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Original Article

The effect of depth of cure on microhardness between bulk-fill and hybrid composite resin material

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ABSTRACT

Background: Functional properties should be considered when selecting composites for restorations. With bulk-fill resin composites, the disadvantages of conventional composites were overcome regarding the increments necessitating the application of a 2 mm thick layer of resin and the amount of polymerization. The most significant advantage of bulk-fill resin composite is the possibility of applying the material in 4 mm layers. Multiple studies have examined the polymerization, mechanical, and adhesive properties of bulk-fill resin composites and proven them successful.
Objective: This study aimed to compare the effect of the depth of cure on the microhardness between different bulk-fill composites and a hybrid composite material by following the manufacturer's instructions.
Methods: In this in vitro study, five composite types obtained from different companies were used: two high-viscosity bulk-fill composites, Filtek and OPUS bulk; two low-viscosity bulk-fill flowable composites, Any-com and OPUS flow; one light-cure hybrid composite, Cavex, totaling 25 samples. The composite was applied at a depth of 4 mm in the bulk-fill composite and 2 mm in the hybrid composite and light-cured. The microhardness was measured at three different times. The first time was performed immediately after light-curing, the second time was evaluated 24 h after polymerization, and the third time was after thermocycling.
Result: A total of 25 samples showed that the microhardness increased the first and second times and decreased the third time after thermocycling on the top and bottom sides of Filtek, OPUS bulk-fill, OPUS flow, and Any-com bulk. The Cavex microhardness at the bottom was equal at all three-time points. Two-way repeated measures ANOVA showed that the microhardness between all types of composite resin materials was significantly different at different times when $p = 0.00$.

1. Introduction

Composite restoration material was first used in dentistry in the 1960s by Bowen's invention of Bis-GMA (Kelić et al., 2016). Functional properties, including improved restoration durability with excellent mechanical characteristics, such as high strength, fracture toughness, surface hardness, optimized elastic modulus, low wear, low water solubility, and low polymerization shrinkage, should be considered when selecting composites for restorations (Ilie and Hickel, 2011). Composites are frequently used in dentistry owing to increasing knowledge and patient desire for more aesthetic restorations (Melo et al., 2019; Dugar et al., 2022).

Essential requirements for conventional composite include a dry surface, crucial etching, priming, bonding procedures, and a maximum incremental thickness of 2 mm; the most significant disadvantage is the polymerization shrinkage and the depth of cure, limited to approximately 2 mm (Kelić et al., 2016; Reis et al., 2017).

Polymerization shrinkage occurs by transforming the weak van der Waals forces between monomers into covalent bonds, thus reducing the distance between them (Kim et al., 2015), and various elements of fillers, including the quantity, type, and size, influence shrinkage (Abbasi et al., 2018). As fewer monomers are available for the curing process when more fillers are present in the resin matrix, the total shrinkage of composite resins is often minimized (Abbasi et al., 2018). The elastic modulus of the material may increase, resulting in significant shrinking stress (Abbasi et al., 2018), and the resulting gap causes microleakage, increasing the risk of post-restoration hypersensitivity, undesirable pulpal effects, marginal discoloration, recurrent caries, and

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enamel cracking (Kaisarly and Gezawi, 2016).

Resistance to penetration or permanent surface indentation is known as microhardness, a measure of resistance to plastic deformation. Microhardness was determined by dividing the force by the area of the indented surface. In this regard, the Vickers test is among the most used hardness test (Saati et al., 2022). Given the correlation between increasing hardness and curing depth, it is crucial to evaluate the curing depth of composite resins by measuring their hardness (Saati et al., 2022). Furthermore, the microhardness measurements for a particular material also reveal information about its wear, polishability, and abrasive effect on the teeth (Kelić et al., 2016).

The bulk-fill resin composites overcame the disadvantages of conventional composite regarding the increments necessitating 2 mm increment application and the amount of polymerization; the most significant advantage of bulk-fill is the possibility of applying the material in a 4 mm layer (Nascimento et al., 2018).

Bulk-fill resin composites are divided into two categories: those with better mechanical qualities, such as high viscosity and wear resistance, allowing them exposure to the oral cavity without any coverage, and those that need to be used as a base or liner, usually low viscosity/flowable; however, because of their low wear resistance due to their low filler content, the top of the bulk-fill must be coated with bulk-fill and conventional composites (Rizzante et al., 2019; Arbildo-Vega et al., 2020).

Multiple studies examining the polymerization, mechanical, and adhesive properties of bulk-fill resin composites demonstrated that these bulk-fill composites are successful (Akgül et al., 2021). Failure in these studies to follow the manufacturer's instructions regarding the curing time and exceeding the recommended time posed a limitation; hence, the practitioner must follow the manufacturer's guidelines for the curing procedure and maximum incremental thickness (Fan et al., 2002; Bucuta and Ilie, 2014).

This study aimed to compare the effect of the depth of cure on the microhardness between different bulk-fill composites and one hybrid composite material by following the manufacturer's instructions.

2. Materials and method

Five types of composites used in this in vitro study were two high-viscosity bulk-fill composites, two low-viscosity bulk-fill flowable composites, and one light-curing hybrid composite. Different companies shade A1, and each composite type had five samples totaling 25 samples. Table 1 shows the characteristics of composite resins.

Two customized molds were fabricated. As shown in Fig. 1**,** the first mold, measuring 5 mm in diameter and 4 mm in depth, and the second mold, measuring 5 mm in diameter and 2 mm in depth, were used to produce composite resin samples. An open-ended cavity at the mold base allowed for composite material placement. The base of the mold was created to adapt and support the table of the microhardness machine. Bulk-fill composite was applied at a depth of 4 mm and hybrid composite (controlled) at 2 mm depth in each sample and light cured using an LED light-curing unit (BluePhase) at a constant distance of 1 mm from the tip of the light curing unit as per the manufacturer's

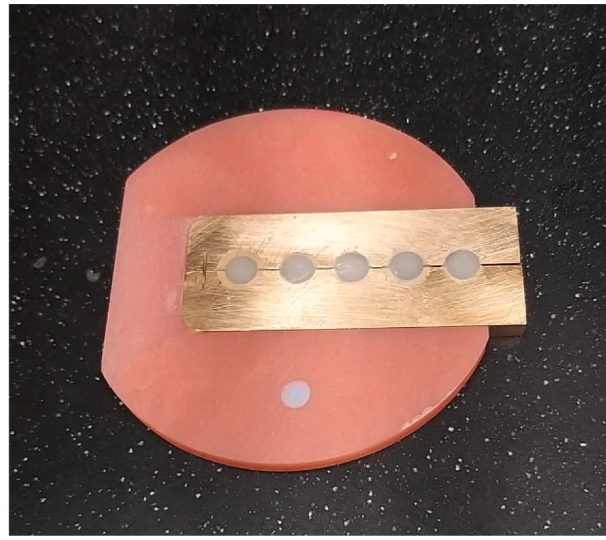

Fig. 1. Customized mold.

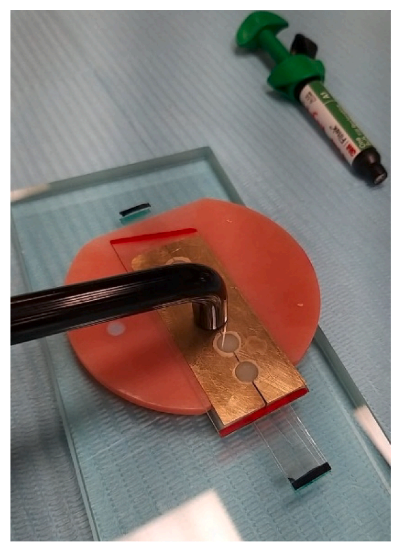

Fig. 2. During the curing procedure

Table 1
Characteristics of bulk-fill and conventional composite resins in this study.

Commercial Brand	Type of Composite	Manufacturer	Composition	Lot numbers
Any-Com™ Bulk	Low-viscosity bulk-fill flowable composite	Gongdan-ro, Heungdeok-gu, Cheongju-si, Chungbuk, Korea	– Modified methacrylate resin – Inorganic filler	EB202130711
Opus Bulk Fill Flow APS	Low-viscosity bulk-fill flowable composite	FGM, Joinville, SC, Brasil	– Urethane dimethacrylate monomers – Photoinitiators and APS co-initiators -silicon dioxide (silica), stabilizers, pigments	260,522
3 M™ Filtek™ One Bulk Fill Restorative	High-viscosity bulk-fill composite	3 M ESPE, St. Paul, MN, USA	– AFM (dynamic stress-relieving monomer), AUDMA, UDMA, and 1, 12-dodecane-DMA.	NE19412
Opus Bulk Fill APS	High-viscosity bulk-fill composite	FGM, Joinville, SC, Brasil	– Urethane dimethacrylate monomers – Photoinitiators and APS co-initiators -silicon dioxide (silica), stabilizers, pigments	200,522
Quadrant universal lc (Cavex)	Hybrid composite	Cavex Holland BV, Fustweg Haarlem, The Netherlands	– Methacrylate-based monomers – Silica, silicate glass, and fluoride-containing fillers – Polymerization catalysts – Inorganic pigments	K010740

instructions. The material is secured with a celluloid strip at the bottom and top and pressed with a glass slide during the curing procedure Fig. 2. Subsequently, the micro-hardness was assessed for each side of the sample the top side directed toward the light cure and the bottom side, which was the deepest and furthest from the light curing unit, using micro Vickers and Knoop hardness machine. The specimens were loaded into the machine for scanning with a 200 g load and 15 sec dwelling time.

The microhardness was measured at three different times. The first time was performed immediately after light-curing, the second time was 24 h after polymerization, and the third time after 10,000 cycles of thermocycling at a dwell time of 30 s per cycle, simulating one year of clinical usage.

2.1. Statistical analysis

The analysis was performed using SPSS version 26.0 (IBM Inc., Chicago, IL, USA) statistical software. Multiple comparison tests (Dunnett's test, T3) were used to compare the microhardness of the composites at each of the three different times separately for the top and bottom sides, and the Welch test was used to calculate the p-value of the microhardness of the composites at each of the three different times separately. Repeated measurements were performed to compare the microhardness of each type of composite separately at three different time points on the top and bottom sides. Two-way repeated-measures measurement ANOVA was used to measure the microhardness of each type of composite separately for the top and bottom sides at three different times and to compare the microhardness between the groups.

3. Results

A total of 25 samples showed that the microhardness increased at the first and second time points and decreased at the third time point after thermocycling on sides 1 and 2 for the Filtek, OPUS bulk-fill, OPUS flow, and Any-com bulk, as shown in Tables 2 and 3. For Filtek side 1 at time 1, the mean was 40.84, and for side 2 at time 1, the mean was 40.01; for the OPUS bulk-fill, side 1 at time 1, the mean was 57.15 and side 2 time 1, the mean was 41.45; for the OPUS flow, side 1 time 1, the mean was 22.96 and side 2 time 1, the mean was 13.42; for the Any-com bulk-fill, side 1 time 1, the mean was 29.85 and side 2 time 1, the mean was 26.64, indicating that the microhardness in side 1 is larger than in side 2 for all types of bulk-fill resin material at all three times. Microhardness of Filtek and OPUS bulk-fill was larger than that of OPUS flow and Any-com bulk on sides 1 and 2 at three different times. The lowest microhardness was observed for the OPUS flow, especially on side 2, and the highest microhardness was observed for the OPUS bulk-fill, particularly on side 1, as shown in Fig. 3. For Cavex (controlled), as shown in Tables 2 and 3, the microhardness increased at the first and second times and decreased at the third time on side 1 only; however, for side 2, it was equal at all three different times, with a mean of 43. Dunnett's test showed that the microhardness of all types of composites at three different times for side 1 and side 2 were significantly different, except that the microhardness of Filtek and OPUS bulk-fill were not significantly different on side 1 at times 2 and 3. The Welch test showed that p = 0.00 when comparing the microhardness of the composites at each of the three different times separately for sides 1 and 2. Repeated measurements showed that p = 0.00 when comparing the microhardness of each composite type separately at three different times for sides 1 and 2. Two-way repeated measures ANOVA showed that p = 0.00 when comparing the microhardness in all different types of resin composite materials at three different times according to the manufacturer's instructions, as shown in Table 4.

4. Discussion

In this study, the microhardness of the composites at each of the three different times separately for the top and bottom sides was significantly different, except that the microhardness of Filtek and OPUS bulk fill was not significantly different on the top side at times 2 and 3. When comparing the microhardness, each type of composite at three different times were significantly different on the top and bottom sides. Microhardness of all types of composite resin materials was significantly different at different times, indicating that the curing depth of bulk-fill composite resins was comparable to that of hybrid composite resins (Saati et al., 2022).

The Filtek and OPUS bulk-fills (high-viscosity bulk-fills) showed higher microhardness than the OPUS flow and Any-com bulk-fills (low-viscosity bulk-fills). Filtek and OPUS bulk-fill composites are recommended for restoration and can be exposed on occlusal surfaces because they have sufficient mechanical properties to withstand masticatory function in a clinical setting (Rizzante et al., 2019). On comparing the bulk-fill low- and high-viscosity resin composites, the microhardness values were expectantly lower in bulk-fill low-viscosity resin composites, requiring a capping layer (Rizzante et al., 2019). The OPUS bulk-fill flow had the lowest microhardness in this study compared to other bulk-fill materials. Owing to their low microhardness, low-viscosity bulk fill materials should not be used without the capping layer since the masticatory forces would be too high for them (Kelić et al., 2016). Both viscosities of the bulk-fill material showed that the microhardness increased with the first and second times. The microhardness decreased for the third time after thermocycling at the top and bottom sides. Reduced polymerization in the deeper layers of restorations can weaken the material and make it more prone to clinical problems such as disintegration, increased cytotoxicity, discoloration, marginal defect susceptibility, decreased hardness, wear resistance, and bond strength (Mendonça et al., 2021). Furthermore, the Filtek Bulk Fill showed the greatest microhardness value on the bottom side while placed in a single 4 mm increment in depth, according to the manufacturer's instructions (Melo et al., 2019).

The Cavex (hybrid composite) in the control group showed increased microhardness with the first and second times and decreased microhardness with the third time on the top side only. For the bottom side, it was equal at all three different times, and because conventional resin composites demonstrated a sufficient depth of cure of up to 2 mm, it is not recommended to use them in large increments (Rizzante et al., 2019; Mendonça et al., 2021). As the depth increases, the resin hardness

Table 2
The microhardness of composite resins evaluated in this study at three different times for the top side.

Side	Material	T1			T2			T3			P-value
		Mean	Std. Deviation	Sig.[b]	Mean	Std. Deviation	Sig.[b]	Mean	Std. Deviation	Sig.[b]	
Side 1	Filltek	40.840	0.7642	c	62.087	1.0816	d	58.707	1.2186	d	0.000
	OPUS/bulk	57.153	0.7809	e	62.180	1.2852	d	59.320	1.2768	d	0.000
	OPUS/Flow	22.967	0.6149	a	31.300	0.4440	a	26.827	0.7878	a	0.000
	Any-com/bulk	29.853	0.8741	b	40.493	0.8455	b	38.973	0.6112	b	0.000
	CAVEX	49.200	0.8652	d	53.393	1.3280	c	51.053	0.7210	c	0.000
P-value			0.000			0.000			0.000		

Side 1: Top *a < b < c < d < e significantly at 0.05*.

Table 3
The microhardness of composite resins evaluated in this study at three different times for the bottom side.

Side	Material	T1			T2			T3			P-value
		Mean	Std. Deviation	Sig.[b]	Mean	Std. Deviation	Sig.[b]	Mean	Std. Deviation	Sig.[b]	
Side 2	filtek	40.013	1.1630	c	58.407	0.9632	c	50.793	1.2314	c	0.000
	OPUS/bulk	41.453	1.0084	d	48.307	1.3977	d	47.367	1.0376	d	0.000
	OPUS/Flow	13.427	0.4317	a	22.727	1.1529	a	18.727	0.5457	a	0.000
	Any-com/bulk	26.640	0.7557	b	35.167	1.0581	b	32.593	0.9684	b	0.000
	CAVEX	43.240	0.7529	e	43.273	0.7216	e	43.367	0.8837	e	0.000
P-value			0.000			0.000			0.000		

Side 2: Bottom *a < b < c < d < e significantly at 0.05*.

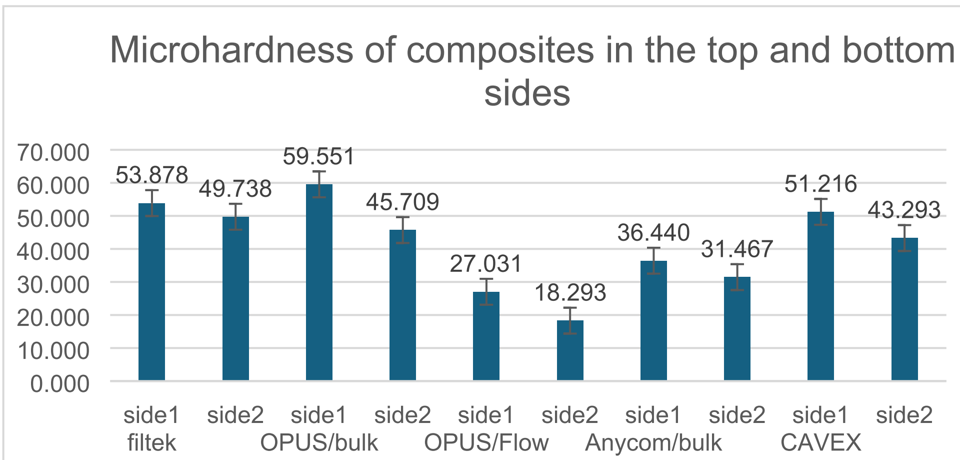

Fig. 3. Microhardness of composites in the top and bottom sides. *Side1: Top, Side2: Bottom*

Table 4
Comparing the microhardness for all resin composite evaluated in this study at three different times.

Comparing the microhardness of composite resins				
Material		Mean Difference	Standard Error	p-value
Filtek	OPUS/bulk	−0.822*	0.159	0.000
	OPUS/Flow	29.146*	0.159	0.000
	Any-com/ bulk	17.854*	0.159	0.000
	CAVEX	4.553*	0.159	0.000
OPUS/bulk	Filtek	0.822*	0.159	0.000
	OPUS/Flow	29.968*	0.159	0.000
	Any-com/ bulk	18.677*	0.159	0.000
	CAVEX	5.376*	0.159	0.000
OPUS/Flow	filtek	−29.146*	0.159	0.000
	OPUS/bulk	−29.968*	0.159	0.000
	Any-com/ bulk	−11.291*	0.159	0.000
	CAVEX	−24.592*	0.159	0.000
Any-com/ bulk	Filtek	−17.854*	0.159	0.000
	OPUS/bulk	−18.677*	0.159	0.000
	OPUS/Flow	11.291*	0.159	0.000
	CAVEX	−13.301*	0.159	0.000
CAVEX	Filtek	−4.553*	0.159	0.000
	OPUS/bulk	−5.376*	0.159	0.000
	OPUS/Flow	24.592*	0.159	0.000
	Any-com/ bulk	13.301*	0.159	0.000

Side1: Top, Side2: Bottom.

gradually decreases (Melo et al., 2019). Similarly, samples filled using the 2-increment procedure exhibited increased hardness at the bottom surface because of its proximity to the polymerization light of the first composite part (Melo et al., 2019).

Thermocycling is a standard procedure for evaluating the deterioration of dental materials over time. Cracks form when restorative materials are submerged in water, either with or without thermocycling, which can reduce flexural strength (Bahari et al., 2021). The thermal procedures used in this study adhered to the same guidelines as those used in the previous studies. The heat exchange cycles occurring in the oral cavity for approximately one year can correlate with 10,000 thermocycles. The thermal protocol can assess the behavior of materials subjected to heat stress, even if it does not replicate actual situations, such as those observed in the oral cavity (Bahari et al., 2021). Due to water absorption, a reduction in microhardness might be anticipated during thermocycling. Water weakens the structure of polymers by acting as a plasticizer (Tuncer et al., 2013). In addition, the silane–filler interface and filler–particle surface are hydrolytically broken down, immediately deteriorating the matrix–filler interface (Tuncer et al., 2013). This study showed that the microhardness decreased for the third time after thermocycling for all types of resin composites used in this study, except for the bottom side of Cavex, which remained the same as in the first and second times. One study found that laboratory composite resins were susceptible to decreased surface microhardness after thermocycling (Pereira et al., 2007).

This study supports following the manufacturer's instructions regarding using conventional composite in a 2 mm layering technique to enhance polymerization. As for the high viscosity bulk fill, it can be used in 4 mm layering depth due to its great microhardness, allowing it to resist the masticatory forces; the low viscosity bulk fill should always have the capping layer (Kelić et al., 2016). Practitioners must adhere to the curing method and maximum incremental thickness instructions provided by the manufacturer (Bucuta & Ilie, 2014).

One limitation of this study is that only five types of composites (two high-viscosity bulk-fill composites, two low-viscosity bulk-fill flowable composites, and one light-cure hybrid composite) were tested.

Therefore, we recommend experimenting with different types of composites produced by other companies.

5. Conclusion

Following the manufacturer's instructions, the conventional composite has good properties; however, the high-viscosity bulk-fill composite has a higher microhardness that can be used in a single layer without capping, and the low-viscosity bulk-fill flowable composite must be followed by a bulk-fill or conventional composite at the top layer.

Ethical approval

This article does not include any studies involving human participants or animals performed by the author. An exemption letter was provided by the Institutional Review Board (IRB) (No. E-23-8050).

CRediT authorship contribution statement

Ahmed A. Elhejazi: Conceptualization, Project administration, Supervision, Writing – review & editing, Validation. **Asim Alosimi:** Resources, Writing – review & editing, Writing – original draft. **Faisal Alarifi:** Resources, Writing – review & editing. **Abdullah almuqayrin:** Resources, Writing – review & editing.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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