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Effects of thickness and aging of bulk-fill composite resins on surface microhardness and fluorescence properties

Efeitos da espessura e envelhecimento de resinas compostas bulk-fill na microdureza superficial e propriedades de fluorescência

Efectos del espesor y el envejecimiento de las resinas compuestas de relleno em bloque sobre la microdureza superficial y las propriedades de fluorescência

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Abstract
Objective: Evaluation of various bulk-fill composite materials with respect to the microhardness values of the top and base surfaces upon variation in the composite thickness. The effect of aging and comparison of the fluorescence properties exhibited by the materials at different thicknesses will also be examined. Materials and Methods: For all samples, calculations were carried out using a family F probability, with a repeated family design including interactions within and among the factors, which resulted in 135 sample units, with 27 samples per experimental group. Z350, AURA, TETRIC, SDR, and FBF composites were employed with thicknesses of 2, 3, and 4 mm, initially being submitted to microhardness measurements (n=9). Aging was carried out using an aqueous ethanol solution. After aging the microhardness values of the samples were measured again, and a comparative analysis was performed for the fluorescence of all samples. Results: Aging, sample thickness, and the type of material were found to affect both the surface microhardness and the fluorescence values. In addition, the AURA and SDR bulk-fill composites were the least susceptible to differences in thickness in terms of the top and base surface microhardness values. Conclusions: Although the manufacturers of some bulk-fill composites recommend the use of material increments of 4 mm, the results of this study suggest that further research is required before clinical application.
Keywords: Composite resins; Chemical properties; Physical properties; Fluorescence.

Resumo
Objetivo: Avaliação de várias resinas compostas bulk-fill em relação aos valores de microdureza das superfícies superior e base mediante variação na espessura do compósito. O efeito do envelhecimento e a comparação das propriedades de fluorescência exibidas pelos materiais em diferentes espessuras também serão examinados. Materiais e Métodos: Para todas as amostras, os cálculos foram realizados usando uma probabilidade de família F, com um

desenho de família repetida incluindo interações dentro e entre os fatores, o que resultou em 135 unidades amostrais, com 27 amostras por grupo experimental. Os compósitos Z350, AURA, TETRIC, SDR e FBF foram empregados com espessuras de 2, 3 e 4 mm, sendo inicialmente submetidos a medidas de microdureza (n=9). O envelhecimento foi realizado usando uma solução aquosa de etanol. Após o envelhecimento os valores de microdureza das amostras foram medidos novamente, e uma análise comparativa foi realizada para a fluorescência de todas as amostras. Resultados: O envelhecimento, a espessura da amostra e o tipo de material afetaram tanto a microdureza superficial quanto os valores de fluorescência. Além disso, os compósitos bulk-fill AURA e SDR foram os menos suscetíveis a diferenças de espessura em termos dos valores de microdureza da superfície do topo e da base. Conclusões: Embora os fabricantes de alguns compósitos bulk-fill recomendem o uso de incrementos de material de 4 mm, os resultados deste estudo sugerem que mais pesquisas são necessárias antes da aplicação clínica.
Palavras-chave: Resinas compostas; Propriedades químicas; Propriedades físicas; Fluorescência.

Resumen
Objetivo: Evaluación de varios materiales compuestos de relleno en bloque con respecto a los valores de microdureza de las superficies superior e inferior al variar el espesor del compuesto. También se examinará el efecto del envejecimiento y la comparación de las propiedades de fluorescencia exhibidas por los materiales en diferentes espesores. Materiales y Métodos: Para todas las muestras, los cálculos se realizaron utilizando una familia de probabilidad F, con un diseño de familia repetida que incluye interacciones dentro y entre los factores, lo que resultó en 135 unidades de muestra, con 27 muestras por grupo experimental. Se emplearon composites Z350, AURA, TETRIC, SDR y FBF con espesores de 2, 3 y 4 mm, siendo sometidos inicialmente a mediciones de microdureza (n=9). El envejecimiento se llevó a cabo utilizando una solución acuosa de etanol. Después del envejecimiento, se midieron nuevamente los valores de microdureza de las muestras y se realizó un análisis comparativo de la fluorescencia de todas las muestras. Resultados: se encontró que el envejecimiento, el grosor de la muestra y el tipo de material afectan tanto la microdureza de la superficie como los valores de fluorescencia. Además, los composites de relleno en bloque AURA y SDR fueron los menos susceptibles a las diferencias de espesor en términos de los valores de microdureza de la superficie superior y de la base. Conclusiones: Aunque los fabricantes de algunos composites de relleno masivo recomiendan el uso de incrementos de material de 4 mm, los resultados de este estudio sugieren que se requiere más investigación antes de la aplicación clínica.
Palabras clave: Resinas compuestas; Propriedades físicas; Propriedades químicas; Fluorescência.

1. Introduction

Bulk-fill composite resins have recently been introduced in the dentistry market, with the aim of simplifying procedures and reducing the time required for the manufacture of restorations through the application of a single increment of 4–5 mm thickness. This is possible thanks to the high translucency of these materials, as light can reach to deep layers during the photoactivation process (Latta et al., 2020).

In cases where the cavities are larger than 5 mm, it is necessary to apply a conventional resin surface layer to replace the occlusal or vestibular enamel. An innovative polymerization initiation system that determines the shortening of the exposure time to light, while increasing the photopolymerization depth of the bulk-fill resin composites, was also observed. The low polymerization contraction and high inorganic load content enable the contraction stress to be reduced, thereby allowing the use of layers that are thicker than the conventional 2 mm layers (Ilie & Stark, 2014; Yao et al., 2020).

The effectiveness of the polymerization of composite resins to the deeper layers is related to the microhardness of the material. Manufacturers determine a suitable depth and thickness at a specific polymerization time be able to present acceptable resin composites microhardness standards. In terms of bulk-fill materials, many studies have concluded that they do not present the depth of polymerization reported by the manufacturers, with shallower depths than expected being observed (Garcia et al., 2014; Aggarwal et al., 2019).

Despite the greater translucency of bulk-fill resins, information regarding the fluorescence of these materials is scarce. As this property has makes an important contribution to esthetics by conferring vitality to the natural tooth, restorative materials would be expected to mimic this optical feature. Sunlight is the largest source of ultraviolet rays, although other black-light environments favor the chromatic perception of fluorescence. Although several brands of composite resins do not exhibit this property, various manufacturers have added luminophores from rare-earth metals to satisfactorily reproduce the fluorescence of natural teeth (Alkhurdhairy et al., 2020).

Bulk-fill composites can exhibit both high and low viscosities. High viscosity bulk-fillers are easy to handle, being thus suitable for restoring narrow access cavities. As such, they exhibit a higher bulk-fill content and similar handling characteristics to hybrid composites (Guiraldo et al., 2009; Kim, et al., 2015).

We therefore wish to evaluate a number of bulk-fill composites with respect to the variables of top and base microhardness upon variation of the composite thickness. In addition, the effect of aging and comparison of the fluorescence exhibited at different thicknesses will be examined.

2. Methodology

For all samples, calculations were carried out using a family *F* probability, with a repeated family design including interactions within and among the factors. An effect size of 0.15, type 1(α) error of 0.05, and analysis power of 0.92 were selected, which resulted in 135 sample units, with 27 samples per experimental group. GPower software (version 3.1.9.2, University of Düsseldorf, Germany) was used for the sample calculations. Each group (n=27) was subdivided into 3 subgroups of samples with thicknesses of 2, 3, and 4 mm initially being submitted to microhardness measurements (n=9). After aging in a solution of ethanol and water, the microhardness values of the samples were measured again, and a comparative analysis was performed for the fluorescence of all samples.

A total of 135 samples were prepared in 5 groups of each composite. Within each group, the samples presented depths of 2, 3, and 4 mm (n=9). The samples were prepared with the aid of a bipartite rigid steel matrix with a circular format (8 mm diameter).

Each steel matrix was supported on a polyester strip against a glass plate and filled with an increment of composite to prevent the inclusion of air bubbles. An additional polyester matrix strip was applied onto the composite and pressed with another glass plate for 10 s against the rigid steel matrix to flatten the sample. This plate was removed prior to photoactivation, which was carried out using LED light (Bluephase G1, irradiance: 1200 mW/cm^2, Ivoclar Vivadent) and standardization over 20 s for each material (Ilie & Stark, 2014).

The samples were identified and stored individually in hermetically-sealed plastic containers with 100% relative humidity at 37 °C in an oven for 24 h prior to the initial evaluation.

Each sample was positioned under a Full-Automatic Microhardness Testing System (Kawasaki, Kanagawa, Japan), which determined the microhardness (Knoop hardness, HK) using 50 gF of applied load over 10 s.

The measurement was performed at three points, two on the top surface, which is closest to the tip of the photopolymerization unit, and one at the base of each sample. The measurements were carefully recorded for each group before and after the aging process to calculate the mean microhardness values.

The aging process was carried out by storage in a 75 vol% aqueous solution of ethanol (replaced every 3 d) in hermetically-sealed plastic containers over 30 d in an oven at 37 °C (Miletic, et al., 2019). Again, microhardness measurements were performed at the same three points of each sample.

Following the microhardness and aging tests, the 135 samples were randomly arranged on black paperboard prior to evaluation. More specifically, a cardboard box measuring 22 cm in width, 26 cm in height, and 37 cm in length, painted with matte black paint, was manufactured to avoid reflections and action from other sources of light. For the same reason, this experiment was performed in a darkroom with the samples illuminated only by ultraviolet radiation lamps. A BLB-9W/G23 (90-230V/60Hz) fluorescent lamp (Masterkey, Tokyo, Japan) measuring 12.3 cm in length was arranged perpendicularly and positioned 16 cm from the SPs to ensure that the ultraviolet light was emitted perpendicularly.

Three blinded, calibrated, and instructed evaluators assessed the degree of fluorescence of the samples when under illumination by ultraviolet light, and the samples were classified according to their intensity (high, medium, or low), with reference to two central incisors.

The data obtained from the microhardness tests were evaluated using the Shapiro-Wilk test, while the homogeneity of the variances was evaluated using the Cochran test. Once the data were in agreement with the appropriate assumptions, the factorial repeated measures ANOVA test was applied, followed by the LSD-Fisher follow-up test.

The 135 samples that had been subjected to fluorescence analysis were then classified by three evaluators according to their intensity (i.e., high = 3, medium = 2, and low = 1) at each depth (2, 3, and 4 mm). The mean values of the intensities were then obtained. For fluorescence analysis, the double-factor ANOVA test was applied using the statistical program STATISTICA 7®.

It was found that the Kappa values required to specify the concordance between the fluorescence classifications among the evaluators was considered good.

3. Results and Discussion

Statistical analysis of the microhardness results obtained herein was performed initially by comparing the five commercial brands and the different thicknesses tested, and by fixing the analysis surface (top or base) and aging time (24 h or 30 d), as described in Tables 1–2 and as shown in Figures 2–3. In this analysis, the fixed variable was the sample thickness (i.e., 2, 3, or 4 mm). Analysis of the differences between the fluorescence values of the various samples is outlined in Table 3.

Table 1. Mean values and standard deviations of the mean values obtained from the surface microhardness (KHN) tests of the tops of the samples after 24 h and 30 days.

Composite	**Thickness (mm)**					
24H	**2**		**3**		**4**	
Z350	60.40	(±11.61) Aa	63.61	(±13.91) Aa	61.10	(±6.96) Aa
AURA	35.57	(±6.18) BCa	31.77	(±3.04) Ba	37.25	(±6.19)BCa
TETRIC	35.64	(±12.27)BCa	34.90	(±4.14)BCa	37.41	(±6.94)BCa
SDR	25.04	(±2.54) Ba	24.44	(±1.57) Ba	24.03	(±0.85) Ba
FBF	47.12	(±6.39) ACa	48.53	(±4.07)ACa	50.16	(±8.41) Ca
30D						
Z350	48.96	(±8.64) Aa	60.45	(±8.93) Aa	60.50	(±5.71) Aa
AURA	39.26	(±7.79) BCa	44.22	(±3.08) Ba	42.95	(±4.94) BCa
TETRIC	47.48	(±3.58) BCa	48.15	(±13.77) BCa	39.75	(±6.67) BCa
SDR	29.57	(±5.25) Ba	30.36	(±3.20) Ba	26.94	(±4.15) Ba
FBF	48.29	(±4.43) ACa	51.45	(±4.85) ACa	53.64	(±3.91) Ca

* Different letters indicate statistically significant differences (uppercase letters: columns, lowercase letters: rows), $p<0.05$. Source: Authors.

Table 2. Mean values and standard deviations of the mean values obtained from the surface microhardness (KHN) tests of the tops of the samples after 24 h and 30 days.

Composite	**Thickness (mm)**					
24h	**2**		**3**		**4**	
Z350	55.70	(±8.21) Aa	55.13	(±11.72) Aa	45.54	(±9.77) Aa
AURA	31.41	(±5.52) BCa	27.56	(± 2.30) ACa	26.54	(±2.62) ACa
TETRIC	25.62	(±3.12) BCa	23.47	(± 0.23) BCb	24.16	(±1.36) BCab
SDR	23.40	(±0.11) Ba	23.34	(± 0.04) BCa	23.32	(±0.04) BCa
FBF	38.04	(±4.77) ACa	39.57	(± 4.58) Aa	36.50	(±5.59) Aa
30D						
Z350	44.56	(±6.30) Aa	50.13	(±11.56) Aa	48.21	(±8.35) Aa
AURA	35.13	(±5.86) ACa	34.54	(±5.88) ACa	34.76	(±5.69) ACa
TETRIC	38.71	(±3.83) Aa	31.86	(±5.03) BCab	25.30	(±1.84) BCb
SDR	25.90	(±3.02) BCa	24.69	(±2.28) BCa	23.81	(±0.74) Ba
FBF	44.15	(±5.23) Aa	40.46	(±4.14) ABa	39.65	(±3.85) Aa

* Different letters indicate statistically significant differences (uppercase letters: columns, lowercase letters: rows), p<0.05. Source: Authors.

Table 3. Median and interquartile deviations of the scores obtained for analysis of the sample fluorescence after 30 d.

Composite	**Thickness**		
	2	**3**	**4**
Z350	3.00 (1.00) Aa	3.00 (0.00) Aa	3.00 (0.00) Aa
FBF	3.00 (1.00) ACa	2.00 (0.00) ACa	3.00 (1.00) Aa
AURA	2.00 (1.00) BCa	1.00 (1.00) BCa	1.00 (0.00) Ba
TETRIC	1.00 (0.00) Ba	1.00 (0.00) Ba	1.00 (0.00) Ba
SDR	2.00 (0.00) ACa	3.00 (2.00) ABa	3.00 (0.00) Aa

* Different letters indicate statistically significant differences (uppercase letters: columns, lowercase letters: rows), p<0.05. Source: Authors.

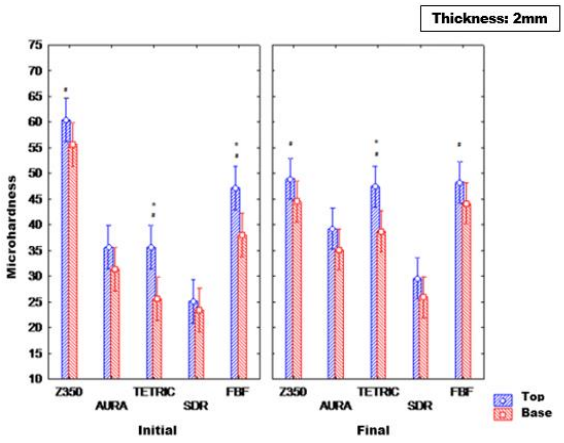
In both the 24 h and 30 d tests, the Z350 composite presented statistically similar values for thicknesses of 2 mm and 3 mm on its top surface upon comparison with the FBF composite. In contrast, for a thickness of 4 mm, statistically different and superior values were found compared to the other composites. In the analysis of the base surface of the Z350 composite, it was found that for the three thicknesses evaluated, statistically similar values were obtained to the FBF composite for both the 24 h and 30 d tests. In addition, on its base surface, the Z350 composite presented values statically inferior to the AURA composite in all evaluated thicknesses and for both evaluation times, with the exception of a 2 mm thickness over 24 h, in which the Z350 composite presented higher values. Comparison between the top surfaces of the Z350 composite and the TETRIC and SDR composites showed that the nanoparticulate composite presented higher values irrespective of the aging time. Upon analysis of the base surface, the SDR composite showed values statistically lower than the Z350 composite in all cases, while the TETRIC composite gave values lower than the Z350 composite, with the exception of a 2 mm thickness, in which the data were similar to the Z350 composite under the same conditions (Tables 1, 2).

Comparison between the bulk-fill composites showed statistically significant differences between the FBF and SDR composites in all analyses, with higher values being recorded for the FBF composite. Comparison between the AURA,

TETRIC, and Z350 composites showed similar values for each combination upon considering the variations in thickness and aging times of the samples (Tables 1, 2).

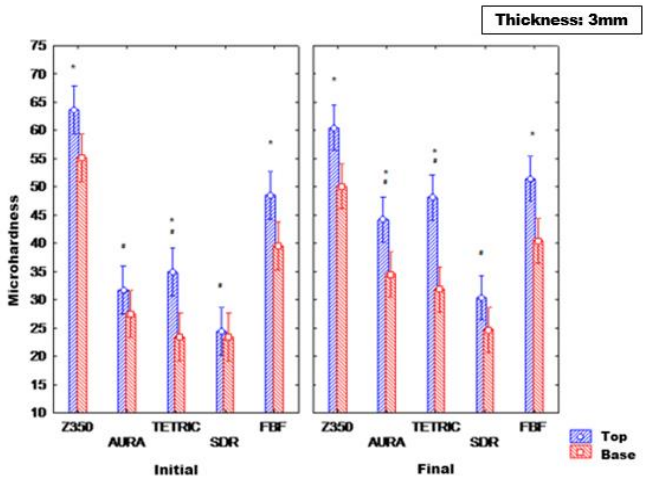
Analysis of Figures 1, 2, and 3 shows the results of statistical differences between the top and base surfaces and between the initial (24 h) and final (30 d) aging periods, where all test samples had a constant thickness.

Figure 1. Mean values and confidence intervals (95%) of the microhardness values obtained for the test samples with a thickness of 2 mm. The asterisk (*) indicates the difference between the top and base surfaces of the samples, and the hash sign (#) indicates the difference between the initial and final aging periods.



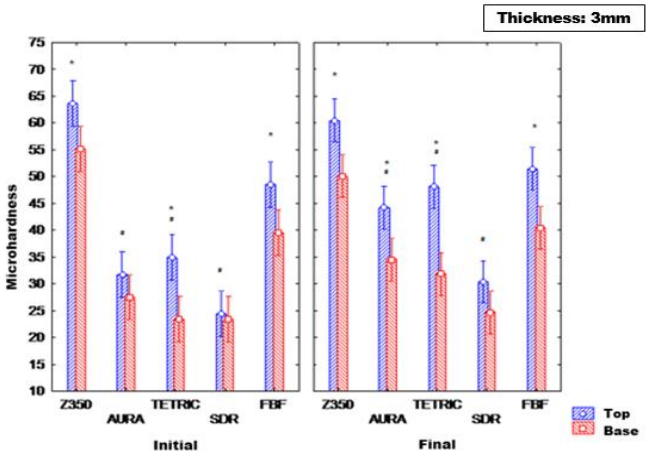
Source: Authors.

Figure 2. Mean values and confidence intervals (95%) of the microhardness values obtained for the test samples with a thickness of 3 mm. The asterisk (*) indicates the difference between the top and base surfaces of the samples, and the hash sign (#) indicates the difference between the initial and final aging periods.



Source: Authors.

Figure 3. Mean values and confidence intervals (95%) of the microhardness values obtained for the test samples with a thickness of 4 mm. The asterisk (*) indicates the difference between the top and base surfaces of the samples, and the hash sign (#) indicates the difference between the initial and final aging periods.



Source: Authors.

In these evaluations, differences between the top and base surfaces at a thickness of 2 mm were found in the TETRIC composite at both times and in the FBF composite only at the final aging time. It was also possible to verify differences in the surface microhardness between the initial and final aging times for the Z350, TETRIC, and FBF composites (Figure 1).

Evaluation of the 3 mm-thick samples confirmed differences between the top and base surfaces for the TETRIC and FBF composites for both aging times, while for the Z350 composite, differences were only observed at the initial aging time, and for the AURA composite, only at the final aging time. Differences between the initial and final again times were observed for the AURA, TETRIC, and SDR samples (Figure 3).

As shown in Figure 3, with a sample thickness of 4 mm, the only difference was observed for the AURA composite when comparing the initial and final aging times. Analysis of the differences between the top and base surfaces showed differences for the AURA and FBF composites at the two evaluation times, whereas the TETRIC composite presented differences between the two surfaces only at the initial time. In the case of the Z350 and SDR composites, differences were found between the top and base surfaces only after 30 d of aging. As shown in Table 5, the SP thickness did not influence the fluorescence results for the samples after 30 d aging. More specifically, the Z350, FBF, and SDR composites showed statistically similar behavior among themselves. When the comparison was made only between bulk-fill composites, it was found that the TETRIC and AURA composites exhibited similar fluorescence properties at all evaluated thicknesses, while the other composites tended to display lower fluorescence properties. Overall, the highest fluorescence values were found for the Z350, FBF, and SDR composites in the majority of comparisons.

In the present study, all materials (bulk-fill and conventional composites, 4 mm thickness) presented significant differences in microhardness between the top and the base surfaces, with the lowest values being observed for the base surfaces. Similar results were reported by Garcia et al., where both the flow composite (control) and the bulk-fill composite exhibited lower microhardness values on the base surface. According to the findings of Kim et al. (2015) a decrease in microhardness was found upon increasing the material thickness. This agrees with our results, and it was apparent that thicker samples exhibited greater differences in microhardness between the top and base surfaces, likely due to the lower degree of conversion of the resinous monomers at greater depths. It was also reported that the microhardness values for the TETRIC bulk-fill composite were significantly lower for the 4 mm-thick sample than for the 2 mm-thick sample (Miletic et al., 2019).

This pattern is comparable to that of conventional composites with an increased thickness. Conversely, Flury et al. (2012) reported that upon increasing the thickness, the microhardness of the conventional composite decreased, but generally remained constant in the case of bulk-fill composites. In this study, this reduction in microhardness for the 4 mm-thick samples can be explained by the fact that the light intensity emitted by the photopolymerization reaction reduces with increasing depth (Benetti et al, 2015; Flury et al., 2012).

It has also been reported that the effectiveness of the polymerization reaction is related to the composition of the material[11]. Instead of the hardness measure being an indicator of the extent of polymerization, factors such as charge content, monomer type, diluent concentration, mode of polymerization, and type and quantity of the initiators also play a key role in determining the microhardness (Costa et al, 2017; Miletic et al., 2019). In our case, the TETRIC and FBF composites exhibited microhardness differences between the top and base surfaces at all thicknesses examined (i.e., 2, 3, and 4 mm). This was not so apparent for the AURA and SDR composites. In addition, Garoush et al. (2016) observed a drastic reduction in microhardness for the base of the TETRIC bulk-fill composite. Furthermore, when performing in vitro testing with extracted teeth and evaluating the microhardness values at different restoration depths (i.e., at 1, 2, 3, and 4 mm), Fronza et al. (2015) found no statistically significant differences between the SDR, FBF, and the Herculite composite at all depths examined, although lower values were observed for the TETRIC composite at increasing depths. This can be explained by the presence of prepolymer particles in this material, which result in lower microhardness values. Moreover, the lower microhardness values of the SDR bulk-fill composite can be attributed to the lower quantities of inorganic filler, thereby corroborating the findings of Kim et al. (2015) and Al Shaafi et al. (2016).

Due to the characteristics of the oral environment, which is susceptible to solubility and biodegradation issues, studies such as the current one seeks to mimic the aging process by immersion in ethanol/water solutions (Borges et al., 2019; Kim et al., 2015). Thus, the significant decreases in microhardness at the top and base surfaces of the Z350 composite after aging can be explained by the time-dependent degradation of the composite attributed to ester group hydrolysis in the composite matrix, and to the hydrolysis of silane between the inorganic filler and the organic matrix (El Gezawi et al., 2012; Rosa et al, 2015). In addition, the decrease in the hardness of some composites when in contact with ethanol can be explained by solvent interactions with the polymer. This action of the solvent on the polymer chain is known as a plasticization effect, where separation of the polymer chains occurs through molecules that do not form primary bonds with one another. The polymer may not be dissolved but is swollen when in contact with the solvent, and so the attraction forces between the polymer chains are weaker than those between the molecules (Lima et al., 2018; Mansourin & Zidan, 2018).

An interesting result was found for the TETRIC, AURA, and SDR composites, which exhibited significantly increased microhardness values after aging in the ethanol/water solution. This finding may be justified by the presence of secondary bonds between the polymers, and the inability of the solvent (in the case of ethanol) to break down the highly polymerized cross-linked, non-soluble covalent bonds, despite the effect of swelling.

In terms of the fluorescence, the Z350, FBF, and SDR composites showed statistically similar behaviors. When comparing bulk-fill composites only, TETRIC and AURA showed similar fluorescence values at all evaluated thicknesses, while upon comparison with the other composites, lower fluorescence values were generally observed. In addition, the highest fluorescence values were found for the Z350, FBF, and SDR composites in the majority of comparisons.

These findings can be attributed to the fact that the basic components of some restorative materials do not exhibit fluorescence; however, manufacturers often add rare-earth metal phosphors such as europium, terbium, ytterbium, and cerium, which satisfactorily reproduce the fluorescence of natural teeth. According to the manufacturers of the materials examined herein, TETRIC contains ytterbium trifluoride, Surefil (SDR) contains iron oxide, and FBF contains ytterbium fluoride,

thereby accounting for the higher fluorescence values observed for FBF and SDR. Nevertheless, a higher value would have been expected for the TETRIC composite.

Upon aging of the composites, adsorption or absorption may occur on the surface, which may interfere with the fluorescence properties of the materials. In the former case, the material absorbs part of the ultraviolet light that falls on it, reducing the fluorescence. In the latter case, absorption of part of the fluorescence radiation in the blue spectrum occurs. It can be argued that the first phenomenon has a greater influence than the second, since alcoholic solutions exhibit greater absorptions at higher energies with a shorter wavelength[15]. Indeed, this was confirmed by our results, as the TETRIC and AURA composites showed lower fluorescence values after 30 d of aging in the ethanolic solution.

4. Conclusion

We evaluated a number of bulk-fill composite materials with respect to the microhardness values of the top and base surfaces upon variation in the composite thickness. In addition, the effect of aging and comparison of the fluorescence properties of the materials at different thicknesses was examined. We found that aging, sample thickness, and the type of material affected both the surface microhardness and the fluorescence values. In addition, the AURA and SDR bulk-fill composites were the least susceptible to differences in thickness between the top and base surfaces in terms of the microhardness values. The literature is inconsistent regarding the determination of DOC and therefore, some results are contradictory, like this the results of this study suggest that clinical research with these materials is required before clinical application.

In order to obtain a literary consistency that confirms the findings of this research, it is necessary that more studies are carried out on the subject, encompassing aspects of longer aging periods and using different tests to evaluate the bond strength of the material.

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