The Journal of Turkish Dental Research
Türk Diş Hekimliği Araştırma Dergisi

e-ISSN: 2822-4310, Cilt 3, Sayı 3, Eylül - Aralık 2024
Volume 3, Number 3, September - December 2024

Comparative Evaluation of Water Absorption, Solubility, Degree of Conversion, and Color Stability in Ormocer-Based and Flowable Bulk-Fill Composites

Ormocer Bazlı ve Akışkan Bulk-Fill Kompozitlerde Su Emilimi, Çözünürlük, Dönüşüm Derecesi ve Renk Stabilitesinin Karşılaştırmalı Değerlendirmesi

Water Absorption, Solubility, and Color Stability of Flowable Bulk-Fill and Ormocer-Based Composites

Betül Kübra KURUCU KARADENİZ[1], Seyit Bilal ÖZDEMİR[2], Büşra ÖZDEMİR[3]

[1]Asst. Prof. Giresun University, Faculty of Dentistry, Department of Restorative Dentistry, Giresun, Turkey
kurucubetulkubra@hotmail.com
ORCID: 0000-0002-4037-7852

[2]Asst. Prof. Giresun University, Faculty of Dentistry, Department of Restorative Dentistry, Giresun, Turkey
seyitbilalo@gmail.com
ORCID: 0000-0001-6303-1961

[3]Asst. Prof. Giresun University, Faculty of Dentistry, Department of Restorative Dentistry, Giresun, Turkey
busrakbdy91@gmail.com
ORCID: 0000-0002-7035-3341

Makale Bilgisi / Article Information
Makale Türü / Article Types: Araştırma Makalesi / Research Article
Geliş Tarihi / Received: 23-09-2024
Kabul Tarihi / Accepted: 11-12-2024
Yıl / Year: 2024 | **Cilt – Volume:** 3 | **Sayı – Issue:** 3 |**Sayfa / Pages:** 409-420

Sorumlu Yazar / Corresponding Author: Betül Kübra KURUCU KARADENİZ

https://doi.org/10.58711/turkishjdentres.vi.1555054

Comparative Evaluation of Water Absorption, Solubility, Degree of Conversion, and Color Stability in Ormocer-Based and Flowable Bulk-Fill Composites

Ormocer Bazlı ve Akışkan Bulk-Fill Kompozitlerde Su Emilimi, Çözünürlük, Dönüşüm Derecesi ve Renk Stabilitesinin Karşılaştırmalı Değerlendirmesi

ÖZET

Amaç: *Bu çalışma, akışkan bulk-fill kompozitler ve ormocer bazlı reçinenin su emilimi, çözünürlüğü, dönüşüm derecesi ve renk stabilitesini değerlendirmeyi amaçlamaktadır.*

Materyal ve Yöntem: *Beş bulk-fill kompozit (SDR, Beautifil Bulk, Omnichroma Flow Bulk, Venus Bulk Fill, Charisma Bulk Flow One) ve iki kompozit (Filtek Z250, Admira Fusion 5) test edildi (n=40). Renk ve kütle ölçümleri başlangıçta, 24 saat sonra ve 28 gün sonra yapıldı. İstatistiksel analizler IBM SPSS V23 ve WRS2 paketiyle R programı kullanılarak gerçekleştirildi. Non-normal dağılımlı veriler Kruskal-Wallis H testi ve Dunn testi kullanılarak post-hoc karşılaştırmalar için analiz edildi. Grup ve zaman karşılaştırmaları için Robust ANOVA ile Benforrini düzeltme kullanıldı. Anlamlılık düzeyi p < 0,050 olarak belirlendi.*

Bulgular: *Charisma Bulk Flow en yüksek çözünürlüğe sahipken, Admira Fusion 5 en düşük çözünürlüğe sahipti (p=0.02). Dönüşüm derecesi ve renk stabilitesi arasında anlamlı farklılıklar bulunmuştur (p<0.001).*

Sonuç: *Test edilen kompozit malzemeler, monomer bileşimleri ve dolgu içeriklerinden kaynaklanan su emilimi, çözünürlük, dönüşüm derecesi ve renk kararlılığı açısından önemli farklılıklar gösterdi. Admira Fusion 5, en yüksek dönüşüm derecesinin yanı sıra en düşük su emilimi ve çözünürlük ile üstün klinik performans potansiyeli olduğunu gösterdi. Buna karşılık, SDR ve Venus Bulk Fill, zaman içinde en yüksek renk değişimlerini göstererek, estetik restorasyonlarda malzeme seçiminin önemine işaret etti. Bu bulgular, kompozitlerin fiziksel ve optik özelliklerinin tayininde malzeme kompozisyonunun önemini vurgulamaktadır.*

Anahtar Kelimeler: *Ormocer Kompozitler; Bulk-Fill Kompozitler; Su Emilimi; Çözünürlük; Dönüşüm Derecesi; Renk Stabilitesi*

ABSTRACT

Objective: *This study aimed to evaluate the water absorption, solubility, degree of conversion, and color stability of flowable bulk-fill composites and ormocer-based resin.*

Materials and Methods: *Five flowable bulk-fill composites (SDR, Beautifil Bulk, Omnichroma Flow Bulk, Venus Bulk Fill, Charisma Bulk Flow One) and two composites (Filtek Z250, Admira Fusion 5) were tested (n=40). Color and mass measurements were taken initially, after 24 hours, and after 28 days. Statistical analyses were performed using IBM SPSS V23 and the R program with the WRS2 package. Non-normally distributed data were analyzed using the Kruskal-Wallis H test and Dunn test for post-hoc comparisons, while robust ANOVA with Bonferroni correction was used for group and time comparisons. The significance level was set at p < 0.050.*

Results: *Charisma Bulk Flow had the highest solubility, Admira Fusion 5 the lowest (p=0.02). Degree of conversion and color stability showed significant differences (p<0.001).*

Conclusion: *The tested composite materials showed significant differences in water absorption, solubility, degree of conversion, and color stability, influenced by their monomer composition and filler content. Admira Fusion 5 exhibited the lowest water absorption and solubility alongside the highest degree of conversion, indicating its potential for superior clinical performance. In contrast, SDR and Venus Bulk Fill demonstrated the highest color changes over time, underscoring the importance of material selection for aesthetic restorations. These findings highlight the critical role of material composition in determining the physical and optical properties of dental composites.*

Keywords: *Ormocer Composites; Bulk-Fill Composites; Water Sorption; Solubility; Degree of Conversion; Color Stability*

Introduction

The application of composite resin in 2 mm layers has been considered the gold standard as it allows for complete polymerization of the material, increases light penetration, and reduces polymerization shrinkage. However, this technique has disadvantages such as being time-consuming, risk of creating voids between layers, requiring technical precision, and posing a risk of contamination. To overcome these disadvantages, manufacturers have developed materials known as bulk-fill composite resins, which can be applied in a single layer of 4-5 mm thickness. This facilitates the application of composite resin in large cavities and reduces the time spent on patient treatment.[1] In bulk-fill composites, the filler content and pigment amount have been reduced while the particle content has been increased to achieve a more translucent structure. This increased translucency, coupled with new monomer and initiator systems, allows these composites to have greater depth of polymerization.[2]

To achieve excellent aesthetics, dental restorative materials must not only be resistant to surface discoloration but also maintain their intrinsic color stability.[3] Therefore, in composite resin discoloration, surface properties as well as the resin's affinity to extrinsic coloring agents, polymerization degree of conversion, and water absorption properties are also important.[4]

Composite resins, despite their advantages such as aesthetics and ease of shaping, present significant clinical problems such as water absorption and solubility. High water absorption and solubility can lead to dimensional changes in the material. Additionally, aesthetic and hygienic issues such as discoloration, loss of marginal integrity, and disruption of compatibility with biological structures can occur in restorations. These conditions weaken the physical and chemical properties of composite resins. Moreover, moisture in the oral environment, as well as hydrolysis and enzymatic reactions, can cause deterioration and erosion on the surface of composite resins.[5] Water absorption in materials leads to dimensional changes, resulting in discoloration and fractures at the edges of restorations. On the other hand, water solubility increases the chemical dissolution of restorations, negatively affecting their compatibility with biological structures. As a result, water absorption and solubility values are important parameters for both the integrity and mechanical properties of restorations, as well as their surface characteristics and aesthetic appearance.[6,7] Complete polymerization of composite resins is a crucial factor that influences various physical properties such as mechanical characteristics, solubility, dimensional stability, color change, and biocompatibility of the material.[8] Determining the degree of polymerization is important in evaluating the success of composite restorations. Sufficient polymerization reduces the cytotoxicity of dimethacrylate-based composites and enhances their physical properties.[9,10] Filling particle size and quantity, concentration of polymerization initiators, monomer type[11] and quantity, material color, and translucency[12] , wavelength and intensity of the light source, and light curing time[13] various factors such as the wavelength and intensity of the light source, and light curing time can affect the degree of polymerization of dental composite materials.

The aim of this in vitro study was to comparatively evaluate the water absorption, solubility, degree of conversion, and color stability over time of different flowable bulk-fill composites and ormocer-based resin composite, which are increasingly used in clinical practice.

The null hypothesis of the study is that the water absorption, solubility, degree of conversion, and color changes among the tested composite groups are not significantly affected by the differences in their monomer types and filler compositions.

Materials and Methods

ISO 4049 standards were strictly followed in preparing the samples, and the specimen dimensions and light-curing parameters were adjusted accordingly. The light-curing device was used with an 8 mm diameter tip to ensure uniform light distribution for homogenous polymerization. Mass measurements were also carried out with high-precision scales in compliance with ISO 4049 standards.

In this study, six different composite resins were used: Filtek Z250, Beautifil Bulk Flowable, SDR, Admira Fusion 5, Omnichroma Flow Bulk, Charisma Bulk Flow One, and Venus Bulk Fill (Table 1). A total of 40 samples were prepared for each material. Samples were formed

https://doi.org/10.58711/turkishjdentres.vi.1555054

Table I. Compositions and manufacturer details of the tested restorative materials.

Restorative material	Material category	Composition	Manufacturer
Filtek Z250 (Z250) Shade A2	Micro- hybrid Resin Composite	Shade: A2 Bis-GMA, Bis-EMA, UDMA, PEGDMA, TEGDMA, Zirconia silica, silica filler: 81.8% (wt)	3M ESPE, StPaul, MN, USA
SDR Shade universal	Bulk-Fill flowable composite	Bis-EMA, modified UDMA, TEGDMA, Barium-aluminum-fluorosilicate glass, (wt.%/vol.%) 68/45	3M ESPE, Dental Products, Saint Paul, MN, USA
Omnichroma Flow Bulk (OFB) Shade universal	Bulk-Fill flowable composite	(wt.%/vol.%) 69/55 Spherical silica zirconia filler, 1,6-bis UDMA,TEGDMA,Mequinol, Dibutyl,hydroxyl toluene	Tokuyama Dental (Tokyo, Japan)
Beautiful Bulk Flowable (BBF) Shade universal	Giomer Bulk-Fill flowable composite	Shade: Universal Bis-GMA, UDMA, Bis-MPEPP, TEGDMA, S-PRG fillerbased on fluoroboroaluminosilicate glass	Shofu,Japan
Venüs Bulk Fill(VBF) Shade universal	Bulk-Fill flowable composite	Shade: Universal UDMA,EBADMA, %65 w/w and %38 vol inorganic fillersBa-Al-F silicate glass,YbF_3, SiO_2	Kulzer GmbH, Germany
Charisma Bulk Flow One(CBF) Shade universal	Bulk-Fill flowable composite	UDMA, EBADMA, 65 % by weight, or 41 % by volume inor- ganic fillers such as Ba-AI-F silicate glass, YbF_3 and SiO_2.	Kulzer GmbH, Germany
Admira Fusion 5 (AF5) Shade A2	Universal nano-hybrid ORMOCER restorative material	Shade:A2 %83 weight inorganic fillers, Barium aluminium borosilicate glass, OR-MOCER® resin, silicon dioxide, initiators, stabilisers, pigments	Voco, Germany

using special stainless steel molds with a diameter of 8 mm and a thickness of 2 mm. The sample size was determined according to the light device tip diameter to ensure even light distribution during the polymerization process.

The composites were placed into the molds. The molds that contain composite were placed on 5 mm thick glass and transparent cellulose acetate strips. A force of 500 grams was applied by placing a second transparent strip and glass on top. Light-cured materials were polymerized using a LED light device (Bluephase G2, Ivoclar Vivadent) for the manufacturer-recommended times. The light device was set to 1200 mW/cm²intensity and applied to each sample for 40 seconds. The light intensity was regularly monitored with a Curing Radiometer during the polymerization process.

After polymerization, the samples were removed from the molds, and the surface exposed to light during polymerization was polished using aluminum oxide (Al_2O_3) impregnated discs. For the polishing procedure, Sof-Lex™ (3M ESPE) discs were used in sequence: coarse, medium, fine, and super fine grit, with 10 seconds of polishing for each disc. Polishing was not applied to the opposite surface of the composite samples and the polishing discs were renewed for each sample.

Determination of Conversion Degree

The FTIR-ATR spectra of the composite materials were obtained using a Perkin Elmer SpectrumTwo FTIR spectrophotometer, which measures in the range of 4000-600 cm^-1. Samples were stored in light-tight boxes before analysis. Initially, spectral measurements were taken for each unpolymerized composite resin. The bottom surface of each polymerized sample to be measured was placed in contact with the ATR crystal, and the compression arm of the device was closed. Spectral measurements were then taken for each sample in this manner. The percentage degree of conversion (%DC) values were calculated by substituting the obtained values into the formula below.

Formula used in the study:

Degree of conversion (%DC): %DC=1 - (AC=C/ AC−C) Polymer / (AC=C/AC−C) Monomer X 100

In this formula, AC=CAC=C represents the absorbance of carbon-carbon double bonds, and AC−CAC−C represents the absorbance of a stable reference bond. Measurements for monomer and polymer samples were normalized using this ratio, providing a reliable measure of polymerization efficiency.

Water Sorption and Solubility

After polymerization, the samples were placed in a glass vacuum desiccator at 37±1 °C for 22 hours, then transferred to a second desiccator at 23±1 °C for 2 hours. Samples were weighed using a digital balance (accuracy 0.01 g) until a constant mass (within ± 0.1 mg) was achieved, recorded as m1. The samples were immersed in distilled water at 37±1°C for 28 days. They were weighed after 1, 7, and 28 days (m2), dried again using the same protocol, and final mass values (m3) were recorded once constant mass was achieved.The water absorption (WSP) and water solubility (WSL) of the each sample were calculated separately with the following equations:[14]

Formula used in the study:

Water sorption (WSP): WSP = (m2 - m1) / V (mg/mm^3)

Water solubility (WSL): WSL = (m1 - m3) / V (mg/mm^3)

Where m1, m2, m3 represent different mass measurements, and V represents the volume of the sample.

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 taken using a spectrophotometer (VITA Easyshade, Vita Zahnfabrik, Germany) under standard lighting conditions with a white background. Each measurement was repeated three times, and the average L, C, and H values were recorded based on the CIEDE2000 system. To determine color differences (ΔE00), baseline measurements (T1) were taken after specimen preparation, and repeated after 24 hours (T2) and 28 days (T3) of water immersion. The ΔE00 values were calculated separately for both T1-T2 and T1-T3 intervals to assess color stability.

The formula provides brightness, saturation, and hue values in order with ΔL*, ΔC*, and ΔH*. Rt represents the interaction between saturation and hue values. Kl, Kc, and Kh are parametric values taken as 1. The clinically acceptable threshold is set at 50%:50%, with ΔE00≥1.8.[15]

$$\Delta E_{00} = \left[\left(\frac{\Delta L'}{K_L S_L}\right)^2 + \left(\frac{\Delta C'}{K_C S_C}\right)^2 + \left(\frac{\Delta H'}{K_H S_H}\right)^2 + R_T \left(\frac{\Delta C'}{K_C S_C}\right)\left(\frac{\Delta H'}{K_H S_H}\right)\right]^{1/2}$$

Statistical Analysis

The data were analyzed using IBM SPSS V23 and R program with the WRS2 package. Normality of the data was assessed using the Shapiro-Wilk test. For data that did not follow a normal distribution in three or more groups, the Kruskal-Wallis H test was used followed by multiple comparisons using the Dunn test. For comparing data that did not follow a normal distribution across groups and time, the Robust ANOVA test was used, with multiple comparisons adjusted using the Bonferroni correction. Analysis results were presented as medians (min–max).The significance level was set at $p < 0.050$.

Results

The water absorption, degree of conversion, and solubility values of the tested materials are presented in Tables 2, Table 3. There is a statistically significant difference in water absorption values among the composites ($p < 0.001$). The highest water absorption was observed in BBF on the 7th day, while the highest absorption on the 28th day was in OFB. The lowest water absorption was recorded in AF5 on both the 7th and 28th days.

Similarly, there is a statistically significant difference in solubility values among the composites ($p < 0.001$).

The highest solubility was observed in CBF, while the lowest solubility was in AF5.

Regarding the degree of conversion, there is a statistically significant difference among the composites ($p < 0.001$). The highest degree of conversion was observed in AF5, while the lowest degree of conversion was in BBF.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 4.

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, OFB showed the lowest ΔE_{00} and the difference was significant for all the comparisons ($p<0.05$). SDR showed the highest ΔE_{00}. After 28 days, the lowest ΔE_{00} was observed in BBF and VBF showed the highest ΔE_{00}.

Discussion

Dentists increasingly need restorative materials that can be applied more quickly and easily compared to traditional composites, glass ionomers, and amalgam, while still possessing sufficient polymerization, mechanical, and

Table II. Water solubility results and degree of conversion values

Material	Water solubility	Degree of conversion
Z250	0.48 (0.34 – 0.59)^{d}	65.12 (64.08 – 66.89)^{cd}
SDR	3.19 (3 – 3.82)^{abc}	56.09 (56.01 – 56.34)^{ac}
AF5	0.26 (0.1 – 0.64)^{cd}	74.1 (74.01 – 74.64)^{c}
VBF	4.04 (2.76 – 4.26)^{ab}	47.23 (47.06 – 47.67)^{ab}
CBF	4.24 (4,02 – 4.67)^{b}	47.18 (46.26 – 47.58)^{ab}
BBF	2.16 (2,01 – 3.74)^{acd}	46.29 (46.02 – 46.71)^{b}
OFB	3.6 (3.01 – 4.1)^{ab}	47.83 (47.27 – 48.44)^{ad}
p*	**<0.001**	**<0.001**

a,b,c,d: Same columns different superscripts indicate statistically significant difference.(p<0.001)

Table III. Water sorption after 7 days and 30 days

Material	Water sorption 7th days	Water sorption 28th days
Z250	1.44 (1.14 – 1.86)^{A}	5.12 (4.09 – 6.59)^{B}
SDR	1.2 (1 – 1.47)^{AC}	6.07 (6 – 6.26)^{D}
AF5	0.94 (0.56 – 1.04)^{C}	4.09 (4 – 4.64)^{E}
VBF	2.4 (2.13 – 2.7)^{F}	7.23 (7.06 – 7.68)^{G}
CBF	2.72 (2.45 – 3.07)^{F}	7.19 (6.26 – 7.58)^{G}
BBF	4.24 (4.02 – 4.67)^{E}	6.29 (6.02 – 6.71)^{D}
OFB	2.16 (2.01 – 3.74)^{AF}	7.83 (7.27 – 8.44)^{H}

A, B, C, D, E, F, G, H: Same columns different superscripts indicate statistically significant difference. (p<0.001)

Table V. Color change for restorative materials.

Material	T1-T2	T1-T3
Z250	1.48 (1 – 1.83)^{A}	3.5 (3.24 – 4.88)^{BC}
SDR	2.44 (1.01 – 3.35)^{ABDEFG}	3.27 (2.16 – 3.73)^{BCD}
AF5	1.61 (1.05 – 1.87)^{AE}	2.6 (2.35 – 2.95)^{F}
VBF	1.97 (1.06 – 2.56)^{ADEFG}	4.62 (4.25 – 6.61)^{C}
CBF	1.47 (1.06 – 1.77)^{AE}	2.68 (2.03 – 4.4)^{F}
BBF	1.39 (1.09 – 2.97)^{AEG}	2.35 (2.15 – 2.85)^{FG}
OFB	1.13 (1.01 – 1.55)^{E}	2.55 (2.21 - 3)^{DF}

A, B, C, D, E, F, G, H: Same columns different superscripts indicate statistically significant difference. (p<0.001) physical properties comparable to these materials.

In this study, the comparative examination of water absorption, solubility, degree of conversion, and color changes of bulk-fill composites and ormocer-based resin, which are becoming more commonly used in clinical practice, was aimed. In this study, a null hypothesis was established based on the assumption that water absorption, solubility, degree of conversion, and color changes among the tested composite groups are not significantly affected by differences in monomer types and filler compositions. However, the findings obtained from the study led to the rejection of this hypothesis. This indicates that the structural differences in composite materials have a significant impact on their physical and chemical properties.

After the polymerization reaction of monomers, numerous residual monomers remain in the polymerized material. The degree of conversion (DC) typically represents the degree of polymerization of the composite resin. It is the percentage of polymerizable double bonds converted to single bonds. The DC value of composite resin ranges from 52% to 75%.[16,17] The DC significantly influences the mechanical and physical properties of the material. According to our study results, the highest degree of conversion was observed in AF5, while the lowest was found in BBF. The Ormocer structure facilitates the development of an inorganic siloxane (Si–O–Si) network through hydrolysis and polycondensation processes. This process forms a durable inorganic ceramic polysiloxane matrix with attached organic side units, which can participate in conventional light-induced polymerization.[18]The presence of multiple polymerizable organic units in Ormocers enhances the likelihood of interactions and chemical bonding with neighboring molecules, thereby increasing the degree of cross-linking and monomer conversion. In contrast, the Bis-GMA molecule found in nanohybrid composites contains only two polymerizable units, limiting its cross-linking potential.[19] These structural differences provide Ormocer-based composites, such as AF5, with superior polymerization efficiency and higher cross-link density. The increased cross-linking capacity of the Ormocer matrix promotes a more compact and homogenous polymer network, which reduces the amount of residual monomers and enhances the material's physical and mechanical properties. This explains why AF5 showed the highest degree of conversion (DC) in this study. In a study, it was determined that BBF composite had a lower degree of conversion (DC) compared to other bulk-fill and conventional giomer composites.[20] These findings are consistent with the results of our study.

In our study, although 2 mm thick samples were prepared, the DC of BBF was found to be lower than that of other composites. One of the main reasons for this is the limited number of polymerizable units in the matrix structure of BBF. While ormocer-based structures have a higher number of polymerizable units, the monomers in BBF contain a more limited number of polymerizable units. This reduces the cross-linking capacity and decreases the degree of conversion.

Another reason is the high filler content and large particle size, which restrict light transmission. Although the samples had a thickness of only 2 mm, the inability of light to sufficiently penetrate the inner regions of the sample caused polymerization to be effective only in areas close to the surface. Similarly, in the study,[20] it was reported that bulk-fill composites with a high filler content exhibited lower DC values.

Additionally, the steric hindrance effect is another reason for the low DC observed in BBF. The dense packing of filler particles prevents the reactive double bonds from participating in the polymerization process. This can result in a lower degree of conversion, even in 2 mm thick samples.[20]

Borges et al. [21] examined the spectra of C=C double bonds of different composite resins and reported differences in the spectra. They suggested that these differences could be related to the structural and/or geometrical diversity of composite resins, and that the conversion fraction of the material is directly proportional to the peak intensity or area. In our study, we determined that the C=C double bonds peaked at 1717 cm^-1 and the C-C bonds at 1509 cm^-1, and these absorption values at these wavelengths were used. The values obtained in our study were used to calculate the polymerization percentages by substituting them into the formula to determine the conversion values. According to the results of our study, the obtained values indicate that all samples

https://doi.org/10.58711/turkishjdentres.vi.1555054

in all groups were adequately polymerized.

Guimaraes et al.[21] investigated the polymerization degree of samples with different thicknesses of Venus, SDR, and traditional flowable composites using FTIR analysis. They reported that the polymerization depth was satisfactory in samples with thicknesses of up to 6 mm in bulk-fill composites. In our study, the AF5 restorative material exhibited higher polymerization percentages compared to other bulk-fill composite resins. Additionally, factors affecting light transmission (such as the thickness of the restorative material and the distance of the light tip to the restoration surface) were standardized across all groups. Any differences in degree of conversions in this study may stem from variations in the composition of the materials.

Faria et al.[22] reported that light penetration to the underlying layers is more challenging in darker shade composite resins compared to lighter shades, which can negatively affect polymerization.Similarly, in a study by Koupis et al.[23] A2 and A4 shade composite resins were examined, and it was reported that the polymerization depth was greater for the A2 shade compared to the A4 shade. This situation may explain why Filtek Z250 exhibited a lower degree of conversion (DC) compared to AF5, despite AF5 having a higher DC in our study.

ISO 4049 is one of the widely recognized standards used to determine the water absorption and solubility values of composite materials. According to ISO 4049:2009 standards, the water absorption value of materials should be equal to or less than 40 µg/mm^3, and the solubility should be equal to or less than 7.5 µg/mm^3.[24] All composite materials demonstrated water absorption of less than 40 µg/mm^3, complying with the ISO 4049 criteria. Similarly, all materials tested in this study exhibited solubility of less than 7.5 µg/mm3, meeting the ISO 4049 standards.

It has been emphasized that one of the crucial factors for the clinical success of composite resins is their dimensional stability in the oral cavity and low water absorption. Water absorption has been noted to negatively affect the physical and mechanical properties of composite resins by disrupting the connection between the inorganic filler particles and the organic matrix.[25,26] Water molecules can penetrate the structure of composite resin through three different mechanisms via diffusion. These include the gaps within the organic matrix, the gaps between the filler particles constituting the inorganic structure, and the interfacial bonding surfaces between the inorganic filler and the organic matrix.[27,28] One of the factors affecting the water absorption of composite resins is the type, amount, size, and shape of the filler particles in their structure.[29] Filler particles themselves generally do not absorb water molecules. However, over time, water diffuses to the interface between the filler and the organic matrix and reacts with the filler. In this process, both the structure of the filler particles and the structure of the silane at the interface between the inorganic and organic matrices are important.[30] In their study investigating the water absorption and solubility of nanocomposites, Mirsasaani et al.[31] reported that composite resins with a higher amount of inorganic filler exhibited lower water absorption values. In their study examining the water absorption and solubility values of four traditional composite resins and one flowable composite resin, Wei et al.[40] found that the flowable composite resin exhibited higher water absorption values. They attributed this to the lower content of inorganic filler in the flowable composite compared to traditional composite resins.

Filler ratio is one of the most important factors affecting water absorption. Since Filtek Z250 and AF5 have a high filler ratio and large filler particles, it is difficult for water molecules to penetrate the polymer matrix in these materials. The higher the filler fraction, the lower the volume of the organic matrix and the less space is left for the passage of water. This finding is in agreement with the findings reported by Mirsasaani et al.[31] that composites with higher filler content have lower water absorption values.

In contrast, in flowable composites such as BBF, CBF and OFB, the filler ratio is lower, which increases the volume of the organic matrix, resulting in easier penetration of water molecules into the polymer network structure. This finding is in agreement with the data of Wei et al.[32], where flowable composites showed higher water absorption and this was attributed to the lower filler ratio.

DC is another important factor affecting water absorption. AF5 has a higher DC value due to its ormocer structure. High DC makes the polymer network have a tighter and denser structure. This tight network structure limits the number and size of voids through which water molecules can pass. This is consistent with AF5 showing the lowest water absorption in our study.

In contrast, DC values are lower in BBF and CBF. Low DC causes the polymer network to remain in a looser and irregular structure. This facilitates the passage of water molecules through the voids in the polymer network and increases water absorption. In the literature, Borges et al.[21] and Chaves et al.[33] reported that composites with low degree of polymerisation have higher water absorption due to more voids and low crosslinking capacity. The findings in our study coincide with these literature data.

The quality of the connection between the filler particles and the organic matrix is another factor that directly affects water absorption. This connection is usually achieved through silane coupling agents. However, defects or micro-gaps at this interface can cause water molecules to pass through the filler-matrix interface.

AF5 is an ormocer-based material and contains siloxane (Si-O-Si) linkages in its structure. These connections limit the passage of water molecules by reducing the gaps between the filler particles and the organic matrix. Therefore, the lowest values of water absorption of AF5 in our study can be attributed to this mechanism.

In flowable composites such as BBF and CBF, more voids may remain at the filler-matrix interface. These voids cause water molecules to pass through the filler-matrix interface and penetrate the polymer network structure. This finding is in line with the findings reported by Chaves et al.[33] that micro-voids at the filler-matrix interface cause water absorption. In this context, the higher water absorption values of BBF and CBF can be explained by defects at the filler-matrix interface.

Composite resins containing Bis-GMA typically exhibit water absorption ranging from 0% to 1%, whereas this rate can increase up to 3%-6% depending on the amount of TEGDMA added to the structure. The presence of recurring ethoxy groups in TEGDMA makes it sensitive to water molecules, leading to increased surface hydrophilicity of the resin. The high hydrophilicity of Bis-GMA and TEGDMA results in higher water absorption values. On the other hand, Bis-EMA, due to the hydrophobic group it contains, demonstrates lower water absorption values.[34] Pearson and Longman[35], reported that under normal polymerization conditions, UDMA-based materials exhibit lower water absorption levels compared to Bis-GMA-based materials. In a study conducted by Venz and Dickens[36] investigating monomer structures, it was reported that the hydrophilicity of monomers follows the order of TEGDMA > BisGMA > UDMA, with TEGDMA monomer exhibiting the highest affinity to water.

In our study, it is thought that the higher water absorption values of flowable composites (BBF, CBF, OFB) may be explained by the higher TEGDMA ratio in these composites. As reported by Venz and Dickens,36 TEGDMA interacts more easily with water molecules due to its high hydrophilicity and causes water penetration into the polymer network. In particular, it is known that TEGDMA is used at higher ratios in flowable composites in order to increase fluidity. This is consistent with the high water absorption of the flowable composites in our study.

In our study, the lower water absorption value of Filtek Z250 compared to BBF and CBF may be attributed to the absence of TEGDMA in this composite and the replacement of Bis-GMA with UDMA and Bis-EMA. This finding is in agreement with the findings of Pearson and Longman[35] that UDMA-based composites showed lower water absorption than Bis-GMA-based composites. The hydrophilic structure of Bis-GMA and the hydrogen bonding of -OH groups with water molecules is a factor that increases water absorption. However, in Filtek Z250, partial replacement of Bis-GMA with UDMA and Bis-EMA may have reduced water absorption.

In our study, the low water absorption of AF5 may be attributed to the effect of its ormoser and UDMA-based polymer structure. UDMA has a less hydrophilic structure compared to Bis-GMA and interacts less with water molecules.[35] This explains the water absorption findings in our study. In addition, the siloxane (Si-O-Si) linkages in the ormocer structure of AF5 limit the entry of water molecules into the polymer network.

Gönülol et al.[37] examined the relationship between water absorption and solubility and reported a positive correlation ($r=0.612$, $p<0.001$) between these parameters. Similarly, in our study, it was determined that the solubility values of composite groups with high water absorption were also high. This supports the hypothesis that the voids in the polymer network structure facilitate the release of soluble components by allowing the entry of water molecules. In particular, the higher TEGDMA content of BBF, CBF and OFB may increase solubility by forming hydrogen bonds with water.

Solubility in water can lead to deterioration in the physical properties of the material, such as reducing surface hardness and wear resistance, in addition to dimensional changes.[38] Furthermore, water absorption can adversely affect the color stability of the material by allowing water-soluble monomers to absorb into the restoration.[39] Therefore, water absorption and solubility can result in deterioration of the surface properties of composite restorations, leading to marginal integrity issues and loss of aesthetic appearance, which can significantly impact the clinical performance of composite materials.[7] Using standard parameters, color changes can be detected and the current colors can be objectively evaluated to a quantifiable level in color science, which aims to quantify colors. Spectrophotometers are among the most reliable color measurement devices used for the first stage of color quantification. In calculating color differences, CIELab and currently, CIEDE2000 are the most commonly used systems. Studies have shown that the CIEDE2000 color formula provides better compatibility in assessing translucency and color changes in dental materials in terms of perceptibility and acceptability compared to the CIELab formula.[40,41] Perceptibility refers to the detection of color difference between a tooth and an adjacent stained restoration, while acceptability defines the color difference that is acceptable for this restoration.[42] According to ISO/TR 28642:2016 standards, the threshold values for perceptibility and acceptability of color change are established as 0.8 and 1.8, respectively, as determined by Paravina et al. 15 in their study. In this study, in line with current literature, the CIEDE2000 formula was utilized for the detection of color changes.

The unacceptable discolouration of SDR and VBF in the T1-T2 range can be attributed to the high water absorption capacity of these composites. Chaves et al.[33] stated that water absorption penetrates the resin matrix and triggers colour change. In this context, the high water absorption values of SDR and VBF in our study may explain the unacceptable ΔE_{00} values observed in the T1-T2 range.

The unacceptable ΔE00 values of all composite groups in the T1-T3 range can be explained by the fact that prolonged exposure to water increases the solubility in the polymer matrix. Wei et al.[32] reported that the release of residual monomers caused colour change in composites exposed to water for a long time. This finding supports the high ΔE_{00} values observed in our study, especially in BBF, CBF and OFB.

In our study, all composites reached unacceptable ΔE_{00} values in the T1-T3 range, indicating that the colour stability of these materials may be insufficient for long-term restorations. This finding is in line with the data reported by Wei et al.[32] and Chaves et al.[33] that long-term water exposure increases discolouration. In this context, AF5 and Filtek Z250 stand out with their relatively lower ΔE_{00} values and can be considered as a more suitable option in aesthetic restorations.

Overall, existing in vitro studies support manufacturers' claims and endorse the use of bulk-fill composite resins. However, long-term in vivo studies are needed to support their clinical behavior. Due to variations among materials not always being fully disclosed by manufacturers and differences in methodology in in vitro studies, it is not possible to reach a common conclusion for all materials classified as bulk-fill. Therefore, until more data is available, clinicians are advised to carefully select materials and adhere to manufacturer instructions.

Conclusion

This study demonstrated that the performance of composite resins is significantly influenced by their monomer composition, filler content, and structural differences. Ormocer-based composites (AF5) exhibited superior properties, including low water absorption, low solubility, high polymerization degree (DC), and better color stability. These findings suggest that AF5 is a suitable material for long-term anterior restorations, where color stability and dimensional integrity are critical.

In contrast, flowable composites (BBF, CBF, OFB) showed higher water absorption and solubility, leading to increased susceptibility to discoloration over time. This suggests that these materials are more appropriate for temporary or posterior restorations, where aesthetic stability is less critical.

The findings of this study emphasize the need to select composite resins based on clinical requirements and the intended duration of the restoration. Ormocer-based and highly filled composites may be better suited for long-term aesthetic restorations, while flowable composites may be more appropriate for temporary restorations or non-load-bearing areas.

References

1. El-Damanhoury H, Platt J. Polymerization shrinkage stress kinetics and related properties of bulk-fill resin composites. Oper Dent. 2014;39(4):374-82.

2. Eakle WS, Bastin KG. Dental materials: clinical applications for dental assistants and dental hygienists: 4th Ed, Elsevier; 2020.

3. Bagheri R, Burrow M, Tyas M. Influence of food-simulating solutions and surface finish on susceptibility to staining of aesthetic restorative materials. J Dent. 2005;33(5):389-98.

4. Um CM, Ruyter I. Staining of resin-based veneering materials with coffee and tea. Quintessence Int. 1991;22(5).

5. Lundin S-Å, Noren JG. Marginal leakage in occlusally loaded, etched, class-II composite resin restorations. Acta Odontol Scand. 1991;49(4):247-54.

6. Kalachandra S, Wilson T. Water sorption and mechanical properties of light-cured proprietary composite tooth restorative materials. Biomaterials. 1992;13(2):105-9.

7. Yap A, Lee C. Water sorption and solubility of resin-modified polyalkenoate cements. J Oral Rehabil. 1997;24(4):310-4.

8. Yoon TH, Lee YK, Lim BS, Kim CW. Degree of polymerization of resin composites by different light sources. J Oral Rehabil. 2002;29(12):1165-73.

9. Poskus LT, Placido E, Cardoso PEC. Influence of placement techniques on Vickers and Knoop hardness of class II composite resin restorations. Dent Mater. 2004;20(8):726-32.

10. Sigusch BW, Pflaum T, Völpel A, Gretsch K, Hoy S, Watts DC, et al. Resin-composite cytotoxicity varies with shade and irradiance. Dent Mater. 2012;28(3):312-9.

11. Miletic V, Santini A. Optimizing the concentration of 2, 4, 6-trimethylbenzoyldiphenylphosphine oxide initiator in composite resins in relation to monomer conversion. Dent Mater J. 2012;31(5):717-23.

12. Tarle, Meniga, Ristic, Sutalo, Pichler. The effect of the photopolymerization method on the quality of composite resin samples. J Oral Rehabil. 1998;25(6):436-42.

13. Uctasli S, Tezvergil A, Lassila L, Vallittu P. The degree of conversion of fiber-reinforced composites polymerized using different light-curing sources. Dent Mater. 2005;21(5):469-75.

14. Organization IS. ISO 4049: Dentistry-resin based fling materials. The Organization Geneva; 1988.

15. Paravina RD, Ghinea R, Herrera LJ, Bona AD, Igiel C, Linninger M, et al. Color difference thresholds in dentistry. J Esthet Restor Dent. 2015;27:S1-S9.

16. Stansbury J, Dickens SH. Determination of double bond conversion in dental resins by near infrared spectroscopy. Dent Mater. 2001;17(1):71-9.

17. Baroudi K, Saleh AM, Silikas N, Watts DC. Shrinkage behaviour of flowable resin-composites related to conversion and filler-fraction. J Dent. 2007;35(8):651-5.

18. Monsarrat P, Garnier S, Vergnes J-N, Nasr K, Grosgogeat B, Joniot S. Survival of directly placed ormocer-based restorative materials: A systematic review and meta-analysis of clinical trials. Dent Mater. 2017;33(5):e212-e20.

19. Torres C, Augusto M, Mathias-Santamaria I, Di Nicoló R, Borges A. Pure ormocer vs methacrylate composites on posterior teeth: a double-blinded randomized clinical trial. Oper Dent. 2020;45(4):359-67.

20. Yu P, Yap A, Wang X. Degree of conversion and polymerization shrinkage of bulk-fill resin-based composites. Oper Dent. 2017;42(1):82-9.

21. Borges AFS, Chase MA, Guggiari AL, Gonzalez MJ, de Souza Ribeiro AR, Pascon FM, et al. A critical review on the conversion degree of resin monomers by direct analyses. Braz Dent Sci. 2013;16(1):18-26.

22. Faria-e-Silva AL, Fanger C, Nguyen L, Howerton D, Pfeifer CS. Impact of material shade and distance from light curing unit tip on the depth of polymerization of composites. Braz Dent J. 2017;28:632-7.

23. Koupis NS, Vercruysse CW, Marks LA, Martens LC, Verbeeck RM. Curing depth of (polyacid-modified) composite resins determined by scraping and a penetrometer. Dent Mater. 2004;20(10):908-14.

24. Heintze SD, Zimmerli B. Relevance of in vitro tests of adhesive and composite dental materials, a review in 3 parts. Part 1: Approval requirements and standardized testing of composite materials according to ISO specifications. Schweiz Monatsschr Zahnmed. 2011;121(9):804-16.

25. Giannini M, Di Francescantonio M, Pacheco RR, Boaro LC, Braga RR. Characterization of water sorption, solubility, and roughness of silorane-and methacrylate-based composite resins. Oper Dent. 2014;39(3):264-72.

26. Curtis A, Shortall A, Marquis P, Palin W. Water uptake and strength characteristics of a nanofilled resin-based composite. J Dent. 2008;36(3):186-93.

27. Øysæd H, Ruyter I. Water sorption and filler characteristics of composites for use in posterior teeth. J Dent Res. 1986;65(11):1315-8.

28. Sideridou ID, Karabela MM, Vouvoudi EC. Volumetric dimensional changes of dental light-cured dimethacrylate resins after sorption of water or ethanol. Dent Mater. 2008;24(8):1131-6.

29. Yap AU, Tan C, Chung S. Wear behavior of new composite restoratives. Oper Dent. 2004;29:269-74.

30. 30. Toledano M, Osorio R, Osorio E, Fuentes V, Prati C, Garcıa-Godoy F. Sorption and solubility of resin-based restorative dental materials. J Dent. 2003;31(1):43-50.

31. Mirsasaani SS, Ghomi F, Hemati M, Tavasoli T. Measurement of solubility and water sorption of dental nanocomposites light cured by argon laser. IEEE Trans Nanobiosci. 2013;12(1):41-6.

32. Wei Y-j, Silikas N, Zhang Z-t, Watts DC. Diffusion and concurrent solubility of self-adhering and new resin–matrix composites during water sorption/desorption cycles. Dent Mater. 2011;27(2):197-205.

33. Chaves LP, Graciano FMO, Júnior OB, do Vale Pedreira APR, Manso AP, Wang L. Water interaction with dental luting cements by means of sorption and solubility. Braz Dent Sci. 2012;15(4):29-35.

34. Pearson G, Longman C. Water sorption and solubility of resin-based materials following inadequate polymerization by a visible-light curing system. J Oral Rehabil. 1989;16(1):57-61.

35. Venz S, Dickens B. NIR-spectroscopic investigation of water sorption characteristics of dental resins and composites. J Biomed Mater Res. 1991;25(10):1231-48.

36. Gönülol N, Şen Tunç E, Ozer S, Yıldızlı K. Evaluation of water sorption-solubility and surface roughness of different bulk fill composite resins. Meandros Med Dent J. 2019;20(1).

37. Yap AU. Resin-modified glass ionomer cements: a comparison of water sorption characteristics. Biomaterials. 1996;17(19):1897-900.

38. Cefaly DFG, Franco EB, Mondelli RFL, Francisconi PAS, Navarro MFdL. Diametral tensile strength and water sorption of glass-ionomer cements used in Atraumatic Restorative Treatment. J Appl Oral Sci. 2003;11:96-101.

39. Salas M, Lucena C, Herrera LJ, Yebra A, Della Bona A, Pérez MM. Translucency thresholds for dental materials. Dent Mater. 2018;34(8):1168-74.

40. Ghinea R, Pérez MM, Herrera LJ, Rivas MJ, Yebra A, Paravina RD. Color difference thresholds in dental ceramics. J Dent. 2010;38:e57-e64.

41. del Mar Perez M, Ghinea R, Herrera LJ, Ionescu AM, Pomares H, Pulgar R, et al. Dental ceramics: a CIEDE2000 acceptability thresholds for lightness, chroma and hue differences. J Dent. 2011;39:e37-e44.