

OPTICAL DENSITY AND CHEMICAL COMPOSITION OF MICROFILLED AND MICROHYBRID COMPOSITE RESINS

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ABSTRACT

This study evaluated the optical density of two microfilled and two microhybrid resins, as well as the composition of these materials with regard to their optical density. Cavities prepared in 12 2-mm- or 4-mm-thick acrylic plastic plates were filled with Z250 (3M-ESPE), A110 (3M-ESPE), Charisma (Heraeus-Kulzer) and DurafillVS (Heraeus-Kulzer). The resin increments (2-mm-thick) were light-cured for 40 s. Three 0.12-s radiographic exposures were made of each #2 acrylic plastic plate. DenOptix system optical plates were used to obtain the digital images. Three readings of the composite resin surface were made in each radiograph, totalizing 216 readings. The mean of highest and lowest grey-scale values was obtained. Two specimens of each composite resin were prepared for SEM analysis of the chemical elements related to optical density, using energy dispersive x-ray analysis (EDX). The results were subjected to Shapiro-Wilk's test, ANOVA, Tukey's test at 1% level of significance and Pearson's correlation. The mean grey-scale values at 2 mm and 4 mm were: Z250 = 154.27a and 185.33w; A110 = 46.77b and 63.05y; Charisma = 163.40c and 200.46z; DurafillVS = 43.92b and 58.99x, respectively. Pearson's test did not show any positive correlation between optical density and percentage weight of optical density chemical elements. It was concluded that the microhybrid resins had higher optical density means than the microfilled resins; among the evaluated resins, Charisma had the highest optical density means.

Key words: Composite resins. Dental materials. Radiology.

INTRODUCTION

In 1981, the American Dental Association (ADA) Council on Dental Materials and Devices, in accordance with the #27 specification issued in 1977, suggested the inclusion a statement that optical density is a desirable requirement in restorative materials^{6,7}. This would make it possible to determine the difference between the restorative material and a primary or secondary caries as well as to identify material excesses, presence of bubbles and other defects in radiographic images^{6,7,11}.

Several dental studies have been conducted to evaluate the optical density of restorative materials^{1,8,10,14,15}. With the advent of digital technology, it became possible to measure the optical density of different materials in pixels.

Because of the importance of material's optical density and the development of digital images, it has become important to determine whether composite resin composition and filler particle size could influence their differentiation from other

materials and dental structures, making it possible to identify flaws in restorations. In view of this, the purpose of this study was to verify the optical density of two microfilled and two microhybrid composite resins, as well as the composition of these materials regarding the presence of chemical elements responsible for optical density, at 2 and 4mm thickness. The null hypothesis was that there is no difference of optical density between the microfilled and microhybrid composite resins under the tested conditions.

MATERIALS AND METHODS

Four composite resins were used: two microfilled and two microhybrid, all of shade A3, according to Table 1.

Twelve transparent acrylic plates measuring approximately 4.4 cm x 3.2 cm were obtained with an area corresponding to the area of #2 periapical dental film. A total of six 2-mm-thick plates and six 4-mm-thick plates were used; their thickness

TABLE 1- Composite resins: particle size, manufacturer, lot and composition

Composite resins	Classification	Manufacturing	Lot	Composition
Z250	Microhybrid	3M-ESPE Dental Products, St. Paul, MN	5BR	Inorganic filler (Zirconium/silica) loading is 60% by volume with a particle size range of 0.01 to 3.5 microns. BIS-GMA, UDMA and BIS-EMA. Encore-GMA, UDMA, Encore-EMU, Zirconium/Silicon 60% (0.01 to 3.5 micrometers)
Charisma	Microhybrid	Heraeus Kulzer Inc., Irvine, CA	010081	Based on a BIS-GMA matrix and contains 64% filler (by volume), which is: <ul style="list-style-type: none"> • Barium aluminum fluoride glass (0.02 - 2 microns) • Highly dispersive Siliciumdioxide (0.02 - 0.07 microns) silicon dioxide (0.01–0.07 $\mu\text{m}/\text{Ø}$ 0.04 μm).
A110	Microfilled	3M-ESPE Dental Products, St. Paul, MN	2AX	Organic filler, inorganic (silica) filler loading is 56% by weight or 40% by volume. Encore-GMA, TEGDMA, aluminum oxide and methacryloxi-propil-methoxy-silane
DurafillVS	Microfilled	Heraeus Kulzer Inc., Irvine, CA	010142	Produced on basis of urethanedimethacrylate. Highly disperse silicon dioxide (0.02 - 0.07 μm). Splinter polymer (10 - 20 μm). Solids content: 75.3%.

was checked with digital caliper. Each plate was bisected and a cavity with 4 mm in diameter and depth corresponding to the plate thickness was prepared in the center of each half and filled with the composite resins. Three plates of each thickness (2 mm and 4 mm) were used for each type of composite resin (microfilled and microhybrid).

To identify the plates, they were marked at the top left edge: lines were used to number the plate (1, 2 or 3) and cavities were used to mark the thickness (1 cavity corresponded to 2-mm-thick plates and 2 cavities to 4-mm-thick plates). A spherical mark was made at the top right corner of the plates corresponding to the microhybrid composite resins, while the plates with microfilled resins were not marked in this position. These demarcations were filled with radiopaque composite resin to enable them to be visualized in the radiographic exposure (Figure 1).

The composites were inserted into the cavities in alphabetical order. To obtain flat resin specimen surfaces in the acrylic plates, a microscope glass slide was attached with a polyester strip where the resin should be inserted. A polyester matrix was placed on the unpolymerized composite resin, and manual pressure was applied with the glass slide. In the 2-mm-thick plates, the composite resins were inserted in a single increment, while and in the 4mm plates in two increments, and as soon as the glass slide was removed, each

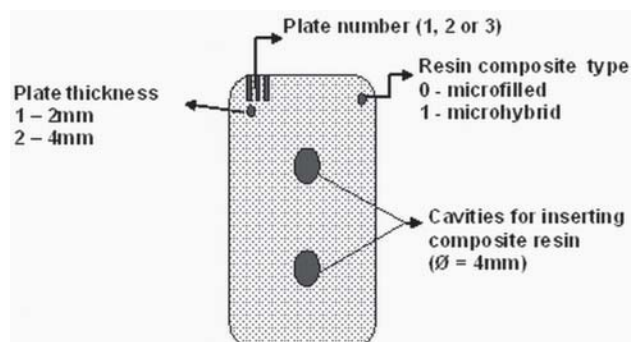


FIGURE 1- Acrylic plate with the cavities for inserting the composed resins and showing plate demarcations

increment was light-cured for 40 seconds. The light intensity of the light-curing unit (XL3000; 3M/ESPE, St. Paul, MN, USA) was checked with a curing radiometer (Demetron; Kerr Corporation, Orange, CA, USA) and remained between 450 and 600 mW/cm^2 .

The radiographic exposure of each plate was analyzed by the DenOptix indirect digital image system (Dentsply International/Gendex Dental X-Ray Division, Des Plaines, IL, USA) in order to evaluate different grey levels for each thickness and composite. For such purpose, #2 optical plates

wrapped with the specific protector were used.

The digital images were obtained using x-ray unit Gnatus Timex-70DRS (Gnatus, Ribeirão Preto, SP, Brazil), which operates at 80 kVp, 8 mA electrical system, with an exposure time of 0.12 s, focal distance of 40 cm, and central X-ray beam incident at an angle of 90° to the center of the plate. Three radiographic exposures of each plate were made (three plates for each resin, n=9), totalizing 36 exposures. Image digitization was done with VixWin and a DenOptix Laser Scanner imaging software (Dentsply International/Gendex Dental X-Ray Division).

Three radiographic image readings were taken of the same radiographic exposure for each composite resin in the top, middle and bottom thirds of the specimen, avoiding the interference of flaws and bubbles. For the optical reading in each third, two points were selected, forming a line, where the maximum and minimum grey levels values were logged. These values were recorded on a spreadsheet and the mean value for each composite resin specimen was made. Thirty-six radiographic exposures were made (12 plates, with 2 composite resins each, 3 exposures of each plate) in total 216 optical readings (Figure 2).

To analyze the chemical composition of the composite resins, other two 2-mm-thick specimens of each material were

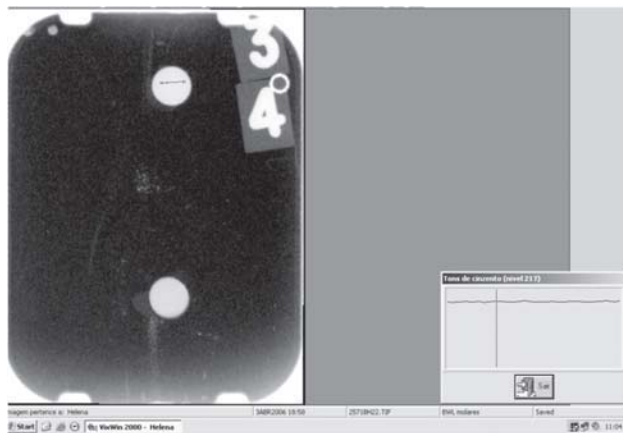


FIGURE 2 - Optical density means in the VixWin program of the DenOptix system

made. Next, the specimens were subjected to gold-ion deposition and examined by scanning electronic microscopy (SEM). Three areas of each resin specimen were selected for taking the reading by energy dispersive X-ray analysis (EDX).

The optical density results were submitted to Shapiro-Wilk's test, ANOVA and Tukey's test at 1% level of significance. These values and the percentage by weight of the chemical elements of optical density were correlated by Pearson's correlation.

RESULTS

The microhybrid resins (Z250 and Charisma) presented higher mean optical density than the microfilled resins (A110 and DurafillVS) for both evaluated thicknesses (2 mm and 4 mm).

In the 2-mm-thick specimens, DurafillVS and A110 presented statistically similar optical density means to each other, while the resins Z250 and Charisma differed significantly (Table 2). In the 4-mm-thick specimens, the evaluated resins presented different optical density means to each other (Table 2).

Table 3 shows the percentage by weight (mean value) of the elements of optical density for each resin. Table 4 shows

TABLE 2- Comparison among the optical density of the resins at the thicknesses of 2 mm and 4 mm

Resins (n=9)	2mm	S.D.	4mm	S.D.
DurafillVS	43.92 ^a	2.94	58.99 ^x	1.63
A110	46.77 ^a	2.18	63.05 ^y	1.94
Z250	154.27 ^b	1.78	185.33 ^w	1.34
Charisma	163.40 ^c	2.15	200.46 ^z	1.72

Different letters indicate statistically significant difference at 1%.

TABLE 3- Means and sum (% w) of elements of resin optical density

Resins	Means of elements of resin optical density (%w)			Sum (%w)
	Al	Zr	Ba	
Durafill VS	0a ± 0b (0-0) ^{c*}	0 ± 0 (0-0)	0 ± 0 (0-0)	0 ± 0 (0-0)
A110	0.51 ± 0.13 (0.35-0.78)	0 ± 0 (0-0)	0 ± 0 (0-0)	0.51 ± 0.13 (0.35-0.78)
Z250	0 ± 0 (0-0)	19.75 ± 2.53 (16.39-23.48)	0 ± 0 (0-0)	19.75 ± 2.53 (16.39-23.48)
Charisma	3.94 ± 0.56 (2.9-4.8)	0 ± 0 (0-0)	18.76 ± 2.22 (15.34-21.98)	22.7 ± 2.35 (19.12-24.88)

* a=mean values, b= standard deviation, c= minimum and maximal values.

TABLE 4- Results of the correlation between the optical density means and the percentage by weight of the chemical elements for each resin

Resins	r*	p**
A110	0.0493	0.9262
Charisma	- 0.1301	0.8059
Z250	0.0507	0.9240
Durafill VS	0	0

* r = correlation's index . ** p = significance level.

the results of the correlation between the optical density means and the percentage by weight of the chemical elements for each resin. The mean value of the six readings for each chemical element was obtained and the sum of these means was considered the total value of chemical elements for each resin.

Optical density results and the sum of the percentage by weight of the chemical elements were assessed by Pearson's correlation (Table 4). The results of the EDX analysis, the microfilled resin DurafillVS showed no chemical elements of optical density. Pearson's correlation showed that for the tested resins, there was no correlation between the optical density means and the percentage by weight of the evaluated chemical elements.

DISCUSSION

This null hypothesis was rejected, since there was significant difference between the tested materials. The microhybrid resins (Z250 and Charisma) presented higher mean optical density than the microfilled resins (A110 and DurafillVS) with both measured thicknesses. These results suggest that the tested microhybrid resins could be more easily distinguished than the microfilled resins, when evaluated by the DenOptix system. The clinical relevance of this study relies on the benefits of accurately distinguishing radiographic images of restorative materials from dental structures, caries lesions and structural defects^{13,17}.

When evaluated at 2-mm thickness, the microfilled composites did not differ to each other. The microhybrid composites, on the other hand, presented different optical density to each other, Charisma obtained the highest mean value (163.40). The microhybrid composites presented significantly higher means when compared to the microfilled composites ($p=0.01$) (Table 2). Turgut, et al.²⁰ (2003) and Attar, et al.⁴ (2003) evaluated the optical density of composites, among which Z250 and A110, at thickness of 1 mm, and found higher optical density results for Z250 and lower values for A110. Likewise, in the present study, Z250 presented high optical density means (154.27) while A110 presented the lowest values (46.77). In other studies^{11,16} Charisma, DurafillVS and Z250 were compared to enamel

and dentin, at thickness of 2 mm, using the Digora digital system. Those authors demonstrated that Charisma and Z250, both microhybrid composites, provided higher optical density than the enamel, while DurafillVS microfilled resin showed lower optical density values than the dentin. Table 2 shows that Z250 and Charisma presented high optical density means, while DurafillVS presented the lowest optical density mean. All these studies^{4,11,16,20} seem to indicate a trend of microfilled composites to be radiolucent. However, a comparison of enamel and dentin optical densities of this study to those of previous investigations is not possible because there are differences in specimen thickness and methods for indirect digital image analyzing.

When the 4-mm-thick plates were evaluated, all tested resins presented significantly different grey level values to each other. Resin's optical densities in an increasing order were: DurafillVS, A110, Z250 and Charisma (Table 2). Apart from the fact that microhybrid resins present excellent mechanical properties, it is possible to suggest, based on these results of optical density, that it would be also suitable to use these resins in wide cavities.

Some kinds of particles are added to the composition of restorative materials, specifically to confer optical density, to allow them to be observed radiographically and distinguished from other structures and materials. In order to provide this property, the added chemical elements should present a high atomic number³. Barium, strontium, zirconium, zinc, yttrium, ytterbium, lanthanum, aluminum and potassium are some of the elements described in the literature as having this characteristic, and are used by manufacturers to confer optical density to restorative materials^{2,3,5,9,19}. In the resin specimens made for this study, the following elements to confer optical density were found: aluminum, zirconium and barium. In the studies of Sabbagh, et al.¹⁶ (2004) and Toyooka, et al.¹⁹ (1993), the authors found a linear correlation between load percentage and the optical density of the tested materials. Elements of low atomic number, such as silicon, result in radiolucent materials, while added materials with elements of high atomic number (Ba, Y, Yb, Zr, Sr) become more radiopaque^{16,18,20}.

EDX is a powerful instrument for making a qualitative and quantitative analysis of the chemical elements present in the materials' composition^{3,19}. In the DurafillVS specimen, no chemical element of optical density was found (Table 3), in the same way as described by Sabbagh, et al.¹⁶ (2004). Only 0.51% of aluminum was found in A110. Perhaps, this tiny percentage of aluminum was added to the chemical composition of this material for other reason than optical density, because in such small amount it could not achieve this objective. To obtain optical density, an amount approximately 20% of radiopaque elements is required²¹.

The manufacturer of A110 and Z250 (3M-ESPE) informs that these materials present zirconium/silicon particles and are radiopaque. Toyooka, et al.¹⁹ (1993) affirmed that zirconium would give higher optical density to the materials than barium. In the EDX analysis of Z250, a mean percentage of 19.75% of zirconium was found, and it is possible that this amount of zirconium could be responsible for the

significantly higher optical density means of this resin.

The chemical composition of Charisma has 3.78% of aluminum and 18.76% of barium, as reported elsewhere^{16,19}. Accordingly, in the present study, this resin showed the highest optical density values, differing from the other tested resins; its composition presents exactly these elements in a total percentage weight of 22.7%.

Regarding the correlation results, it can be stated that no association exists between the optical density values and the composition of the microfilled and microhybrid resins tested.

CONCLUSIONS

Based on the results obtained, it may be concluded that

1. The microhybrid resins obtained higher optical density values than the microfilled resins; among all the resins evaluated, Charisma obtained the highest optical density means;
2. There was no correlation between the optical density means of the tested resins and the percentage by weight of chemical elements of optical density.

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