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Evaluation of the Microhardness of Six Different Colored Flowable Composites Cured at Different Times with LED Light Device

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ABSTRACT

Objectives: Colored flowable composites are frequently used in pediatric dentistry to motivate children. Colored resin-based materials exhibit varying capacities for light absorption and reflection, which can significantly influence the effectiveness of the polymerization process. The aim of this study was to comparatively evaluate the surface microhardness of flowable composite resins in different colors (purple, blue, orange, pink, green, and yellow) after curing with an LED light device for 20 and 40 seconds.

Materials and Methods: In this study, flowable composites (Rainbow Flow, PPH CERKAMED, Poland) in purple, blue, orange, pink, green, and yellow colors were used. A total of 120 specimens were prepared, with 10 specimens for each color and curing time. The samples were placed in 5x2 mm disk-shaped molds and cured for 20 and 40 seconds using a VALO™ LED device (1000 mW/cm²). After polymerization, the specimens were polished using the Sof-Lex disc system and stored in distilled water at 37°C for 24 hours. Microhardness measurements were performed using a microhardness tester (Shimadzu Micro Hardness Tester HMV-2, Japan) with a 100 g load applied for 10 seconds. The average Vickers hardness value was calculated from five measurements per specimen. Data were analyzed using the Kruskal-Wallis and Mann-Whitney U tests ($p < 0.05$).

Results: Statistically significant differences in microhardness were found between different curing times and composite colors ($p < 0.001$). The purple composite showed the highest microhardness values for both curing times, while the orange composite exhibited the lowest. All groups cured for 40 seconds had significantly higher microhardness values than those cured for 20 seconds ($p < 0.05$). For 20 seconds, the highest average microhardness was recorded in the purple group (24.69 ± 3.43), and the lowest in the orange group (17.71 ± 1.92). After 40 seconds of curing, the purple (28.34 ± 3.47) and blue (25.54 ± 3.42) composites had the highest values, while orange remained among the lowest (21.24 ± 2.53).

Conclusions: In conclusion, microhardness increased with extended curing time for all colors. Additionally, darker colors such as purple and blue exhibited higher microhardness values regardless of curing duration. Further studies are required to investigate the physical properties of colored flowable composites. Curing time and composite color should be considered together to optimize clinical outcomes. Such studies may help establish accurate curing time recommendations for colored composites.

Keywords: Colored flowable composites , curing times, microhardness, led light.

LED Işık Cihazı ile Farklı Sürelerde Kürlenen Altı Farklı Renkli Akışkan Kompozitin Mikrosertliğinin Değerlendirilmesi

Araştırma Makalesi

Süreç

Geliş: 04/02/2025
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ÖZ

Amaç: Renkli akışkan kompozitler pediatrik diş hekimliğinde çocukları motive etmek için sıklıkla kullanılmaktadır. Renkli rezin bazlı materyaller, polimerizasyon sürecinin etkinliğini önemli ölçüde etkileyebilen ışık emilimi ve yansıması için farklı kapasiteler sergilemektedirler. Bu çalışmanın amacı, farklı renklerdeki (mor, mavi, turuncu, pembe, yeşil ve sarı) akışkan kompozit rezinlerin LED ışık cihazı ile 20 ve 40 saniye süreyle kürlenmeleri sonrası yüzey mikrosertliklerini karşılaştırmalı olarak değerlendirmektir.

Gereç ve Yöntemler: Bu çalışmada mor, mavi, turuncu, pembe, yeşil ve sarı renklerde akışkan kompozitler (Rainbow Flow, PPH CERKAMED, Polonya) kullanılmıştır. Her renkten, her kürleme süresi için 10'ar adet olmak üzere toplam 120 örnek hazırlanmıştır. Örnekler 5x2 mm disk kalıplara yerleştirilerek, kürleme işlemi VALO™ LED cihazı (1000 mW/cm²) ile 20 ve 40 saniye süreyle gerçekleştirilmiştir. Polimerizasyon sonrası örnekler Sof-Lex disk sistemi ile parlatılmış ve 24 saat boyunca 37°C'de distile suda bekletilmiştir. Mikrosertlik ölçümleri, mikrosertlik test cihazı (Shimadzu Micro Hardness Tester HMV-2, Japonya) kullanılarak, numunelere 10 saniye boyunca 100 gram yük uygulanmıştır. Her numuneden 5 ölçüm alınarak ortalama Vickers sertlik değerleri hesaplanmış ve elde edilen veriler Kruskal-Wallis and Mann-Whitney U testleri ile analiz edilmiştir ($p < 0.05$).

Bulgular: Farklı kürleme süreleri ve renkler arasında mikrosertlik açısından istatistiksel olarak anlamlı bir fark bulunmuştur ($p < 0.001$). Mor akışkan kompozitin tüm kürleme sürelerinde sayısal olarak daha yüksek bulunurken, en düşük mikrosertlik değeri ise turuncu renkte gözlenmiştir. 40 saniye kürlenen gruplardaki tüm renklerin mikrosertlik değerleri, 20 saniye kürlenen gruplardaki mikrosertlik değerlerinden istatistiksel olarak daha yüksek bulunmuştur ($p < 0.05$). 20 saniyelik kürlemede en yüksek ortalama mikrosertlik değeri mor renkte (24.69 ± 3.43), en düşük ise turuncu renkte (17.71 ± 1.92) ölçülmüştür. 40 saniyelik kürlemede mor kompozit (28.34 ± 3.47) ve mavi kompozit (25.54 ± 3.42) istatistiksel olarak en yüksek değerlere ulaşmıştır. Turuncu kompozitin 40 saniyedeki değeri 21.24 ± 2.53 olup, yine en düşük mikrosertlik değerlerinden biri olmuştur.

Sonuçlar: Özetle, mikrosertliğin tüm renkler için kürleme süresinin artmasıyla artış gösterdiği belirlenmiştir. Bununla birlikte, mor ve mavi gibi koyu renklerin mikrosertlik değerleri daha açık renklere kıyasla kürleme süresinden bağımsız olarak daha yüksekti. Renkli akışkan kompozitlerin fiziksel özelliklerini araştırmak için daha fazla çalışma yapılması gerekmektedir. Kompozitlerin klinik başarısında kürleme süresi ve kompozit rengi birlikte ele alınmalıdır. Bu tür çalışmalar, renkli kompozitler için kürleme sürelerinin doğru bir şekilde önerilmesine olanak sağlayacaktır.

Anahtar Kelimeler: LED ışık, mikrosertlik, renkli akışkan kompozitler.

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Introduction

A wide variety of materials have been used for preventive and restorative purposes in pediatric dentistry from past to present. Composite resins, which are one of the most commonly used restorative materials for the restoration of primary teeth, structurally consist of inorganic fillers and a bonding agent within a resin matrix.[1-3] Depending on the size of its constituents, the physical and mechanical properties of a material vary.[4] Flowable composite resins, which are somewhat different from traditional composite formulations, were first introduced in 1996. The filler particle content of flowable composite resins is 20-30% lower than that of conventional hybrid composite resins, while the amount of monomers such as Triethylene Glycol Dimethacrylate (TEDGMA) has been increased. Flowable composite resins, which have a lower filler content, also have lower viscosity and elastic modulus. Due to their viscosity, they spread well on cavity walls, adapting effectively, and can be used in many types of cavities.[1,2] Their flowable nature allows them to be packaged in syringes, making them easier to apply in small cavities.[4]

Since the different color options in restorative materials are appealing to pediatric patients, they help increase children's motivation during dental treatment and reduce the level of stress they experience.[5-7] Nowadays, flowable composite resins in various colors have been introduced to the market, specifically for use in pediatric patients. Manufacturers have suggested that colored flowable composites can be used in Class 1, 2, and Class 5 cavities in primary and permanent teeth, as fissure sealants, and for sealing canal openings. However, studies have indicated that due to the low mechanical properties of flowable composite resins, they should be used with caution in areas subjected to high forces.[8]

The polymerization process is crucial for enhancing the mechanical and physical properties of resin-containing dental restorative materials, thereby improving their clinical performance.[9] Factors such as cavity depth, the thickness of the resin material applied, the distance between the light source tip and the resin, exposure time, the type of light source, light intensity, the diameter of the light tube, and the color and transparency of the restorative material all play a role in the polymerization of resin-based materials.[10-12] Studies have shown that darker shades of composite resins may reduce the degree of curing, leading to a decrease in material hardness, while the composition and color of the material may also affect the degree of polymerization by influencing light transmission.[13] The duration of light exposure is also among the key factors for achieving proper polymerization. The appropriate light exposure times for light-curing units and composite resins remain a topic of ongoing research today.[14]

Various methods are used to evaluate the degree of polymerization, with surface microhardness being the most commonly employed method.[15] Factors such as color, transparency, and the type of material can affect surface microhardness.[16] Studies examining color and microhardness have reported that darker shades of composite resins exhibit higher microhardness values compared to lighter shades, according to the Vickers hardness scale.[17,18] While there are various studies in the literature concerning the microhardness of tooth-colored resins, the number of studies related to colored resins is limited.[18,19]

The objective of this study is to assess the impact of different curing times, employing an LED light, on the surface microhardness of six different colors (purple, blue, orange, pink, green, and yellow) of flowable composites.

Materials and Methods

Ethical approval was obtained from the Afyonkarahisar Health Sciences University Clinical Research Ethics Committee (decision number 2024/02).

In this in vitro study, six different colors (purple, blue, orange, pink, green, and yellow) of flowable composite (Rainbow Flow, PPH CERKAMED Wojciech Pawlowski, Poland) were used. The number of samples required for the study was calculated using the G.Power program. Based on the power analysis, $\alpha<0.05$, $1-\beta$ (power)=0.80, 120 samples were prepared, 10 samples for each curing time from each color.

Metal disc molds with a diameter of 5 mm and a thickness of 2 mm were used to prepare the specimens. They were compressed between two glass slides to prevent the formation of air bubbles on both the top and the bottom surfaces of the mold and to remove excess flowable resin. This was aimed at preventing the formation of the oxygen inhibition layer. After the glass slides were removed, the light device's tip was positioned directly in contact with the molds and perpendicular to achieve an equal distance standard for each composite resin sample and to obtain the best polymerization depth. The composite resin samples were set up so that light would only be applied to the upper surface, while the bottom surfaces were marked with a waterproof pen. Each color of flowable composite was polymerized in two separate groups for 20 and 40 seconds curing times in standard power mode (1000 mW/cm²) using a VALO™ LED unit (Ultradent, South Jordan, USA), held in tight contact with the glass slides at equal distances. Upon completion of the polymerization process, the specimens were removed from the metal disc molds without deformation and each specimen was post-treated and polished using the Sof-Lex disc system (3M, Dental Products, USA). The samples were then stored in a distilled water solution at 37°C for 24 hours in light-proof containers with different compartments. The microhardness analyses of the prepared composite resin samples were conducted at Afyon Kocatepe University Technology Application and Research Center using a microhardness measurement device (Shimadzu Micro Hardness Tester HMV-2, Shimadzu Corporation, Kyoto, Japan). A pyramid diamond tip in the Vickers microhardness measurement device was applied with a load of 100 g for 10 seconds, and five measurements were taken from the upper surfaces of the composite resin samples. The marks left on the samples were evaluated at 40x magnification to obtain the Vickers microhardness values. The measurement points were determined such that one was at the center and the other four were equidistant from the center. A single value was obtained by averaging the five measurements for each composite resin sample. Additionally, visual records of the samples were taken by examining the microscopic images with the microscope of the microhardness measurement device.

The statistical analysis of the study was performed using IBM SPSS Version 26 (Statistical Package for Social Science, IBM SPSS®, Chicago, U.S.A.). The microhardness values of the flowable composites in different colors under two different curing times were evaluated using the Kruskal-Wallis H test. The microhardness values of each color of flowable composite according to curing times were assessed using the Mann-Whitney U test. The significance level was set at $p < 0.05$.

Results

In this study, six different colored flowable composites were grouped for 20 and 40 seconds curing and compared according to their colors and different curing times of each color statistically.

A statistically significant difference was identified in the microhardness of composites with both 20 s and 40 s curing time according to their colors ($p < 0.001$).The microhardness of the purple colored flowable composite was found to be numerically higher than the other colors when cured for both 20 and 40 seconds. However, the microhardness of the orange colored flowable composite was found to be numerically lower than the other colors when cured for both 20 and 40 s. The values of microhardness according to colors were shown in Table 1.

A statistically significant difference in microhardness values was identified among the flowable composites at a curing time of 20 seconds (Figure 1). Specifically, the purple flowable composite exhibited a higher microhardness value compared to the blue, pink, and orange flowable composites, with a p-value less than 0.05.

A statistically significant difference in microhardness values was observed among the flowable composites at a curing time of 40 seconds (Figure 2). Specifically, the microhardness values of the purple and blue flowable composites were found to be higher than those of the pink and orange flowable composites ($p < 0.05$).

A subsequent comparison of microhardness values according to curing times revealed that the microhardness values of all colors in the groups cured for 40 seconds were found to be statistically significantly higher than those in the groups cured for 20 seconds ($p < 0.05$, Table 2).

Table 1. Comparison of microhardness of colored flowable composites according to their colors

Curing time	Color of flowable composite	Mean ± SD	Median (minimum – maximum)
20 second	Purple	24.69 ± 3.43	25.10 (19.43 – 28.89)[a]
	Blue	18.95 ± 1.53	18.70 (16.90 – 21.35)[b]
	Pink	19.14 ± 1.23	19.31 (16.00 – 20.28)[ab]
	Yellow	18.04 ± 0.63	18.07 (17.17 – 19.10)[b]
	Orange	17.71 ± 1.92	16.62 (15.90 – 20.13)[b]
	Green	19.61 ± 1.29	19.53 (18.34 – 21.32)[ab]
40 second	Purple	28.34 ± 3.47	29.02 (22.09 – 31.78)[A]
	Blue	25.54 ± 3.42	23.90 (22.51 – 31.10)[A]
	Pink	20.72 ± 1.43	20.16 (18.90 – 22.90)[BC]
	Yellow	21.88 ± 1.31	21.74 (20.20 – 24.01)[ABC]
	Orange	21.24 ± 2.53	19.99 (19.80 – 25.99)[C]
	Green	21.83 ± 1.42	22.20 (20.04 – 24.04)[ABC]

SD: Standard Deviation

[a-c, A-C]Each subscript letter denotes a subset of colorful flowable composite categories whose column proportions do not differ significantly from each other at the 0.05 level.

Table 2. Comparison of microhardness of colored flowable composites according to curing times

Flowable composite color	20 second		40 second		P value
	Mean ± SD	Median (minimum – maximum)	Mean ± SD	Median (minimum – maximum)	
Purple	24.69 ± 3.43	25.10 (19.43 – 28.89)	28.34 ± 3.47	29.02 (22.09 – 31.78)	0.023
Blue	18.95 ± 1.53	18.70 (16.90 – 21.35)	25.54 ± 3.42	23.90 (22.51 – 31.10)	<0.001
Pink	19.14 ± 1.23	19.31 (16.00 –20.28)	20.72 ± 1.43	20.16 (18.90 – 22.90)	0.029
Yellow	18.04 ± 0.63	18.07 (17.17 – 19.1)	21.88 ± 1.31	21.74 (20.20 – 24.01)	<0.001
Orange	17.71 ± 1.92	16.62 (15.90 – 20.13)	21.24 ± 2.53	19.99 (19.80 – 25.99)	0.029
Green	19.61 ± 1.29	19.53 (18.34 – 21.32)	21.83 ± 1.42	22.20 (20.04 –24.04)	0.031

SD: Standard Deviation

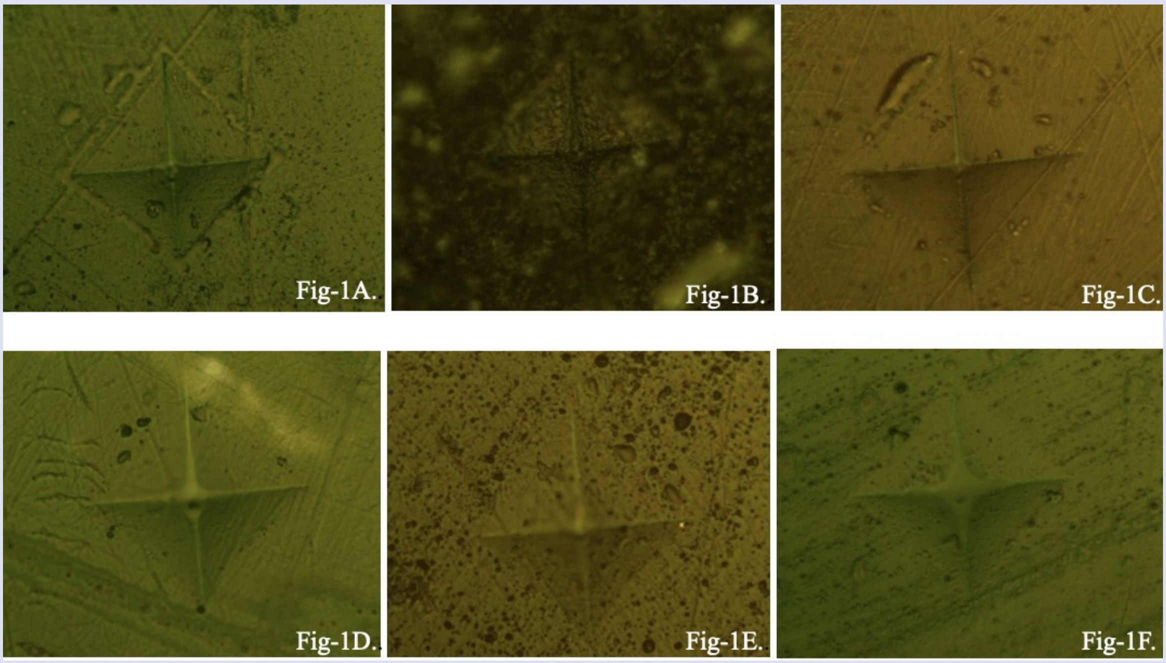

Figure 1. Microscopic images of colored flowable composites at 20 seconds of curing time. (Fig = Figure) Fig-1A: Blue color, Fig-1B.: Purple color, Fig-1C.: Pink color, Fig-1D.: Yellow color, Fig-1E.: Orange color, Fig-1F.: Green color.

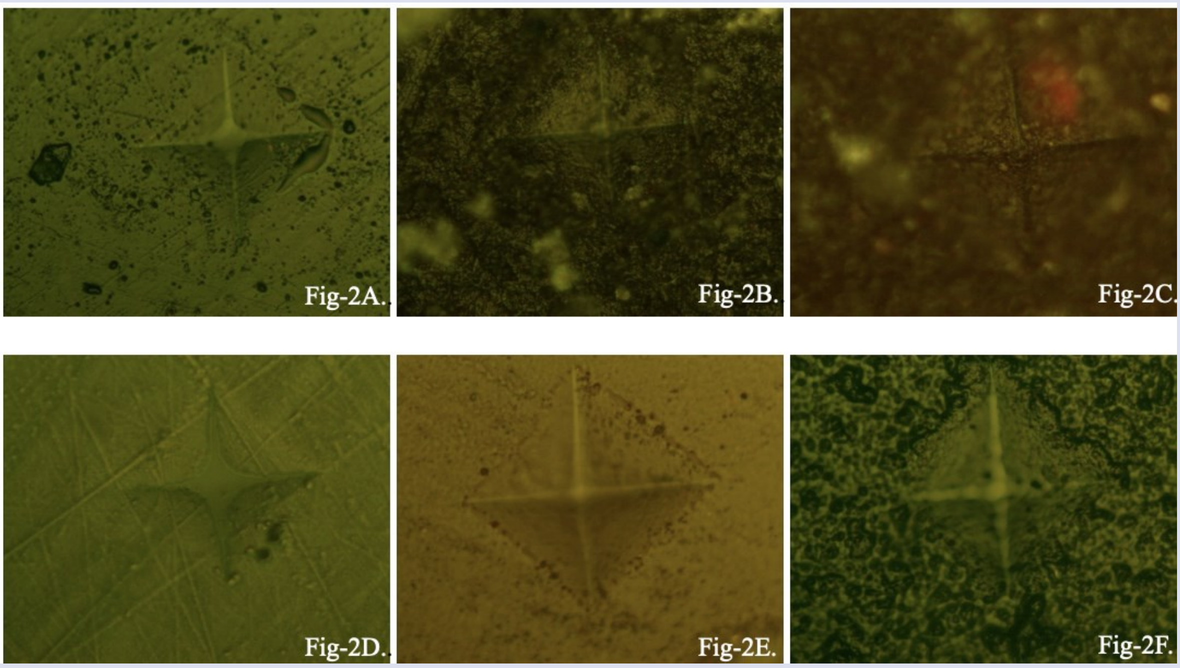

Figure 2. Microscopic images of colored flowable composites at 40 seconds of curing time.(Fig=Figure)Fig-2A.: Blue color, Fig-2B.: Purple color, Fig-2C.: Pink color, Fig-2D.: Yellow color,Fig-2E.: Orange color, Fig-2F.: Green color.

Discussion

Surface microhardness of composites is one of the indicators of polymerization efficiency and is evaluated through microhardness measurements under in vitro conditions[20]. The color and translucency of composites can influence the polymerization process and consequently, the microhardness of the material. Various resin composites with different color ranges developed by manufacturers are available on the market. Studies have emphasized that the color and translucency of these resin composites affect the transmission of light through the material. In the present study, the effect of different curing times on the microhardness of flowable composite materials in six different colors was investigated.

One of the important mechanical properties of materials tested is the surface hardness of dental restorations. Surface hardness is one of the important properties indicating whether the material is strong enough to resist masticatory forces, abrasion and orthodontic forces.[21] Among the most commonly used methods for assessing the degree of polymerisation are surface hardness tests.[15]

Insufficient polymerisation of the material may result in residual monomers, which may pose a problem for pulp health and general health.[22] In the context of polymerization, the utilized light source constitutes a pivotal factor. In the present study, the VALO™ (Ultradent, South Jordan, USA) LED light device was operated in standard power mode (1000 mW/cm²). As demonstrated in earlier studies, increasing the distance between the light source and the material has been shown to reduce the surface hardness of the material. To ensure standardization in the present study, the light source was applied perpendicularly at the closest possible distance to the samples.[23]

Curing time is a critical factor influencing polymerization and, consequently, surface hardness. The literature reports that increasing the curing time significantly enhances surface hardness.[24,25] In a study that sought to explore the impact of curing time and distance on the surface hardness of compomers across a range of colors, an examination of surface hardness values was conducted after 20 and 40 seconds of curing. The findings indicated that the surface hardness values in the 20-second group were found to be statistically significantly lower in comparison to those in the 40-second group.[26] Similarly, in this study, when microhardness values were compared based on curing times, the microhardness values of all color groups cured for 40 seconds were significantly higher than those cured for 20 seconds.

Factors that cause inadequate polymerisation of resin materials include the colour of the material and insufficient light exposure.[27] Hwang et al.[16] examined under SEM that the distribution of light transmission can be affected by the glitter particles and the glitter particles contain irregular shapes and sizes that can affect the hardness value of the material. Therefore, they suggested that compomers with different colours of sparkle particles and colour pigments may exhibit different hardness values. Vandenbulcke et al.[28] examined the light penetration depth of composites and compomers polymerized in various colors and the lowest penetration depth was observed in pink and gold, while the highest was found in blue. Another study evaluating the effect of different curing lights (QTH and LED) on the surface hardness of compomers (Twinky Star®) of different colors reported that the lowest hardness was observed in the gold colored compomer with both light sources.[29] Similarly, Bakkal et al.[21] found that the gold colored compomer demonstrated lower cure, surface hardness, and mechanical properties compared to the mulberry and silver colored compomers. In line with these findings, the current study also found that the microhardness of darker-colored composites, such as blue and purple, was higher. The observed outcomes can be attributed to the fact that light scatters more in light-colored restorative materials, while it is better absorbed in darker-colored restorative materials.[16]

In contrast to the results of the present study, Jafari et al.[30] reported in their research using LED units that the lowest surface hardness among colored compomers was observed in the blue color. This result suggests that darker pigments may absorb more light, thereby reducing the depth of light penetration into the resin. Additionally, since the blue light emitted by the curing device matches the color of the blue compomer samples, it may cause light scattering. In contrast, pink and white pigments create a complementary combination with blue light, resulting in higher light transmission, which could explain these differences.[16] The differences in microhardness values observed among the various colored flowable composite materials used in this study may be attributed to the different levels of light absorption by each color, the organic or inorganic nature of the color pigments, and the non-homogeneous type and amount of pigments within the different colored composites.[16,29,31] Additionally, it is believed that factors such as sample size, curing time and source, the type of microhardness measurement device, and variations in in vitro test conditions may also have influenced the microhardness of the materials.

Another study found that in darker resin materials, light transmission to the underlying layers is weaker compared to lighter-colored materials, resulting in lower hardness values for the bottom surfaces of darker resins.[32,33] This assertion is corroborated by the findings of Pires et al., who reported that initial exposure to light exerts a immediate effect on the photosensitive molecules located on the surface, thereby initiating polymerization. However, this polymerization process hinders further light transmission, preventing the underlying layers from receiving sufficient light.[24] In a parallel manner, the present study, the top surface hardness of the darker composite samples was found to be higher. However, the fact that the microhardness of the bottom surfaces was not evaluated is a limitation of the present study.

In a present study by Atabek et al.[19] which investigated the degree of cure of different colored compomers (Twinky Star®) using LED units, it was found that the blue and pink colors exhibited higher polymerization and hardness levels than gold, green, lemon, orange, and silver after 40 seconds of curing. The differences in polymerization and hardness were explained by variations in the size, type, and quantity of glitter components present in the materials.

In this study evaluating colored composites, similar to Atabek et al.'s findings, the highest microhardness value was observed in the blue-colored compomer. However, different results were obtained for the pink-colored compomer. Hwang et al.[16] suggested that the distribution of light transmittance could be affected by the irregular shape and size of the glitter particles embedded within the material, which, in turn, may influence the material's hardness. They emphasized that compomers with different glitter particles and color pigments might exhibit varying hardness values, even across different colors. These findings indicate that light does not distribute homogeneously within the material. Therefore, the differences between studies could be attributed to the variability in the composition, filler ratios, and the specific pigments used, even if the materials share the same color.

One of the limitations of the study is the uniformity of the specimen surfaces. It should also be noted that flat specimen surfaces were used in the present study and in clinical practice, restorations created with resin composites consisting of convex or concave irregular structures may cause differences in polymerisation. Therefore, clinicians should consider that the optical and mechanical properties of the material to be used before restoration may change in the long term.[34]

The literature shows that studies on colored restorative materials used in pediatric dentistry have generally focused on colored compomers, with relatively few studies on colored flowable composites.[16] There is no accessible literature that specifically investigates the microhardness of colored flowable composites. The present study is a first in this regard and may provide insights for future research in this area.

Colored resin-based materials exhibit varying capacities for light absorption and reflection, which can significantly influence the effectiveness of the polymerization process.[21] Inadequate polymerization reduces the surface microhardness of the material, compromising its mechanical durability and wear resistance, potentially leading to clinical failures.[35,36] In this study, both curing time and composite color were found to affect surface microhardness. These findings highlight the importance of adopting color-specific curing strategies when using colored flowable composites in clinical practice. Therefore, in addition to aesthetic considerations, the selection of restorative materials should be based on their physical properties and long-term clinical performance.

Conclusions

In summary, the present study identified substantial variations in microhardness values among flowable composites with different colors and curing times. Dark-colored flowable composites, such as blue and purple, exhibited statistically higher microhardness values regardless of curing time compared to other groups. It is crucial for clinicians to consider the curing times on a color-specific basis when using colored composites, as this can significantly impact restoration success. Further studies are needed to evaluate the physical properties and long-term clinical success of colored flowable composites, which are frequently used in pediatric dentistry to motivate children.

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Conflicts of Interest Statement

The authors have stated that they have not received financial support or experienced a conflict of interest. They have also indicated that they have no financial relationships with any company and no external funding.

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