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Degree of conversion and water sorption of self-adhesive and conventional flowable composites: an in vitro study

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Aim: Self-adhesive flowable composite resins have been recently introduced to the market. Degree of conversion (DC) and water sorption (WS) are two important parameters affecting the properties of restorative materials. This study aimed to assess the DC and WS of a self-adhesive flowable composite resin in comparison with two conventional flowable composite resins. **Methods:** Vertise Flow (VF) self-adhesive and Tetric-N Flow (TF) and Grandio Flow (GF) conventional flowable composites were evaluated in this in vitro, experimental study. The DC (n=3) was determined by attenuated total reflectance-Fourier transform infrared spectroscopy (ATR-FTIR). The WS (n=7) was measured after 7 days of immersion in artificial saliva according to ISO 4049 specifications. Data were analyzed by one-way ANOVA and a post-hoc test ($p<0.05$). **Results:** VF showed the highest DC percentage (84.3%) followed by GF (72.79%) and TF (68.7%). The latter two had no significant difference ($p=0.8$). WS was the highest in VF (55.2 $\mu g/mm^3$), and the two conventional flowable composites had a significant difference in WS (19.5 $\mu g/mm^3$ in TF and 11 $\mu g/mm^3$ in GF; $p<0.001$). Conclusions: Flowable composite resins had significant differences in DC and WS, and VF demonstrated the highest DC and WS.

Keywords: Absorption. Composite resins. Polymerization. Vertise flow.

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Introduction

Composite resins are widely used due to the increased esthetic demands of patients and improvements in their mechanical properties. Composite resins can be classified into putty-like and flowable composite resins according to their consistency[1].

Dental composites consist of two main components namely the organic matrix and the inorganic fillers. The organic phase of composite resins is dominantly composed of dimethacrylate resins. The monomers that are most widely used in commercially available composite resins include bisphenol-A glycidyl methacrylate (Bis-GMA) and urethane dimethacrylate (UDMA), which are high molecular weight monomers[1]. Low-viscosity monomers, such as triethylene glycol dimethacrylate (TEGDMA), are added as diluents to the highly viscous resin matrix in various concentrations to improve the degree of conversion (DC) and increase the filler content[1,2]. Flowable composites were designed for enhanced adaptation to the cavity walls and easy handling and application. They either have less filler loading or high proportion of diluent monomers added to viscous monomers[1,2].

Researchers have focused on simplifying the adhesive and composite application steps by developing self-adhesive restorative materials with favorable handling properties[3]. Self-adhesive flowable composite resins have been recently introduced to the market, which are similar to the solvent-free self-etch adhesive systems[4]. They combine the merits of adhesion to tooth structure and tooth restoration in one product[5]. They bond to tooth structure by mechanical interlocking and chemical interactions[3,6]. Their chemical formulation contains acidic functional monomers such as 4-methacryloxyethyltrimetellitic acid as in Fusio Liquid Dentin (Pentron Clinical, Orange, CA, USA) or glycerophosphate dimethacrylate as in Vertise Flow (VF; Kerr, Orange, CA, USA) self-adhesive flowable composite. The acidity of monomers varies from 1.9 to 3[3]. The composition of resin matrix also includes hydroxyethyl methacrylate (HEMA)[3]. HEMA is responsible for improvement of miscibility of hydrophilic and hydrophobic components, and prevention of phase separation[7]. It also improves the wetting of dentin substrate, and enhances resin penetration into dentin[6,7].

The self-adhesive flowable restorative materials are indicated for small class I and class V cavities, and for sealing of the pits and fissures[3,8]. In the hostile oral environment, the restorative materials need to meet some certain specifications to ensure optimal product quality[9].

DC is one of the most important parameters affecting the properties of restorative materials. The DC of carbon-carbon double bonds to single bonds determines the DC percentage (DC%) of composite resins[10]. After the polymerization reaction of monomers, a cross-linked structure is formed[10,11]. The DC and cross-linking density are important factors affecting the physical and mechanical properties of composite resins[10,11]. However, the monomer conversion is never complete, and a number of double bonds often remain[10,11]. When the polymerized material is exposed to oral fluids, the unreacted monomers can leach out of the resin mass and cause adverse effects such as allergic reactions[10,11]. In addition, a correlation has been reported between the residual double bonds and internal color change of light-cure composite resins[10]. The possible causes include oxidation of unreacted double bonds, and chemical degradation and formation of colored degradation products[10,12].

Several studies have demonstrated that the DC and density of polymer network are important factors affecting the polymer/solvent diffusion behavior[13]. The solvent permeability of polymer networks decreases as the DC and cross-linking between the polymer chains increase[14,15]. On the other hand, water sorption (WS) is a diffusion-controlled process[14,15]. It has been demonstrated that water sorption highly depends on the hydrophilicity/hydrophobicity of monomers in the resin matrix[14-16]. WS can cause several detrimental effects. Its major effect is polymer plasticization, mediated by different mechanisms. The absorbed water may cause hydrolytic breakdown of the resin matrix-filler interface and cause filler debonding from the resin matrix, hydrolytic cleavage of polymer chains, elution of unreacted monomers and leachable components, and deterioration of the mechanical properties of polymer such as its wear resistance[10,11,17]. In addition, WS is of particular importance in external discoloration of composite resins[18]. It could be related to different mechanisms including penetration of staining agents into the surface and subsurface layers of composite resin, and superficial degradation[18]. Resin matrix hydrophilicity is also associated with external color change[14,18].

Therefore, DC and WS are important parameters affecting the physical and mechanical properties of composite resins, and longevity and clinical service of adhesive restorations[10,11].

The purpose of this study was to compare VF (Kerr, Orange, CA, USA) a self-adhesive flowable composite with two conventional flowable composites namely Tetric-N Flow (TF; Ivoclar Vivadent AG, Schaan, Liechtenstein) and Grandio Flow (GF; Vocco, Cuxhaven, Germany) in terms of DC and WS. The null hypotheses were that the DC and WS of the three composite resins would not be significantly different.

Materials and Methods

Sample size calculation:

One-way ANOVA of PASS 11 software was used for sample size calculation, assuming type one error of 0.05, statistical power of 80%, and a standard deviation of 2.87. Accordingly, the minimum sample size to ensure detecting a difference of at least 10% between the groups was found to be 3 samples for the DC test[8]. For the WS test, assuming a standard deviation of 4.86, the minimum sample size to ensure detecting a difference of at least 8 μg/mm^3 between the groups was found to be 7 samples[18].

Specimen preparation for the DC test

Table 1 lists the materials used in this study. The A2 shade of composite resins was used. For preparation of composite specimens (4 mm in diameter, 1 mm in thickness, n = 3 of each material), the flowable composite resins were dispensed into plexiglass molds, pressed between two glass slides (0.5 mm thick), and covered with a polyester strip form a flat surface and to prevent the formation of oxygen inhibited layer upon polymerization. Each specimen was polymerized for 20 s using a LED curing unit (Bluephase; Ivoclar Vivadent, Schaan, Liechtenstein). The tip of the light curing unit was in contact with the top glass slide. The light intensity was 1100 mW/cm^2.

Table 1. Resin composites used and their classification, manufacturer, and composition

Product	Composite type	Manufacturer	Resin composition	Filler system	Filler load (wt%)
Vertise flow	Nanohybrid	Kerr, Orange, CA, USA	GPDMA, HEMA, BisGMA, MEHQ	Prepolymerized filler, Barium glass, Nanosized colloidal silica, Nanosized ytterbium fluoride	70
Grandio flow	Nanohybrid	VOCO, Cuxhaven, Germany	Bis-GMA, TEGDMA, HEDMA	Inorganic fillers: Silicate oxide, Silicate glass pigment	80
Tetric-N flow	Nanohybrid	Ivoclar Vivadent, Schaan Liechtenstein	Bis-GMA, UDMA, TEGDMA	Barium glass, Ytterbium fluoride, Silica	63.8

GPDMA: Glycerol Prosphate dimethacrylate; HEMA: hydroxyethyl methacrylate; Bis-GMA: bisphenol A glycidyl dimethacrylate; MEHQ: monomethyl ether hydroquinone; TEGDMA: triethylene glycol dimethacrylate; HDDMA: 1,6-Hexanediol dimethacrylate; UDMA: urethane dimethacrylate

The specimens were demounted, and the DC was measured at the center of the top surface of the specimen immediately after polymerization by attenuated total reflectance-Fourier transform infrared spectroscopy (ATR-FTIR; Nicolet IS10; Thermo Fisher Scientific, Waltham, MA, USA). The IR spectra of the specimens were determined in the wave number range of 650-4000 cm^{-1} at a resolution of 4 cm^{-1} and IR depth of ~2 μm.

Uncured resin specimens were also subjected to ATR-FTIR, to serve as non-cured references. Unpolymerized composite was directly placed on the device's ATR crystal.

The DC was determined according to the changes in the absorbance values of the aliphatic C-C bonds (1635 cm^{-1}), which are consumed during polymerization, and the aromatic C…C bonds (1608 cm^{-1}), which are not affected during the curing process; this peak is almost constant and therefore serves as an internal standard.

The ratios of the absorbance peak intensities ascribed to (C=C) before and after polymerization, representing the unreacted carbon-carbon double bonds, were compared and accordingly, the DC was calculated by the following equation:

$$DC\% = \left[1 - \frac{(1635\ \text{cm}^{-1} / 1608\ \text{cm} - 1)\ \textit{peak after curing})}{(1635\ \text{cm}^{-1} / 1608\ \text{cm} - 1)\ \textit{peak before curing})} \right] \times 100$$

Specimen preparation for the WS test

The same flowable composite resins were used for the WS test according to ISO 4049:2009. Seven disc-shaped specimens of each composite were fabricated using plexiglass molds (15 ± 1 mm in diameter and 1.0 ± 1 mm in thickness). Composite resins were applied into the molds, their surface was covered with a polyester strip and a glass slide, and compressed to prevent voids and porosities.

The composites were then light-cured from both sides for 20 s through the glass slide/polyester strip with a LED curing unit (Bluephase; Ivoclar Vivadent, Schaan, Liechtenstein) with a light intensity of 1100 mW/cm^2 and a tip diameter of 10 mm using the overlapping exposure technique to ensure adequate exposure of all areas. The mean output intensity of the light curing unit as assessed with a calibrated radiometer (Bluephase meter; Ivoclar- Vivadent), was 1100 mW/cm^2. The periphery of all specimens was ground with 1200-grit silicon carbide abrasive paper.

Water sorption test

The specimens were placed vertically on a metal rack to avoid sample-to-sample contact. The specimens were placed in a desiccator containing dehydrated silica gel. The desiccators were placed in an incubator (Kavosh Mega; Kavosh Azma Co, Tehran, Iran) at 37°±1° C for 22 h followed by an additional 2 h at 23°±1°C. Each Specimen was then weighed on a scale (GF-3000; A&D, Tokyo, Japan) with 0.1 mg accuracy. This drying cycle was repeated until the weight loss of each specimen was lower than 0.1 mg within a 24-h period. At this point, the weight was considered constant (W1) and reported in micrograms (μg). Next, the specimens were immersed in distilled water for 7 days at 37º±1ºC. The water was changed every day. After completion of the storage period, the specimens were removed from the water with tweezers, and blotted dry with a clean tissue paper and weighed again (W2). Next, the specimens were subjected to the same conditioning cycle until achieving a constant weight (W3).

After final drying, the thickness of each specimen was measured using a digital caliper (Mitutoyo Corp., Kawasaki, Japan) at four equally spaced points on the circumference. The thickness at the center of the specimens was also measured. The diameter was measured in two perpendicular lines as well. The average volume (V) of specimens was calculated in cubic millimeters (mm^3).

The WS value of each specimen (in μg/mm^3) was calculated using the following formula:

$$WS\ (\mu g/mm^3) = (W2 - W3)/V$$

Statistical analysis:

Normal distribution of data was evaluated using the Shapiro-Wilk and Kolmogorov-Smirnov tests. The data were analyzed using one-way ANOVA. The Tukey's test and the Games-Howell tests were chosen according to the homogeneity of variances, which was checked by the Levene's test. Multiple comparisons were performed by the Tukey's test for DC (since the homogeneity of variances was met for this variable) while the Games-Howell test was used for WS (since the homogeneity of variances was not met for this variable). P-values < 0.05 were considered significant. All statistical analyses were carried out using SPSS version 20 (SPSS Inc., Chicago, IL, USA).

Results

Table 2 presents the mean values of DC and WS. The tested materials had statistically significant differences in both WS (p=0.001) and DC (p=0.002). VF presented significantly higher WS than other composites (p<0.001); while, GF had the lowest WS

Table 2. Mean (standard deviation) of the degree of conversion (DC%) and water sorption (µg / mm3) data

	Vertise-flow (VS)	Grand flow (GF)	Tetric-N flow (TN)	VS vs. GF mean difference (95%CI)	VS vs. TN mean difference (95%CI)	GF vs. TN mean difference (95%CI)
DC	84.39±1.21[a]	72.79±3.21[b]	71.50±2.91[b]	11.59 (5.08 to 18.11) p-Value=0.004	12.88 (6.37 to 19.40) Value=0.002	1.29 (5.23 to 7.81) Value=0.82
WS	55.21±4.92[a]	11.06±2.37[b]	19.53±3.15[c]	44.15 (38.34 to 49.96) p-Value<0.001	35.68 (29.65 to 41.72) p-Value<0.001	-8.46 (-12.49 to -4.44) p-Value<0.001

Means with the same superscript letters indicate non-significant difference (p≥0.05)

(p<0.001).VF showed significantly higher DC than other composite resins; other composites had no significant difference with each other in this respect (p=0.8).

Discussion

This study evaluated the WS and DC of a self-adhesive flowable composite compared with two conventional flowable composite resins. The results demonstrated statistically significant differences among the materials in WS and DC (Table 2). Therefore, the null hypotheses were rejected.

In the present study, the DC was quite high in the three flowable composites, ranging from 71.5% to 84.3%; VF showed the highest DC. The minimum DC for a clinically acceptable composite restoration has not been exactly determined. However, a DC ranging from 55% to 85% has been reported in the literature[10,19]. The level of polymerization is an important indicator for the quality and characteristics of polymers[10].

In the current study, DC was measured 5 min after light polymerization. Several studies revealed that the DC values measured 24 h after light irradiation were significantly higher than the DC values measured immediately or 5 min after curing[20,21]. However, the DC measured after 5 min is of great importance because the restorative material is exposed to the water spray of low-speed and high-speed handpiece for contouring or occlusal adjustment and is also exposed to the saliva and oral fluids shortly after light irradiation[22]. This description could legitimize our methodology regarding measuring the DC 5 min after light irradiation. Furthermore, FTIR in the current study showed that most of the polymerization occurred within 5 min after light exposure, and it can be assumed that no further statistical increase in the DC values can be attained in up to 24 h.

Several factors affect the DC including the material-related factors. It has been demonstrated that the type, volume and viscosity of monomers in the resin matrix composition could affect the DC. Monomers are selected based on their viscosity and their contribution to the crosslink density of the polymer network[11,23]. The highly viscous monomers have lower mobility, which affects the propagation of free radicals in the resin matrix[11,23]. Increased viscosity causes the termination of diffusion-controlled mechanism at earlier stages of the polymerization process, leading to a low DC[23]. To improve the conversion of the network, low viscosity monomers are required to dilute the highly viscous monomers and provide an optimal monomer ratio. It has an important effect on the mobility of molecules in the resin matrix and polymerization process, and providing the best performance in terms of required parameters such as high mechanical properties and WS[10,22]. Also, various types of photo-initiators can affect the DC of monomers. Filler type, filler size, and loading level can affect the monomer conversion by altering light transmission and viscosity of composite resins as well[1].

Visible light-cured dental composite resins are usually polymerized by the camphorquinone (CQ)/amine photo-initiator system[1]. Free radical polymerization is started upon blue light exposure and excitation of CQ and reduction of tertiary amine electron donor[1]. However, it has been documented that proper polymerization is inhibited by acidic monomers through neutralization of tertiary basic amine as a reducing agent[24,25]. The inhibition of photopolymerization depends on the type, concentration, and pH of the acidic functional monomers[25]. However, neutralization depends on the type of photo-initiator system[24,25]. Therefore, a suitable initiator system is critical for polymerization of acidic photopolymerizable composite resins[24,25].

The possible explanations for the high DC of VF self-adhesive composite resin are as follows: (a) Higher amounts of initiator systems: VF may contain alternative co-initiators as amine substitutes to compensate for inactivation of the amine co-initiator, which improve the DC, (b) since Bis-GMA is highly viscous, some of this monomer is probably replaced with low molecular weight co-monomers to obtain lower viscosity. It allows better monomeric mobility, which can enhance polymerization[8,24-26]. However, these effects cannot be assessed since detailed information about the chemical composition of VF has not been disclosed by the manufacturer[22].

The TF and GF composites had no significant difference in DC, although they have a different filler content. The filler content is 80 wt.% for GF and 63.8 wt.% for TF. An inverse correlation exists between the DC and filler loading[27]. Composites with higher filler content usually have a high viscosity and this factor may interfere with monomer conversion and limit the propagation of polymerization process[1,3,10]. However, we did not find such a causal relationship in the current study. The similar values observed in this study could be related to the differences in the resin matrix composition, and type and proportion of monomers[9].

GF does not contain high molecular weight UDMA (571 g/mol), and is composed of low viscosity monomers such as hexanediol dimethacrylate resin, a hydrophobic low viscosity monomer (η=0.007 Pa.s) with low molecular weight (254 g/mol), and TEGDMA (MW=286 g.mol and η=0.008 Pa.s) as diluents[11,28]. The TF is composed of high molecular weight monomers, Bis-GMA, and UDMA. TEGDMA is the only low viscosity monomer in its formulation (Table 1). Thus, the composition of GF may affect the DC in spite of higher filler content.

VF exhibited the highest amount of water sorption during 1 week of immersion in distilled water (55.2 µg/mm^3), while GF had the lowest water sorption (11 µg/mm^3) during this period. These results agree with other studies[15,18]. Several factors can affect water sorption such as the chemical formulation of matrix monomers and the matrix filler content[18].

The three flowable composites tested in the present study were nano-hybrid composites. One difference between the conventional flowable composites and VF is that they do not contain HEMA. In addition, Bis-GMA and UDMA, which are highly hydrophobic, are incorporated in the resin matrix of conventional flowable composites. However, self-adhesive flowable composites contain an acidic monomer, GPDM, which is a quite hydrophilic monomer due to the presence of one phosphate acidic functional group, as well as HEMA in their resin matrix, which affects the hydrophilicity of the resulting polymer and makes it more susceptible to WS[3,18].

Another factor that may contribute to higher water sorption of VF is the type of filler particles[18]. VF is composed of four types of fillers including pre-polymerized fillers (PPFs). PPFs are ground, cured composite resins initially filled with sub-micron particles[29]. The rate of water sorption depends on the quality of resin-filler bond. If the filler-matrix interface is uncoupled, it can provide paths for water diffusion[15,29]. Integration of PPFs in the resin matrix is weak because the functional groups have already reacted during their preparation, and silanization is difficult; thus, breakdown of the interface may occur[29].

GF had the lowest water sorption in this study, which can be attributed to its filler content. An inverse correlation exists between the filler loading and water sorption[16]. This can be explained by lower free space in the resin matrix as the filler content increases[30]. In the current study, WS behavior of the materials was not inversely correlated with their DC as demonstrated in another study[16]. The self-adhesive composite showed the greatest WS with high DC. It implies that the cross-linking nature of the polymer network does not necessarily decrease WS of the polymer. It can indicate the important role of chemical composition and hydrophilicity of resin monomers in WS[30].

The WS level of VF in the current study did not comply with the requirements of ISO 4049:2009 for WS[31], as the value was above 40 µg/mm^3. It may cause fast intraoral degradation, and lead to decreased physical and mechanical properties, and reduced longevity of composite restorations, questioning their routine clinical application. Future studies are recommended to assess the effects of resin composition, filler content, filler type, and polymer network of these composites on their WS, solubility, color change, and wear resistance.

Conclusion

The results of the current study showed that VF self-adhesive flowable composite had a high DC in comparison with the conventional flowable composites. The mean water sorption of VF self-adhesive composite was 55.2 µg/mm^{3,} which is unacceptable according to ISO4049:2009.

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