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The effect of light-curing distance on the degree of convergence of the top and bottom sides of flowable bulk-fill composite materials

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***Abstract*---**Background: Flow able bulk-fill materials were introduced to be used in one thick increment of an internal base underneath a conventional layer. However, complete polymerization of the whole layer, particularly the bottom surface, is essential to achieve biocompatibility and the best mechanical properties. Objective: To evaluate the effect of three light-curing distances (0, 2, and 4mm)on the degree of convergence(DC) of the top and bottom surfaces for three flowable bulk-fill composite resins; Smart dentin replacement, Opus bulk fill flow, and Tetric N flow. Methods:45 disk-shaped specimens with an internal diameter of 5 mm and a depth of4mm cured for 20 seconds were fabricated from the three composite resins (n=15). For each material, three subgroups were fabricated according to the position of the light tip from the top surface; at 0, 2, and 4mm distances. DC for the top and bottom sides of all the specimens was calculated using FTIR spectroscopy. The statistical significance for the mean values of DC% among the different groups was calculated using a one-way analysis of variance (ANOVA) and the Tukey test with a significance level of 0.05. Results: No significant differences in DC% between the top and bottom surfaces of the specimens at each distance were recorded. For the same material, the DC% values were significantly higher at 0mm compared to 4mm light tip distance for both the top and bottom surfaces ($p \leq 0.05$). For the three tested resins, Opus flow had the significantly highest DC%. Conclusion: Increasing the distances between the tip of the light cure and the surface of flowable bulk-fill resins can significantly affect the DC% of the bottom sides compared to the top sides.

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Introduction

The clinical effectiveness of a composite resin restoration is contingent on several material-related criteria, such as the degree of polymerization, polymerization shrinkage of the resin, linear coefficient of thermal expansion, modulus of elasticity, wear resistance, and depth of cure, etc. (AnandYokesh et al., 2017). One of these factors is the degree of conversion (DC), which is considered vital as fewer clinical problems can arise when complete polymerization of a material is achieved. The degree of conversion is the proportion of carbon-carbon double bonds (-C=C-) that are converted to single bonds (-C-C-) during the formation of a polymeric resin. (Taheri et al., 2021). FTIR spectroscopy was considered a more accurate method to determine the unreacted C=C bonds in a composite material compared to other tests (Reis et al., 2017).

One factor affecting any composite resin's mechanical properties is its filler load. It has been established that the filler size and load influence light penetration through a composite restoration. (Taheri et al., 2021). Flowable bulk-fill resin composites represent low viscosity resin composites resulting from lower filler content (37-53%) by volume than conventional composites (Aung et al., 2021). Thus, the polymerization process can increase the inter/intramolecular tension dissipation capability.

Some researchers have found that increasing the distance between the composite surface and the light cure tip decreases the radiant exposure of the light. (Beolchi et al., 2015). This reduction could be produced due to tooth morphology such as; cusp height, cusp steepness, and cavity depth, forcing the curing tip to move away from the composite surface (Aguiar et al., 2005). The effect of light-curing distance on the curing of bulk-fill flowable composite is clinically relevant since it is cured in increments of 4 mm, making the restoration's bottom surface especially susceptible to light scattering as the distance from the light source increases (AnandYokesh et al., 2017; Diab et al., 2021).

A composite resin restoration with a lower DC % of monomers to the polymer may have a shorter lifespan, since an incomplete conversion may lead unreacted monomers to disintegrate in an excessively wet environment. When DC levels are insufficient, mechanical qualities such as tensile strength, flexural modulus, and temperature resistance are affected, and color stability may deteriorate. Insufficient DC % may increase cytotoxicity, wear, marginal degradation, and microleakage (Balagopal et al., 2021). Human gingival fibroblasts were discovered to be poisoned by monomers produced by inadequate DC % resin composites. Since they are secreted into saliva and diffuse into the tooth pulp, gingiva mucosa, and salivary glands, they may contribute to cancer by immortalizing human keratinocytes (Kleinsasser et al.,2006). When high DC % is reached, the amount of such harmful leachable chemicals is minimized (Balagopal et al., 2021). In bulk-fill composites, the high translucency and equivalent refractive indices of the organic matrix and filler particles allowed sufficient light to flow through and polymerize the deeper layers. In addition, introducing more reactive photoinitiators enhanced the curing depth of bulk-fill composites, resulting in performance equivalent to traditional composites, even though the product was supplied in a single thicker increment. (Sousa-lima et al., 2020).

Several studies were performed to compare the degree of convergence between different bulk-fill resin-based composites. Smart dentin replacement (SDR) composite resin has been reported to have the highest DC% compared to Venus and Surefil bulk-fill composites at a 0 mm cure distance (Czasch and Ilie, 2012). Lempel et al. (2016) compared SDR flow with Xtrabase, Filtek bulk-fill, and Filtek ultimate flow in 4mm thickness and showed that SDR recorded the highest DC value not just at the bottom but also at the top surface. Yu et al. (2017) conducted a study to assess the degree of conversion of bulk-fill resin-based composites such as Beautifil Bulk, Beautifil Bulk Flowable, Tetric N-Ceram bulk-fill, and SDR at 2, 4, and 6mm thickness. They found that SDR had the highest DC and that DC significantly decreased with the increased thickness of the samples.

Several researchers also investigated the influence of the exposure light distance on bulk-fill composites (Suman et al., in.2017, Rezaei et al., 2019). Curing distance strongly affects the DC of high-viscosity bulk-fill composites (Ilie and Stark, 2014). However, little evidence is available on the effect of curing distance on the degree of convergence of two new flowable bulk-fill resins (Opus bulk-fill flow and Tetric N flow bulk-fill). Since low-viscosity bulk-fill materials are suggested to be used as an internal layer adjacent to cavity walls (Comba et al., 2020; Haugen et al., 2020), light exposure to the bottom surface has reduced, and the restoration in deep cavities could be an issue. Therefore, this study was conducted to evaluate the top to bottom degree of convergence of three flowable bulk-fill composites cured at three light-curing distances; 0, 2, and 4mm. The null hypotheses of the study were: 1. At each curing distance (0, 2, and 4mm), no difference in DC% between the top and bottom sides for the three types of flowable bulk-fill composite resins (SDR, Opus bulk-fill flow and Tetric N flow bulk-fill), 2. For each material, no difference in DC% of the top or the bottom sides between the three curing distances, and 3. Similar DC% can be recorded at different curing distances for the three tested materials.

Materials and Method

The three types of flowable bulk-fill composite resins used in the study and their compositions are shown in Table (1).

Table (1): The flowable bulk-fill composite resins used in this study.

Products	Manufacture And shade	Method of Activation & Curing time	Resin Components	Fillers (type- loads(wt/vol)-size)
Smart dentin replace	Dentsply Caulk, Milford,	Visible light cure for 20 sec. by	Modified UDMA resin, TEGDMA, polymerizabledimethacryl	barium-alumino-fluoro-borosilicate glass, silanated strontium

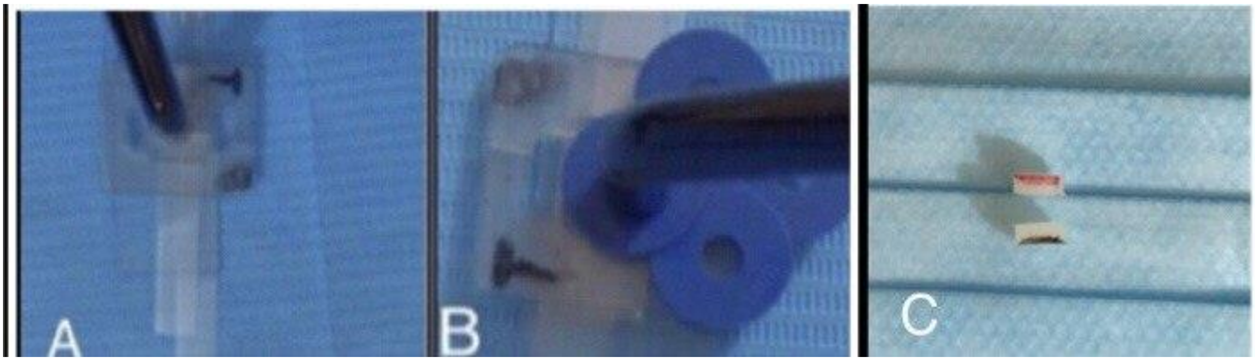
ment	DE, USA, Shade A1	550mW/cm^2 up to 4 mm thickness	ate resin; polymerizabletrimethacry lateresin ;camphorquinone (CQ) photoinitiator; ethyl-4(dimethylamino)benzoat e photoaccelerator; butylated hydroxytoluene (BHT); fluorescent agent, and UV stabilizer.	alumino-fluoro-silicate glass, surface-treated fumed silicas, ytterbium fluoride, synthetic inorganic iron oxide pigments, and titanium dioxide. 70.5 wt%., 47.4 vol%. 4.2 µm filler size.
Opus bulk-fill flow	FGM, Joinville, SC, Brazil. Shade A1	Visible light cure for 20 sec. up to 4 mm	UDMA monomers, Stabilizers, Camphorquinone Co-initiator (Quantities and specifications not provided by the manufacturer).	Inorganic silicon dioxide (silica) loads, stabilizers, and pigments. No available filler load nor filler size
Tetric N flow bulk-fill	IvoclarViv adentInc Amherst, NY, USA. Shade ^{IV}A	Visible light cure. ≥ 500 mW/cm^2, for 20 sec. ≥ 1000 mW/cm^2, for10 sec. up to 4 mm	Bis-GMA, UDMA, TEGDMA	Barium glass, ytterbium trifluoride, and copolymers (71 wt%). Additives, initiators, stabilizers, and pigments are additional ingredients (<1.0 wt%). 68.2 wt%., 46.4 vol%. Ranges between 0.1µm & 30µm with a mean particle size of 5µm.

Sample preparation and grouping

Forty-five specimens were fabricated from the three materials representing three groups (n=15): Group A using SDR, Group B using Opus bulk fill flow, and Group C using Tetric N flow. All the specimens were constructed using a disk-shaped custom-made Teflon mold with a 5 mm diameter and a 4 mm depth. Three subgroups were fabricated from each group according to the light-curing distances (n=5) as follows:
Subgroups A1, B1, and C1 were cured with a curing tip distance of 0 mm. Subgroups A2, B2, and C2 were cured with a curing tip distance of 2 mm, and SubgroupsA3, B3, and C3 were cured with a curing tip distance of 4 mm.

Each specimen was prepared using two split Teflon molds (top and bottom parts of 2-mm depth each). One represented the top part, and the other represented the bottom part used to measure the DC of each surface (Pirmoradian et al., 2020). First, the bottom mold was placed on a glass slide, filled with the experimented flowable bulk-fill composite, and covered by a Mylar strip. Then, the second Teflon mold was placed over the first mold and filled with the same flowable composite, and the mold was covered with another Mylar strip to produce a flat smooth surface (Anand Yokesh et al., 2017).

Then, light polymerization was performed for 20 seconds using a light-emitting diode (LED) system (Guilin Woodpecker Medical Instrument Co., Ltd. China) with a curing tip diameter of 8 mm and light intensity of 1000-1200mW/cm^2. Polymerization for subgroups A1, B1, and C1was performed with the light tip directly contacting the top surface at a 0mm distance. For subgroups A2, B2, and



C2, curing was performed as the tip of the light cure was positioned 2mm away from the specimen's top surface using a 2mm thick plastic ring spacer. For the third subgroups, A3, B3, and C3, the tip of the light cure system was positioned 4mm away from the top of the surface of the specimen using a plastic ring spacer with a thickness of 4mm (Malik and Baban, 2014) as illustrated in Figure (1).

Figure (1): Sample construction. A: Without a spacer. B: With a spacer. C: The two parts of a sample are labeled by a red line on the top side of the upper part and a black line on the bottom side of the lower part.

After light curing, the two parts of each sample were labeled to identify the samples' top and bottom surfaces. Later, these specimens were organized in dry and dark containers at 37^{o}C for 24 hours (Galvão et al., 2013; Aung et al., 2021).

Testing procedure

The degree of convergence was measured using Fourier transform infrared spectroscopy (Bruker Tensor 27 FT-IR Spectrometer). Each specimen was measured from the top side of the top part, while the bottom side was measured using the bottom side of the lower part of the specimen. DC was calculated by estimating the changes in peak height ratio of the absorbance intensities of aliphatic C=C peak at 1639 cm^{-1} and peak of aromatic C=C at 1608 cm^{-1}, polymerization in relation to the degree of convergence of an uncured material from each resin that was tested first (Figure 2).

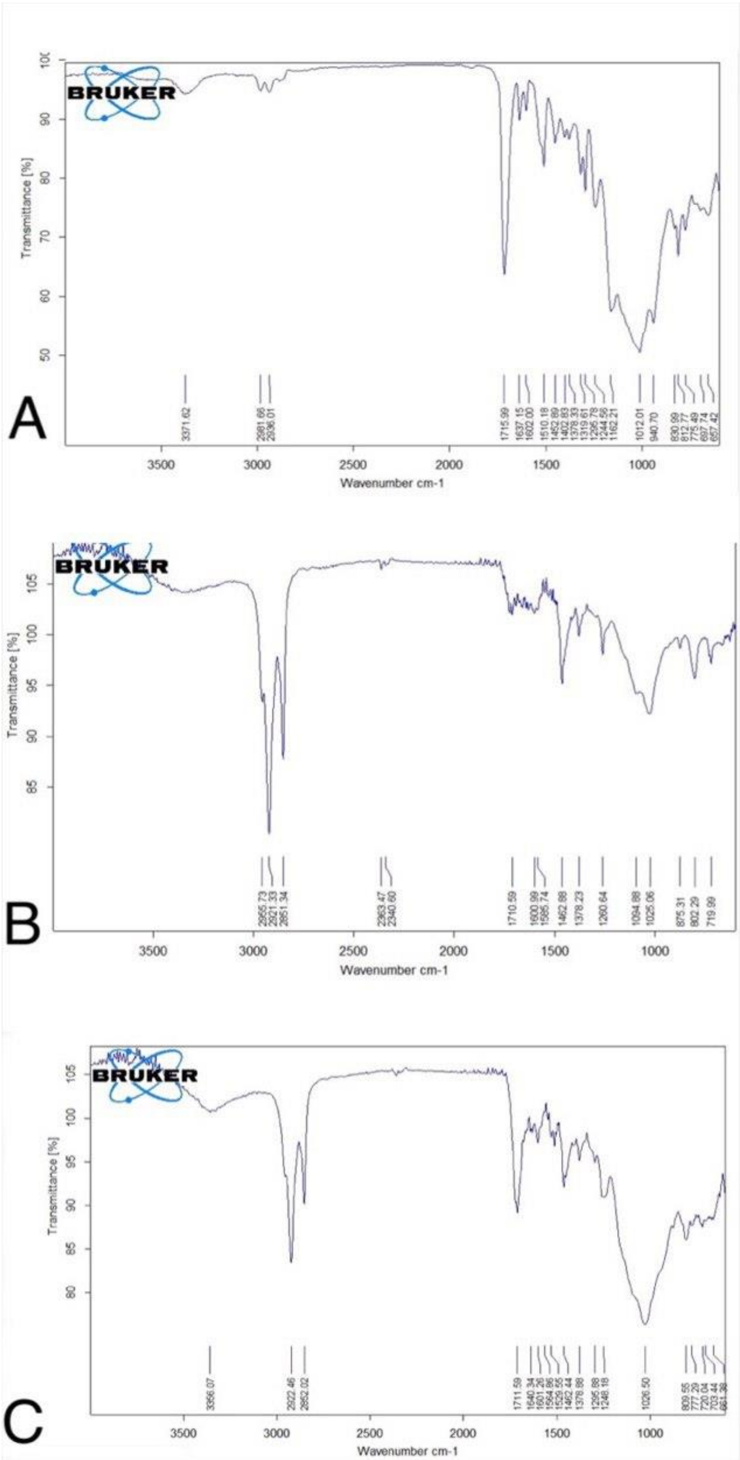


Figure (2): Example of the output reading achieved from the FTIR device. (A) uncured SDR, (B)Top side of cured SDR sample, and (C) Bottom side of cured SDR sample.

The DC % was calculated using the equation below (Wikant et al., 2020):
DC= [1-(1639 cm^{-1}/1608cm^{-1}) cured / (1639 cm^{-1}/1608cm^{-1}) uncured]×100%

The DC% values were statistically analyzed using SPSS 26 (IBM-SPSS Inc., Chicago, USA). The statistical significance for the mean values of DC% among the different groups was calculated using a one-way analysis of variance (ANOVA) and Tukey's test at = 0.05 level of significance.

3. Result

The means of the DC% for the top and bottom sides for all the subgroups are represented in Figure (3).

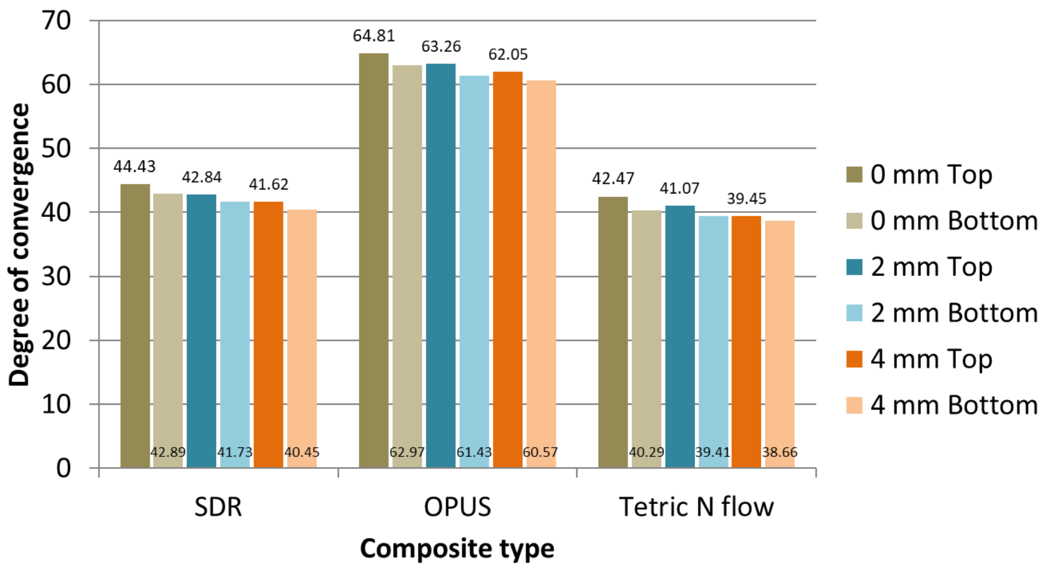


Figure (3): The means of DC% for the top and bottom sides of the three Bulk Fill groups at the three light tip distances

The results showed that the highest DC% value was at 0 mm, followed by 2 mm, and 4 mm of light tip distances, respectively, for both top and bottom sides and all three types of composite resins. The mean DC% values for the top/bottom surfaces at the three curing distances for the three types of flowable bulk-fill composites are shown in Table (2). Significant differences were recorded between the subgroups recorded by the Tukey post hoc test ($p \leq 0.05$).

Table (2): The mean DC%, SD, Max, and Min values for the top/bottom surfaces at the three curing distances for the three types of flowable bulk-fill composites

Groups	Light tip distance (mm)	Top /bottom	Mean (%) *	SD	Max (%)	Min (%)
Group A	0	top	44.43 {a} (d)	0.81	45.32	43. 24
		bottom	42.89 {i } (k,n)	0.68	43.56	42.12

SDR	2	top	42.84(e)	1.16	44.21	41.26
		bottom	41.73 (l,o)	0.67	42.54	40.77
	4	top	41.62 {a}} (f)	0.79	42.54	40.77
		bottom	40.45{i}(m)	0.47	41.21	40.12
Group B Opus flow	0	top	64.81 {b} (d,g)	1.74	66.32	62.54
		bottom	62.97 {j} (k,p)	1.27	64.12	61.11
	2	top	63.26 (e,h)	1.16	64.45	61.87
		bottom	61.43 (l,q)	0.74	62.28	60.28
	4	top	62.05 {b} (f)	0.82	63.11	60.85
		bottom	60.57{j} (m,r)	0.73	61.76	60.01
Group C Tetric N	0	top	42.47 {c } (g)	1.17	44.17	41.23
		bottom	40.29 (n,p)	0.84	41.54	39.21
	2	top	41.07 (h)	1.39	43.47	39.96
		bottom	39.41 (o,q)	1.27	41.14	38.11
	4	top	39.54 {c}	0.83	40.33	38.43
		bottom	38.67 (r)	1.11	39.92	37.12
* mean values with the same letter are sig. different at p≤ 0.05. { sig. differences between the three distances }, and (sig. differences between the three groups)						

No significant difference was recorded between the top and bottom surfaces at all the three curing distances for the three materials (p>0.05). For all the composite types, a significantly higher DC%for the top sides was recorded between 0 and 4mm curing distances. For the bottom side, there was a significant difference between 0 and 4 mm curing distances of SDR and Opus flow only (p≤0.05).

The results also showed significant differences in the degree of convergence between the top side of SDR and Opus flow and between Opus flow and Tetric N at all three levels of curing distances (p≤ 0.05). For the bottom side, there were significant differences in the degree of convergence of the three composite groups at the three curing levels except that between SDR and Tetric N at 4mm curing distance which was not significant (p>0.05).

4. Discussion

The degree of conversion test is an essential tool to determine the physical, mechanical, and biological properties of composite resins. An incomplete or low degree of convergence can affect the restoration performance as uncured monomers are potentially leachable and can act as plasticizers, reducing the mechanical properties of the resin. When these uncured monomers are in a low amount within the composite resin, more biocompatible and extended restoration longevity is expected (Balagopal et al., 2021).

In the current study, the degree of convergence for the top surfaces was greater than those of the bottom surfaces but with no significant differences at each light tip distance and for all the three experimented materials. Hence the first null hypothesis of this study was accepted. This result is in accordance with the study of Suman et., in 2017; and Rezaei et al., 2019 who evaluated the DC from top and bottom using FTIR spectroscopy of different bulk-fill (Ever X Posterior, Filtek bulk-fill Posterior, Sonic Fill 2, Tetric N-Ceram Bulk-Fill, and X-tra Fil bulk-fill).

They found that at a 4 mm depth, the evaluated bulk-fill composites were polymerized correctly, and their DC was within the range of conventional composites. Several modifications achieved this property: decreasing the filler content, increasing the filler volume, increasing the translucency, and changing or adding new photoinitiators (Gan et al., 2018). With their higher translucency, Bulk-fill composite materials transmit light better than conventional composites (Comba et al., 2020). The Tetric N-Ceram Bulk Fill composite contains three distinct photoinitiators: trimethyl benzoyl-diphenylphosphine oxide (TPO), camphorquinone (CQ), and Ivocerin (a dibenzoyl germanium compound). The manufacturer of Tetric N-Ceram uses the Ivocerin photoinitiator because it can increase the depth of cure compared to photoinitiators used in standard resin composites. (Pirmoradian et al.,2020).In addition, the presence of TEGDMA in the resin reduces the resin viscosity and increases the monomers' reactivity (Wilson et al., 2004). The polymerization process in a specific layer (depth) depends not only on the number of photons reaching that layer but also on the presence of an ongoing polymerization process in the upper layers, which advances deeper. (Fronza et al., 2017).

However, the results are inconsistent with those of Hayashi et al., in 2020 and Lempel et al., in 2016, as significant differences were reported between the top and bottom surfaces in 4mm thickness. However, both studies used Roman spectroscopy for testing the degree of convergence. Among the same group, the results of this study showed that the degree of convergence was in the following order; 0mm > 2mm > 4mm curing tip distance for both sides with significant differences between 0mm and 4mm. This was true for all the experimented composites. Thus, the second null hypothesis was rejected. This result can be attributed to the space between the light tip and the top surface of the resin composite that affects the light intensity that reaches the material (Hassan et al., 2022). According to reports, 1mm of air decreases the light intensity by around 10%, thus can reduce the polymerization depth and the degree of convergence (Aguiar et al., 2005). Similar results were reported for the degree of convergence of Tetric Ceram in 6mm thickness (Lindberg et al., 2005). However, different results were reported by other researchers. Froza et al. (2015) studied the degree of convergence and the microhardness of SDR, Filtek bulk-fill, Tetric EvoCerum bulk-fill, and Ever X posterior in a 4mm thickness. They demonstrated that SDR has a similar degree of convergence at all depths. However, they used FT-Roman spectroscopy for the measurement of DC. Bolaos-Carmona et al. (2020) used different measuring devices; Fourier transforms infrared (FTIR), attenuated total reflection FTIR (ATR-FTIR), and FT-Raman spectroscopic techniques to determine the DC for three flowable bulk-fill composites at identical experimental conditions. They concluded that different spectroscopy could produce significantly different results for the same material. FTIR showed significantly lower values of DC, both in peak areas and peak amplitudes.

This study also showed that the degree of convergence of the Opus bulk-fill flow composite was significantly higher than the other two composites for both the top and bottom sides ($p \leq 0.05$), leading to the rejection of the third hypothesis. This result could be attributed to the differences in the composition of these materials. Light scattering and absorption depend on many things about a material, such as the resin matrix, filler content, photoinitiators, pigments, and even how the fillers' surfaces are treated so they can stick to the resin matrix. (Pirmoradian et al., 2020).In addition, the chemical composition of light-cured dental resins has been shown to influence their degree of convergence. It has been reported that different types of monomers can affect the degree of convergence of resins, and it tends to be higher in the order below: Bis-GMA <Bis-EMA < UDMA < TEGDMA (Yu p. et al., 2017). The three experimental composites used in this study contained UDMA resins as the main monomer; as a result, all the experimental groups have a low degree of conversion.

AlShaafi and Maan in 2017 reported that the DC is determined mainly by how much light can get through a composite material. Penetration of light is affected by the filler-resin system. The Microfilled composite has the lowest microhardness and DC values, followed by the Macrofilled type. Since the Tetric N Flow have a filler size range between 0.1µm & 30µm, it is suspected to be that this smaller size filler is the cause of lower DC compared to the two other types (SDR with filler size 4 µm, and no information available about filler size of opus bulk-fill flow). Since there is little evidence about these new types of composite, it needs more research.

Ideally, any dental composite should convert all its monomers into polymers during the polymerization reaction, but this does not occur in all cases. It was reported that the higher the degree of conversion, the higher the strength of the resin matrix (Borges et al., 2013). However, the minimum amount of DC needed for a restoration to be clinically satisfactory has not been established. Galvo et al., 2013 found a negative correlation between abrasive wear and DC value when the last one is between 55% and 65%. However, these studies have recorded the degree of convergence for conventional composite resins but not flowable bulk-fill composite. In this study, all the values recorded were below the mentioned ideal level of DC. Low-viscosity bulk-fill materials with lower mechanical properties are suggested to be used as an internal layer in contact with cavity walls and then covered with a layer of conventional composite (Comba et al., 2020; Haugen et al., 2020). According to this study's results, all three flowable bulk-fill composite resins showed lower DC at the bottom surfaces, and the values decreased by increasing the light tip distances from the top surface. Hence, a 4mm thickness cured for the 20s with a light intensity of 1000-1200 mW/cm^2 was insufficient to obtain similar top-to-bottom parameters, especially when it is cured with a higher than 0mm light tip distance. One of the possible causes is that the time of curing used in this study (the 20s) was insufficient to reach the recommended thickness (4mm). Researchers suggested adding curing time for SDR application into deep cavities (Zorzin et al., 2014). Increasing the photo-activation time may activate the initiator and make free radicals that start the polymerization, which synergistically affects the polymerization rate. This way, the photo-activation time can be lengthened to get a higher conversion rate. (Al-ahdal et al., 2015, Tsuzuki et al., 2020). Increasing the irradiation duration can positively impact the degree of convergence of bulk-fill composite resins.

5. Conclusion

Within the limitations of this study, it can be concluded that:

1. For the three tested flowable bulk-fill composite materials, increasing the curing distances from 0mm to 2 to 4mm, decreased the DC% for the bottom surfaces more than the top surfaces.
2. For each material, the DC values were significantly higher at 0mm than 4mm light tip distance for the top and bottom surfaces, except for Tetric N on the bottom side. The difference was not significant.
3. Opus flow had the highest DC with significant differences with SDR and Tetric N flow at both the top and bottom sides.

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