environment and the detrimental effects on the restorative material structure.[2,3]

Water sorption can produce deleterious effects on the restorative material by leading to swelling, oxidation, hydrolysis, softening and plasticization.[3] Ideally the restorative material should be impermeable and highly resistant to water sorption and solubility.[4] However previous studies reported different extents of water sorption and solubility values for restorative materials varying according to the type and ingredients of the materials.[4-7] Water sorption of resin-based materials occurs through the voids in the structure and binding of water to hydrophilic polymers.[4] For the glass ionomer based restorative materials, as a result of water sorption, calcium and aluminum ions are transported and in fact, ions removed from the material over time.[6] In addition, water sorption and solubility shortens the life span of the restorations by causing color change.[5,8]

The indirect composite resins have been used for about three decades with higher mechanical and physical properties and reduced polymerization shrinkage in comparison with conventional composite resins.[9] Indirect composite resins require longer polymerization time or additional heat, vacuum and pressure treatments applied based on the initiator system used.[10] Owing to the higher polymerization rates and reduced unreacted monomers, less water sorption and solubility can be expected for the indirect composite resins.[11]

Recently, tooth colored and self-adhesive direct restorative material, Cention N (CN; Ivoclar Vivadent, Schaan, Liechtenstein) was introduced. CN is papered by hand mixing the powder and liquid, and can release calcium, fluoride and hydroxide ions during the service life. In addition, CN can be considered as a basic filling material due to it can be used with or without an adhesive system and can be set self-cure or with light activation.[12] Generally, ease of use of restorative materials is desired and user-friendly materials are preferred by the clinicians.[13] In addition, CN can be placed within the cavity in bulk likewise the bulk-fill composite resins and restorative glass ionomers. Bulk-fill composite resins were introduced with the purpose of simplifying the restorative procedure and shorten the chair time. Recently, regular and flowable restorative bulk-fill composite resins that not need to a capping layer are available on the market.

Previous studies reported similar or lower water sorption and solubility values for the bulk-fill restoratives and higher values for the glass ionomer based restoratives compared to composite resins.[4,6,7] Restricted number of previous studies have evaluated the water sorption and solubility of CN, however results are somewhat conflictive. In addition to the mechanic and physical properties, color stability of the restorative materials is an important esthetic parameter that affect the longevity of the restoration.[14]

Although the color stability and water sorption and solubility of restorative materials has been evaluated in many studies, no published article has yet compared different restorative materials that placed in bulk. Therefore, the aim of this study was to evaluate the color stability, water sorption and solubility of recent bulk-fill restorative materials. The null hypothesis tested were that type of restorative material have no effect on the color stability, water sorption and solubility.

Materials and Methods

A universal micro-hybrid resin composite (Z250; Filtek Z250, 3M ESPE, St Paul, MN, USA), two resin-based bulk-fill restoratives (FOB; Filtek One Bulk Fill, 3M ESPE, St Paul, MN, USA and EBF; Estelite Bulk-Fill Flow, Tokuyama Dental Corporation, Tokyo, Japan), a glass ionomer-based bulk-fill restorative (Equia FF; Equia Forte Fil, GC Dental Products Corp, Tokyo, Japan), a bulk-fill alkasite restorative (CN; Cention N) and an indirect composite resin (Gradia P; Gradia Plus, GC Dental Products Corp, Tokyo, Japan) were tested in the present study. The shades of the materials were A2 except for HB-DA2 was used in Gradia Plus group. The composition and polymerization or setting procedures of the materials are described in Table 1.

Specimen preparation

Assuming a medium effect size (F=0.35) of the difference between the groups, at least 10 samples per group were required with a power of 80% and an alpha error of 5%. Therefore, total of seventy disc-shaped specimens (n=10) were prepared using teflon molds (depth: 1 mm and diameter 15 mm). The tested materials were inserted into the mold that was placed on a microscope slide. After inserting, another microscope slide was placed on the mold and pressed. After the removal of excess material, the resin-based composite materials were polymerized with a Valo LED unit (Ultradent Products Inc., South Jordan, UT, USA) at the standard power of 1000mW/cm2 for 20 s. The chemically activated materials (Equia Forte Fil and Cention N self-cure) were left untouched to set for 2.5 min for Equia Forte Fil and 4 min for Cention N self-cure group. After the setting, the surface coating (Equia Coat, GC) was applied on Equia Forte Fil specimens and light polymerized for 20 s. For the Gradia Plus, initial polymerization was performed using a hand light-curing device (Valo, Ultradent Products Inc.) at the standard power for 20 s. Following, post polymerization was performed in a light-curing oven (Labolight DUO, GC, Tokyo, Japan) for 3 min in full-mode.

Water Sorption and Solubility

After the polymerization or setting of the restorative materials, samples were placed in a glass vacuum desiccator at 37±1 °C, and dried for 22 hours. Then, the samples were transferred to a second desiccator and dried for 2 hours at 23±1 °C. The samples were weighed using a digital assay balance (with an accuracy of 0.01 g). This 24 h cycle was repeated until a constant mass of each sample (not more than ±0.1 mg) was obtained and the baseline mass of the samples (m_1) were noted. Afterwards, the samples were immersed in distilled water at 37 ± 1°C for 28 days, in containers that have separate covered compartment for each sample to avoid contact, and evaporation of the distilled water. The samples were dried with absorbent paper and weighed after 1, 7, and 28 days, and mass value was noted as m_2. After the 28 days, using the same protocol as for m_1, specimens were kept in the desiccators until a constant mass was obtained, and the final mass of the samples (m_3) were noted. The water absorption (W_{SP}) and water solubility (W_{SL}) of the each sample were calculated separately with the following equations:

W_{SP}: $m_2 - m_1 / V$ (mg/mm^3)

W_{SL}: $m_1 - m_3 / V$ (mg/mm^3)

Color Stability

Color measurements were performed using a spectrophotometer (VITA Easyshade, Vita Zahnfabrik, Bad Säckingen, Germany) relative to the standard illumination and against a white background. The measurements were repeated for three times and average L, C, H color parameters of Commission Internationale de l'eclairage (CIEDE2000) system were recorded to calculate the color differences (ΔE_{00}) by the following equation:

$$\Delta E_{00} = \sqrt{\left(\frac{\Delta L'}{k_L S_L}\right)^2 + \left(\frac{\Delta C'}{k_C S_C}\right)^2 + \left(\frac{\Delta H'}{k_H S_H}\right)^2 + R_T \frac{\Delta C'}{k_C S_C} \frac{\Delta H'}{k_H S_H}}$$

The baseline color measurements (T_1) were performed after the specimen preparation. The color measurements were repeated after 24 h (T_2) and 28 days (T_3) immersion of the specimens in the distilled water. The ΔE_{00} values between the T_1-T_2 and T_1-T_3 were calculated separately.

FE-SEM observation

After the 28-day immersion in distilled water, one sample from each group was randomly selected for the observation of surface characteristics. The samples were sputter-coated with Pt-Pd and observed using FE-SEM (Mira 3 XMU, Tescan) at 500x and 3000x magnifications.

Statistical Analysis

Statistical analyses were performed using SPSS version 19 software (SPSS Inc, Chicago, IL, USA). Distribution of data was assessed by the Kolmogorov-Smirnov test and normal distribution of data was confirmed. Color change and water sorbtion values were analyzed separately by repeated measures analysis of variance (ANOVA) and pairwise comparisons were performed with Bonferroni tests ($p<0.05$). Water solubility data was analyzed using one-way ANOVA and Tukey's HSD post-hoc tests ($p<0.05$). Pearson's correlation coefficient was used to assess the relationship between water sorption and solubility after 28 d ($\alpha = 0.05$).

Table 1: Compositions and manufacturer details of the tested restorative materials.

Restorative material	Material category	Composition	Application Procedure	Manufacturer
Filtek Z250 (Z250)	Micro-hybrid Resin Composite	Bis-GMA, Bis-EMA, UDMA, PEGDMA, TEGDMA, Zirconia silica, silica filler: 81.8% (wt)	Apply the resin composite in 2 mm layers and light cure for 20 s	3M ESPE, St Paul, MN, USA
Filtek One Bulk Fill (FOB)	Bulk-Fill Resin Composite	AUDMA, AFM, diurethane-DMA, and 1, 12-dodecane- DMA, ytterbium trifluoride, zirconia/silica 76%(wt)	Apply the resin composite in 2-4 mm layers, and light cure for 20 s	3M ESPE, St Paul, MN, USA
Estelite Bulk Fill Flow (EBF)	Bulk-fill Resin Composite	Bis-GMA, TEGDMA, Bis-MPEPP, mequinol, dibutyl hydroxyl toluene, uv adsorber, silicon oxide, zirconium oxide 70% (wt)	Apply the resin composite in 2-4 mm layers, and light cure for 10 s	Tokuyama Dental Corporation, Tokyo, Japan
Equia Forte Fil (Equia FF)	High-viscosity glass ionomer	Powder: fluoro-alumino-silicate glass, polyacrylic acid powder, pigment Liquid: polyacrylic acid, distilled water, polybasic carboxylic acid	Activate the Equia Forte Fil capsule and place it into a mixer (or an amalgamator), and mix for 10 s. Remove the capsule and load it into the GC capsule applier. Apply the material in the cavity, condensed and sculpted and then left to set for 2.5 minutes. After the setting, the surface coating (Equia Coat, GC) was applied on Equia Forte Fil specimens and light polymerised for 20 s.	GC Dental Products Corp, Tokyo, Japan
Cention N self cure (CN-Self cure)	Alkasite restorative	Liquid-UDMA resin, DCP, an aromatic aliphatic-UDMA and PEG-400 DMA. Powder-Ytterbium trifluoride, isofiller, fluorosilicate glass fillers	Manually mix two measuring spoons of powder and two drops of resin till a smooth consistency. The mixing time should not exceed 60 seconds. Leave the material for 10 minutes from the start of mixing (no light curing)	Ivoclar Vivadent AG, Liechtenstein
CentionN light cure (CN-Light cure)	Alkasite restorative	Liquid-UDMA resin, DCP, an aromatic aliphatic-UDMA and PEG-400 DMA. Powder-Ytterbium trifluoride, isofiller, fluorosilicate glass fillers	Manually mix two measuring spoons of powder and two drops of resin till a smooth consistency. The mixing time should not exceed 60 seconds and light cure for 20 s.	Ivoclar Vivadent AG, Liechtenstein
Gradia Plus (Gradia P)	Indirect Micro-hybrid Resin Composite	Bis-GMA, TEDGMA, UDMA, ceramic filler, Photoinitiators, Stabilisers, Pigments 71%(wt)	Apply the composite and light cure for 20 seconds. After that, a light-curing oven was used for further polymerization of the specimens for 3 minutes.	GC Dental Products Corp, Tokyo, Japan

Bis-GMA: bisphenol A diglycidyl methacrylate; Bis-EMA: bisphenol A diglycidyl methacrylate ethoxylated; TEGDMA: triethylene glycol dimethacrylate, UDMA: urethane dimethacrylate; PEGDMA: poly (ethylene glycol) dimethacrylate; AUDMA: Aromatic urethane dimethacrylate; Bis-MPEPP; Bisphenol A polyethoxy dimethacrylate; AFM: addition- fragmentation monomer; DCP: dicalcium phosphate

Table 2: Water sorption after 7 days and 30 days. and water solubility results

Material	Water sorption 7th days	Water sorption 28th days	Solubility
Z250	1.59 ± 0.49^{aA}	4.25 ± 0.78^{aB}	0.59 ± 0.13^{a}
FOB	3.2 ± 0.74^{abA}	6.5 ± 0.74^{abB}	0.97 ± 0.49^{ab}
Gradia P	2.13 ± 0.67^{a}	3.81 ± 0.58^{a}	0.59 ± 0.22^{a}
EBF	7.04 ± 2.56^{bA}	9.6 ± 2.52^{bB}	0.94 ± 0.27^{ab}
Equia FF	29.68 ± 6.64^{cA}	34.16 ± 5.6^{cB}	12.74 ± 2.7^{d}
CN-light-cure	2.64 ± 0.92^{a}	4.28 ± 1.09^{a}	2.31 ± 0.86^{bc}
CN-self-cure	3.34 ± 0.99^{ab}	4.35 ± 0.69^{a}	2.75 ± 1.33^{c}

*The superscripts lower cases indicate significant differences among restorative materials, the superscript uppercases indicate significant difference between the water sorption of restorative materials in different time periods ($p<0.05$).

Table 3: Color change for restorative materials.

Material	T1-T2	T1-T3
Z250	1.7 ± 0.3^{bcA}	2.8 ± 0.44^{abcB}
FOB	1.36 ± 0.18^{bA}	2.95 ± 0.29^{abcB}
Gradia P	1.35 ± 0.24^{bA}	3.29 ± 0.57^{cB}
EBF	1.6 ± 0.33^{bcA}	3.24 ± 0.47^{bcB}
Equia FF	1.93 ± 0.25^{cdA}	2.54 ± 0.33^{aB}
CN-light-cure	2.12 ± 0.2^{dA}	2.61 ± 0.19^{abB}
CN-self-cure	0.8 ± 0.4^{aA}	2.35 ± 0.68^{aB}

*The superscripts lower cases indicate significant differences among restorative materials, the superscript uppercases indicate significant difference between the color change of restorative materials in different time periods ($p<0.05$).

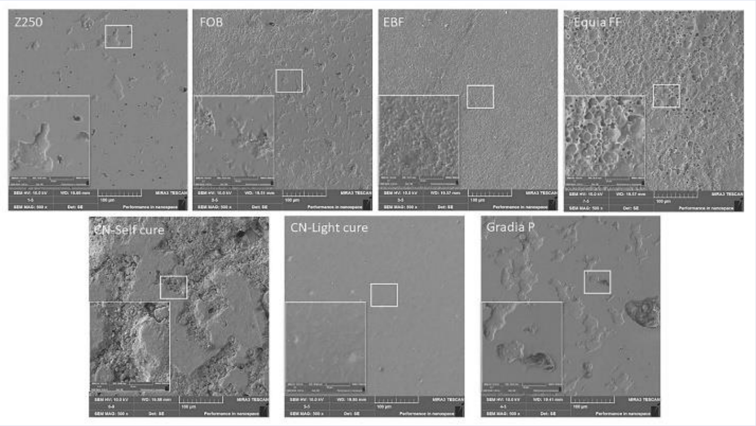

Figure 1. FE-SEM images of the restorative materials after 28-day immersion.

Results

Water Sorption and Solubility

Water sorption after 7 days and 28 days, and water solubility results of the tested restorative material groups are presented in Table 2**.** The water sorption values of the restorative materials were increased over time. The increase between 7 days to baseline and 28 days to baseline were significant for Z250, FOB, EBF and Equia FF groups ($p<0.05$).

After 7 days, Equia FF had the significantly highest water sorption values and, EBF showed the highest water sorption among the composite resins ($p<0.05$). Light-cure and self-cure CN groups demonstrated similar water sorption with Z250, Gradia P and FOB ($p>0.05$). After 28 days, Equia FF exhibited the highest water sorption values and created a statistically significant difference compared to the other restorative materials tested in the study ($p<0.05$). The lowest water sorption value was found in Gradia P and was statistically significant from EBF and Equia FF groups ($p<0.05$). The differences between light-cured and self-cured CN groups and resin composite groups were not significant ($p>0.05$).

Regarding water solubility, the lowest values were observed in Z250 and Gradia P groups. Equia FF showed more solubility compared to Z250 ($p<0.05$). No statistically significant difference was found among the composite resin based restorastives ($p>0.05$).

In addition, a statistically significant and strong positive correlation was found between water solubility and 28-day water sorption (r=0. 905, $p<0.001$), whereas 28-day water sorption and ΔE_{00} presented a negligible negative correlation (r= -0.164, $p>0.05$).

Color Stability

The mean color differences (ΔE_{00}) of restorative materials and standard deviations for 24 h and 28 days after polymerization compared to baseline measurements are presented in Table 3. The ΔE_{00} values were significantly higher for all restorative materials after 28 day immersion in distilled water compared to 1 day immersion ($p<0.05$).

After 24 h, self-cured CN group showed the lowest ΔE_{00} and the difference was significant for all the comparisons ($p<0.05$). However, light-cured CN group showed the highest ΔE_{00} and the difference was not significant only when compared to Equia FF ($p>0.05$).

After 28 days, the lowest ΔE_{00} was observed in self-cured CN group and differences between self-cured CN and, EBF and Gradia P were significant ($p<0.05$). Gradia P showed the highest ΔE_{00} and differences were not significant when compared to other composite resins ($p>0.05$).

FE-SEM observation

The FE-SEM images of the samples are represented in Figure 1. For the Equia FF, a rough surface with intensive porosities were observed. Composite resin based restorative materials demonstrated shallow porosities and rough surfaces except for EBF, which demonstrated a smoother surface. Similar smooth surface was observed in CN-light cure group whereas CN-self cure group demonstrated distinct irregularities.

Discussion

The color of restorative material should be stable after the placement, and restorative materials should be highly resistant to water sorption and solubility. In the present study, different types of restorative materials that can be placed into the cavities in bulk were evaluated regarding water sorption, solubility and color stability characteristics. According to the results of this study, significant differences in color change, water sorption and solubility among the restorative materials were observed after 28-day immersion in distilled water. Therefore, the null hypothesis were rejected.

Water sorption and solubility of resin based restorative materials is of high importance due to the exerting deleterious effects on material structure and adverse effects on the health.[3] In this study, Equia FF showed higher water sorption and solubility compared to resin based materials in accordance with the previous studies [6, 15] and intensive porosities were observed on the FE-SEM images. These can be considered harmless for the health due to it has been revealed that the aluminum release of glass-ionomer cements is negligible to create health hazard.[16] However, the resin content of surface protector (Equia Forte Coat) that reduce water sorption and solubility of the glass ionomer restorative[15] should be kept in mind.

Hydrophilic properties of the resin matrix and filler content of composite resins have significant influence on the water sorption and solubility chracteristics.[4] In accordance with a previous study[17] that tested the water sorption of flowable bulk-fill restorative materials, in the present study the flowable bulk-fill restorative; EBF, showed the highest water sorption and solubility among composite resins. Despite the similar filler rate of EBF with Gradia P and FOB, triethylene glycol dimethacrylate (TEGDMA) content of EBF could be blamed for the higher water sorption and solubility values. TEGDMA has a lower molecular weight and higher hydrophilicity than bisphenol A-glicidil methacrylate (Bis-GMA) and urethane dimethacriylate (UDMA).[3,8] In addition, heterogenic structure of TEGDMA forms micro-pores between the polymers which contributes the higher water sorption.[8] Tanthanuch *et al.*[18] evaluated the degradability of bulk-fill composite resins and explained the lowest sorption of FOB by UDMA and ethoxylatedbisphenol-A dimethacrylate (Bis-EMA) content of the material, which absorbs less water than Bis-GMA.[19] In addition, Melo *et al.*[20] reported similar water sorption for the regular viscosity bulk-fill composite resin and traditional composite resins. In line with previous studies, in the present study FOB showed lower sorption and solubility than the flowable bulk-fill restorative (EBF) and similar values with other resin based and, light-cured and self-cured CN groups.

Indirect composite resins has a longer polymerization time when compared to direct restoratives, thus the higher degree of conversion rate can be expected to result in higher stability against the deterioration process.[10,21] However, in accordance with the results of present study, Iskender *et al.*[11] reported similar water sorption for indirect and direct composite resins. As aforementioned, the TEGDMA content of Gradia P could be responsible for the similar water sorption with direct composite resins, in spite of additional polymerization of indirect composite resin.

The most interesting finding of this study was that the light-cured and self-cured CN groups demonstrated similar water sorption to the resin based materials except of EBF, which is presented higher sorption. Cention N includes a hydrophilic liquid monomer (PEG-400 DMA) in content and capable of releasing fluoride, calcium and hydroxide ions. This could be the reason for distinct irregularities observed in CN-self-cure groups on FE-SEM images. However, CN does not contain Bis-GMA, HEMA or TEGDMA. In accordance with our results, de Araújo-Neto *et al.*[22] reported comparable water sorption for CN with bulk-fill and conventional composite resins. On the other hand, self-cured CN showed higher solubility than composite resins. Resin structure of CN includes mainly low-viscosity monomers and dual-cure initiators, and requires hand mixing. Therefore, more porous resin structure compared to one-paste composite resin sytems[22] can be responsible for the higher solubility.

Color stability of a restorative material is an important esthetic parameter for the long-term evaluation of the restorations. However, all tested restorative materials showed moderately unacceptable ΔE_{00} values (ΔE_{00}; >1.8, ≤3.6) after 28 day immersion in distilled water, according to the classification by Paravina *et al.*[23] The color change of restorative materials in distilled water was defined as intrinsic discoloration that is considered as the consequence of alteration of the interface between matrix and filler and the resin matrix, oxidation of the resin matrix and unreacted monomers.[24] Therefore, lower ΔE_{00} values of self-cured CN could be explained by the presence of thiocarbamide that improves the color stability of the material, instead of amine. Similar or lower color change was reported for bulk-fill composite resins in a previous study[25] and Barutçugil *et al.*[14] stated that color change of bulk-fill restoratives is material dependent due to the different contents. Accordingly, in the present study the difference between resin based bul-fill restoratives (EBF and FOB) and conventional composite resin was not significant.

In this study restorative materials were immersed in water for a period of time, however in oral environment, restorations exposed to various types of physical, mechanical and chemical stresses. In addition, restorations exposed to different colorants from beverages and foods within the dynamic cycle of oral hygiene applications. Therefore, these can be considered as the limitations of this study. Further studies incorporating the other intraoral conditions should be performed for recent restorative materials.

Conclusions

Considering the limitations of this study, the following conclusions can be made;

1- Light-cured and self-cured Cention N groups demonstrated similar water sorption to the conventional composite resin, regular bulk-fill restorative and indirect composite resin. Self-cured Cention N demonstrated higher water solubility than all composite resins.
2- Flowable bulk-fill restorative (EBF) showed higher water sorption than the light-cured and self-cured Cention N groups and other composite resins tested. The highest water sorption and solubility was observed in Equia Forte Fil.
3- All tested restorative materials showed moderately unacceptable color change values after 28 day immersion in distilled water.

Conflict of Interest

There are no conflicts of interest.

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