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Fluorescence Intensity of Resin Composites and Dental Tissues Before and After Accelerated Aging: A Comparative Study

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Clinical Relevance

Fluorescence is one of the optical properties of resin composites and dental tissues that is of great interest today. The fluorescence intensity of restorative materials should be as close as possible to that of human enamel and dentin to ensure an acceptable reproduction of these qualities in esthetic restorations.

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SUMMARY

This study quantitatively evaluated the fluorescence intensity of resin composites with different opacities and translucencies and determined changes in fluorescence after accelerated aging, using human enamel and dentin as controls. Six microhybrid and nanofilled composites, each in three different shades, were tested. Ten sound human incisors were used to obtain enamel and dentin specimens separately. Fluorescence measurements were obtained with a fluorescence spectrophotometer before (baseline) and after accelerated aging at 150 kJ energy for 120 hours. One-way analysis of variance (ANOVA) and Games-Howell multiple comparison tests were performed at a significance level of 0.05. Student's *t*-test was also used for comparison before and after aging. At baseline, there was no statistically significant difference (p>0.05) between the fluorescence intensity of dentin and any of the shades of Charisma or Opallis, Esthet-

X dentin shade or Vit-l-escence enamel, or the translucent shades. After accelerated aging, all shades of the 4 Seasons, enamel and the translucent shades of Esthet-X had fluorescence intensities statistically similar to that of aged dentin (*p*>0.05). A significant reduction in fluorescence after aging (*p*<0.05) was observed for all the materials, except for human enamel and translucent Filtek Supreme XT. Accelerated aging reduced fluorescence in most of the composites evaluated.

INTRODUCTION

In order to achieve predictable esthetic results and a high degree of reproducibility of the original characteristics of teeth, manufacturers are continually launching new resin composites that claim to simulate the optical properties of natural teeth.[1] Fluorescence is an important optical property of tooth structure and is defined as the physical phenomenon that occurs in less than 10^{-8} seconds, wherein the material absorbs ultraviolet light (UV) and emits visible light in the blue spectrum.[2]

Selecting a suitable shade and reproducing the visual effects of the natural layering of dental tissues is difficult,[3] because different light sources emit UV rays at different intensities, resulting in different patterns of the fluorescence of teeth and restorative materials.[4-5] Natural teeth appear whiter and brighter in daylight, because of the large amount of light in the blue spectrum produced by fluorescence.[6-8] There is also a significant UV component in certain fluorescent bulbs, photoflash lights, high intensity studio lights and entertainment lights, known as black light.[9] The latter type of light emits filtered 350-nm and 400-nm UV rays that, when absorbed by a fluorescent body, result in an emission spectrum in the intense blue region.[1]

Fluorescence in dental porcelain is achieved by incorporating small amounts of inorganic oxides of rare earth compounds with fluorescent properties.[10-11] These luminophores are usually composed of cerium, europium, terbium, ytterbium, dysprosium and samarium.[9,12] None of these oxides alone can impart fluorescence to porcelain, similar to human teeth. Instead, they must be blended in specific concentrations.[7-8] Baran and others[12] showed that the fluorescence of several mixed rare earth compounds is not equal to the sum of their individual fluorescences. Thus, it is possible for some of these elements to be used as luminophores in resin composites. However, resin composite manufacturers are reluctant to reveal the compounds responsible for the fluorescence of their products.

Fluorescence spectrophotometry has been widely used to examine the fluorescence of dental porcelains.[7,9,11-12] This method has also been used to evaluate resin composite restorations to detect differences in the fluorescence of composites and teeth[13] and to determine the effectiveness of incorporating rare earth compounds into experimental composites.[14]

Resin composites are constantly affected by physical and chemical agents that can change their optical properties. Previous studies[15-19] have investigated surface and color changes of resin composites after accelerated aging, an artificial method wherein the material is submitted to extreme conditions using UV light and changes in temperature and water.[20]

However, prior to this study, the fluorescence intensities of resin composites had not been compared with the individual fluorescence intensities of human enamel and dentin. This information is of clinical importance, since the fluorescent potential of a material is irrelevant if the material's fluorescence is not compatible with dentin, opaque materials, enamel or translucent materials.

This study quantitatively evaluated the fluorescence intensity of resin composites with different opacities and determined changes in fluorescence after accelerated aging using human enamel and dentin as controls. Two hypotheses were tested: the null hypothesis that the fluorescence properties of resin composites are similar in different materials and human enamel and dentin, and the alternative hypothesis that accelerated aging causes a significant reduction in the fluorescence intensity of composites and dental hard tissues.

METHODS AND MATERIALS

Six microhybrid and nanofilled composites, each in three different shades, were studied (Table 1). A polytetrafluoroethylene mold was used to obtain square specimens (0.5 mm x 7 mm x 7 mm). Ten specimens were made for each shade of composite, for a total of 180 specimens. Polymerization was carried out with a QTH light curing unit (Optilux 501, Kerr Corp, Orange, CA, USA) at 540 mW/cm^2 light intensity, activated for 40 seconds on the top and bottom surfaces. The specimens were then removed from the mold, kept in a light-proof container and stored for 24 hours at 37°C and 100% humidity.

Ten sound human upper central incisors were used to obtain separate enamel and dentin specimens (0.5 mm x 7 mm x 7 mm). The buccal surface of the enamel specimens was cut longitudinally next to the enamel dentin junction to separate them. A second longitudinal cut was made to obtain dentin specimens. The slices were ground and polished to a thickness of 0.5 mm and verified with a micrometer (103-125 Mitutoyo Corp, Kanagawa, Japan) to remove the remaining tissues. The specimens were stored in the same conditions as described for the composite specimens and were used as controls.

Table 1: *Resin Composites Tested in This Study*

Composite	Manufacturer	Shade	Batch #
4 Seasons	Ivoclar Vivadent Schaan, Liechtenstein	A2 Dentin A2 Enamel Trans Clear	H24150 H1981 H12935
Charisma	Heraeus Kulzer GmbH & Co Hanau, Germany	OA2 A2 I	10200 10205 10200
Esthet-X	Dentsply/Caulk Milford, DE, USA	A2-O A2 C-E	503032 50126 502021
Filtek Supreme XT	3M ESPE St Paul, MN, USA	A2 A2E YT	6CE 5BU 4BA
Opallis	FGM Dental Products Joinville, SC, Brazil	DA2 EA2 T-Neutral	310106 80206 150206
Vit-l-escence	Ultradent Products Inc South Jordan, UT, USA	A2 Pearl Amber Trans Ice	B1TM7 B1TLY B1PWV

Baseline fluorescence measurements were carried out using a fluorescence spectrophotometer (F-4500, Hitachi High-Technologies Corp, Tokyo, Japan). The specimens were fixed in a slot made in an acrylic cuvette and placed inside the spectrophotometer chamber using 2.5 nm emission and excitation aperture slits. The location of the cuvette allowed the excitation beam to reach the center of the specimen at a wavelength of 380 nm. The values obtained were then used to produce graphs with the aid of the computer software that accompanied the equipment. The fluorescence intensity values used in this study were within the visible light spectrum and ranged from 400 nm to 600 nm.

After baseline measurements, the specimens were subjected to accelerated aging according to ASTM G155, cycle 1,[21] with a Ci65 Weather-Ometer (Atlas Electric Devices Company, Chicago, IL, USA) by exposure to a xenon-arc light filtered through borosilicate glass at 0.35 W/m^2/nm at a wavelength of 340 nm. The test cycle was carried out for 102 minutes of light plus 50% humidity and 18 minutes of light plus water spray, with a black panel temperature of 63°C ± 2°C. The specimens were aged for 120 hours at a total energy of 150 kJ. The fluorescence intensity of the aged specimens was measured with the fluorescence spectrophotometer, using the same specifications as that of the baseline.

The means of the fluorescence intensity values located in the visible light spectrum between 420 nm and 470 nm were calculated. The data obtained were recorded and submitted to statistical analysis with SPSS 13.0 software (SPSS Inc, Chicago, IL, USA). One-way analysis of variance (ANOVA) and the Games-Howell multiple comparison test were performed at a significance level of 0.05. Student's *t*-test was also used to compare values before and after aging.

RESULTS

The fluorescence intensities of resin composites and dental tissues at baseline are shown in Table 2, and the intensities obtained after aging are shown in Table 3. There were no statistically significant differences between the baseline fluorescence values of composites Charisma OA2, Esthet-X A2-O, Opallis DA2 and human dentin ($p>0.05$). A similar finding was made for some enamel and translucent shades; namely, Charisma A2 and I, Opallis EA2 and TN, and Vit-l-escence Pearl Amber and Trans Ice. Prior to aging, all of the composites that were tested, except for Filtek YT, had fluorescence intensities significantly greater than that of enamel ($p<0.05$). In addition, the resin composites Esthet-X A2 and C-E, Vit-l-escence A2 (dentin) and all the 4 Seasons shades had a significantly higher fluorescent intensity than human dentin ($p<0.05$). All the Filtek materials had fluorescent intensities significantly below that of enamel and dentin.

After accelerated aging, all the shades of 4 Seasons, Esthet-X A2 and C-E had fluorescence intensities statistically similar to aged dentin ($p>0.05$). The only com-

Table 2: *Fluorescence Intensity (standard deviation) of Resin Composites, Dentin and Enamel at Baseline*

		Dentin Shade	Enamel Shade	Translucent
Resin Composites	4 Seasons	443.27 (31.29)	473.52 (22.22)	622.49 (34.19)
	Charisma	301.17 (12.02)[a]	299.60 (10.14)[a]	281.28 (17.86)[a]
	Esthet-X	296.91 (20.76)[a]	475.92 (13.00)	467.43 (18.98)
	Filtek Supreme XT	116.45 (7.75)	130.38 (11.44)	13.83 (3.84)
	Opallis	266.07 (7.70)[a]	257.97 (9.74)[a]	284.01 (11.16)[a]
	Vit-l-escence	1025.88 (21.61)	373.89 (22.90)[a]	324.08 (7.53)[a]
Control	Human Dentin	314.76 (54.82)[a]		
	Human Enamel		54.67 (14.19)	

Resin composites and control group connected by the same letter are not statistically different (p>0.05).

Table 3: *Fluorescence Intensity (standard deviation) of Resin Composites, Dentin and Enamel After Accelerated Aging*

		Dentin Shade	Enamel Shade	Translucent
Resin Composites	4 Seasons	211.20 (28.05)[a]	203.72 (21.78)[a]	206.06 (18.76)[a]
	Charisma	66.90 (4.38)	73.20 (9.86)	67.14 (11.76)[b]
	Esthet-X	104.84 (6.76)	181.58 (11.28)[a]	191.12 (11.54)[a]
	Filtek Supreme XT	73.00 (4.83)	74.67 (9.69)	16.77 (4.60)
	Opallis	130.80 (12.23)	91.64 (10.08)	139.49 (13.80)
	Vit-l-escence	582.83 (26.10)	157.49 (6.64)	142.76 (8.40)
Control	Human Dentin	201.36 (26.39)[a]		
	Human Enamel		45.33 (12.91)[b]	

Resin composites and control group connected by the same letter are not statistically different (p>0.05).

Table 4: *Absolute and Percentage Changes in Fluorescence Intensity After Accelerated Aging*

Material	Fluorescence Intensity Change (SD)	Change (%)	*p*-value
Human Dentin	-113.40 (69.46)	33.82	0.001*
Human Enamel	-9.34 (18.84)	13.04	0.152
4 Seasons A2 Dentin	-232.07 (29.77)	52.33	0.000*
4 Seasons A2 Enamel	-269.80 (24.04)	56.97	0.000*
4 Seasons Trans Clear	-416.44 (38.39)	66.81	0.000*
Charisma OA2	-234.26 (11.41)	77.77	0.000*
Charisma A2	-226.39 (11.54)	75.57	0.000*
Charisma I	-214.14 (19.22)	76.09	0.000*
Esthet-X A2-O	-192.07 (17.86)	64.61	0.000*
Esthet-X A2	-294.33 (22.67)	61.77	0.000*
Esthet-X C-E	-276.30 (23.76)	59.03	0.000*
Filtek Supreme XT A2	-43.46 (6.72)	37.19	0.000*
Filtek Supreme XT A2E	-55.71 (16.39)	42.21	0.000*
Filtek Supreme XT YT	2.94 (5.57)	29.38	0.130
Opallis DA2	-135.27 (11.86)	50.84	0.000*
Opallis EA2	-166.32 (12.67)	64.45	0.000*
Opallis T-Neutral	-144.52 (19.06)	50.78	0.000*
Vit-l-escence A2	-443.05 (34.62)	43.16	0.000*
Vit-l-escence PA	-216.40 (26.46)	57.69	0.000*
Vit-l-escence TI	-181.32 (13.80)	55.89	0.000*

**Values with statistically significant differences between baseline and aged groups (p<0.05).*

posite whose fluorescence did not differ significantly from that of aged enamel was Charisma I ($p>0.05$).

The mean values of the changes in fluorescence intensity after accelerated aging are shown in Table 4. All groups, with the exception of human enamel and Filtek Supreme XT YT, showed a significant reduction in fluorescence after aging ($p<0.05$).

Of all the composites evaluated, Charisma had the highest percentage of reduction in fluorescence after accelerated aging for all opacities; whereas, Filtek Supreme XT had the lowest. However, Filtek Supreme XT was the material with the lowest baseline fluorescence intensity values. The enamel and dentin control groups had relatively small reductions in fluorescence intensity after aging.

Figures 1 and 2 show the fluorescence intensity spectra for the composites, enamel and dentin that were evaluated when excited with a 380 nm wavelength. The spectra for all the materials tested have the shape of a wide band, with a peak in the region of 440 nm.

DISCUSSION

In human teeth, dentin exhibits a higher fluorescence intensity than enamel,[1,7,22] because dentin contains more collagen, which contains fluorescence-emitting amino acids such as trytophan.[23-24]

The fluorescent components of resin composites, however, are still unknown. Some studies[14,25-26] investigated possible ways of incorporating luminophores in composites to increase their fluorescence intensity. Uo and others[14] demonstrated that the fusion of rare earth oxides into glass filler particles increased the fluorescent properties of an experimental composite. Fluorescent emission by an experimental composite was studied by including a fluorescent whitening agent composed of an aromatic complex into the polymer matrix.[25] In another study,[26] terbium coordination polymers composed of PEMA (poly ethyl methacrylate) resulted in a hybrid polymer structure with high fluorescence.

Information on the fluorescent components of only two of the resin composites evaluated in the current study is given by the manufacturers (personal commu-

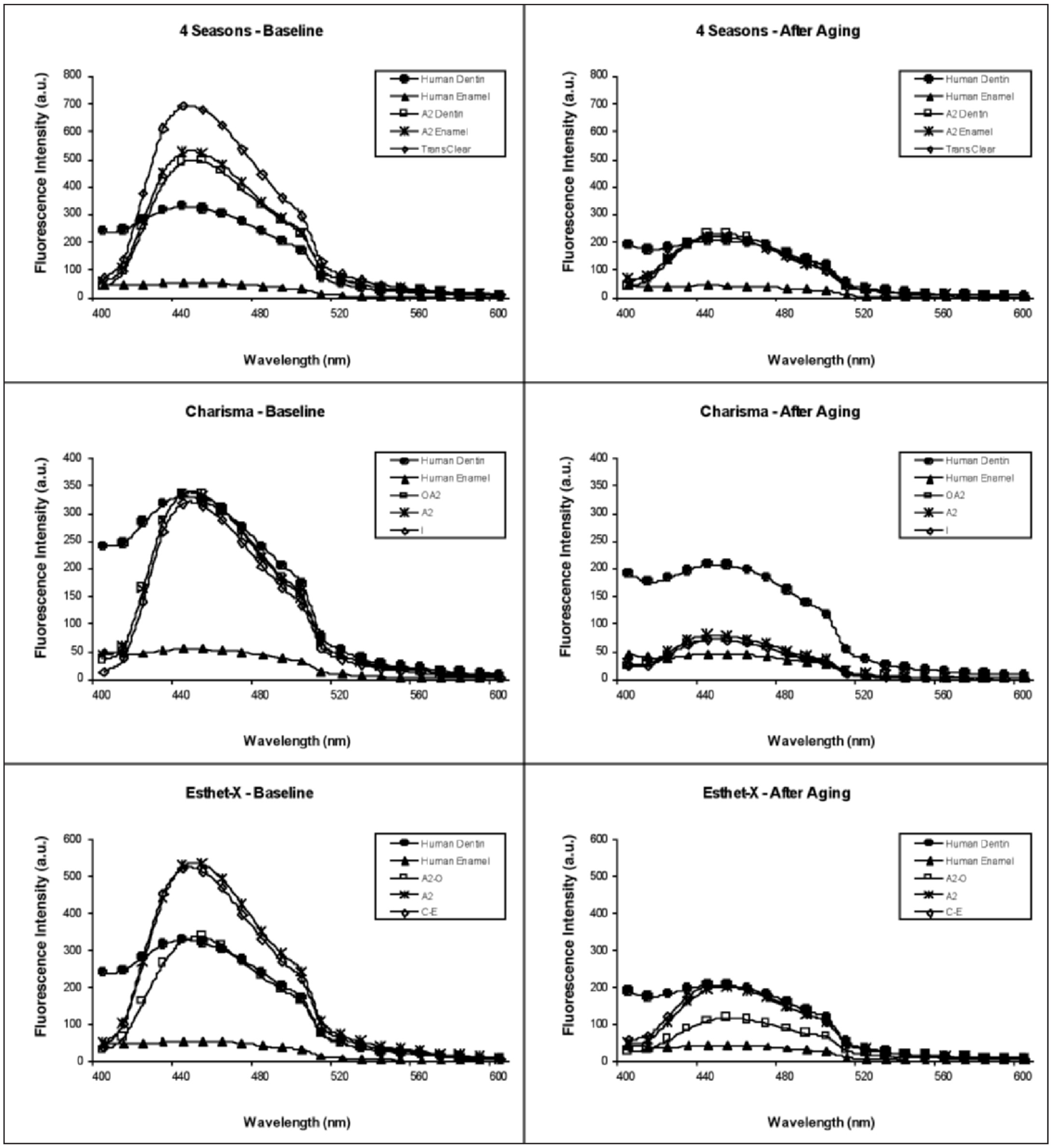

Figure 1. *Fluorescence intensity spectra of 4 Seasons, Charisma and Esthet-X, with the controls enamel and dentin, ranging from 400 nm to 600 nm.*

nication January, 2007). Ytterbium trifluoride and an organic molecule both claim to be the luminophores used in the 4 Seasons and Opallis composites, respectively.

The results of this study showed that only the dentin shades of Charisma, Esthet-X and Opallis had fluorescence intensities similar to human dentin, and none of the enamel or translucent shades had fluorescence intensities similar to human enamel. Surprisingly, the fluorescence intensities of enamel and the translucent shades of Esthet-X and 4 Seasons were much greater than dentin. The esthetic aspect of anterior tooth restorations carried out with these materials could therefore be compromised as a result of excess fluorescence, particularly when such restorations are submitted to irradiation from a light source with a high UV component.

After the accelerated aging process, all the materials evaluated showed a significant reduction in fluorescence intensity, except for translucent Filtek Supreme XT and human enamel. These findings are similar to those of Lee and others,[19] who investigated changes in the opalescence and fluorescence of several composites after accelerated aging. However, these authors used a different type of cycle in the weathering machine and the CIE L * a * b * (CIE) method[2,19,27-28] to determine fluorescence before and after aging, which may account for the reported loss of fluorescence in all of the composites tested.[19]

In the current study, a significant reduction in fluorescence intensity was found in both dentin and composites after accelerated aging under UV light at 150 kJ. This type of irradiation has a photoxidative potential and induces the cleavage of simple and double carbon bonds.[29] These chemical bonds play an important role in the configuration of the polymer backbones found in the organic matrix in resin-based materials and in organic compounds, such as collagen in human dentin. Thus, the reduction of fluorescence could be explained by the degradation of organic complexes found in dentin and resin composites. This could confirm the theory that luminophores are attached to the organic portion of dental composites. Mineral substances, such as rare earth oxides, have also been incorporated into polymer chains by chemical bonds[26] that could equally be broken by accelerated aging.

A previous study[22] reported that the fluorescence intensity of human dentin increased with age, temper-

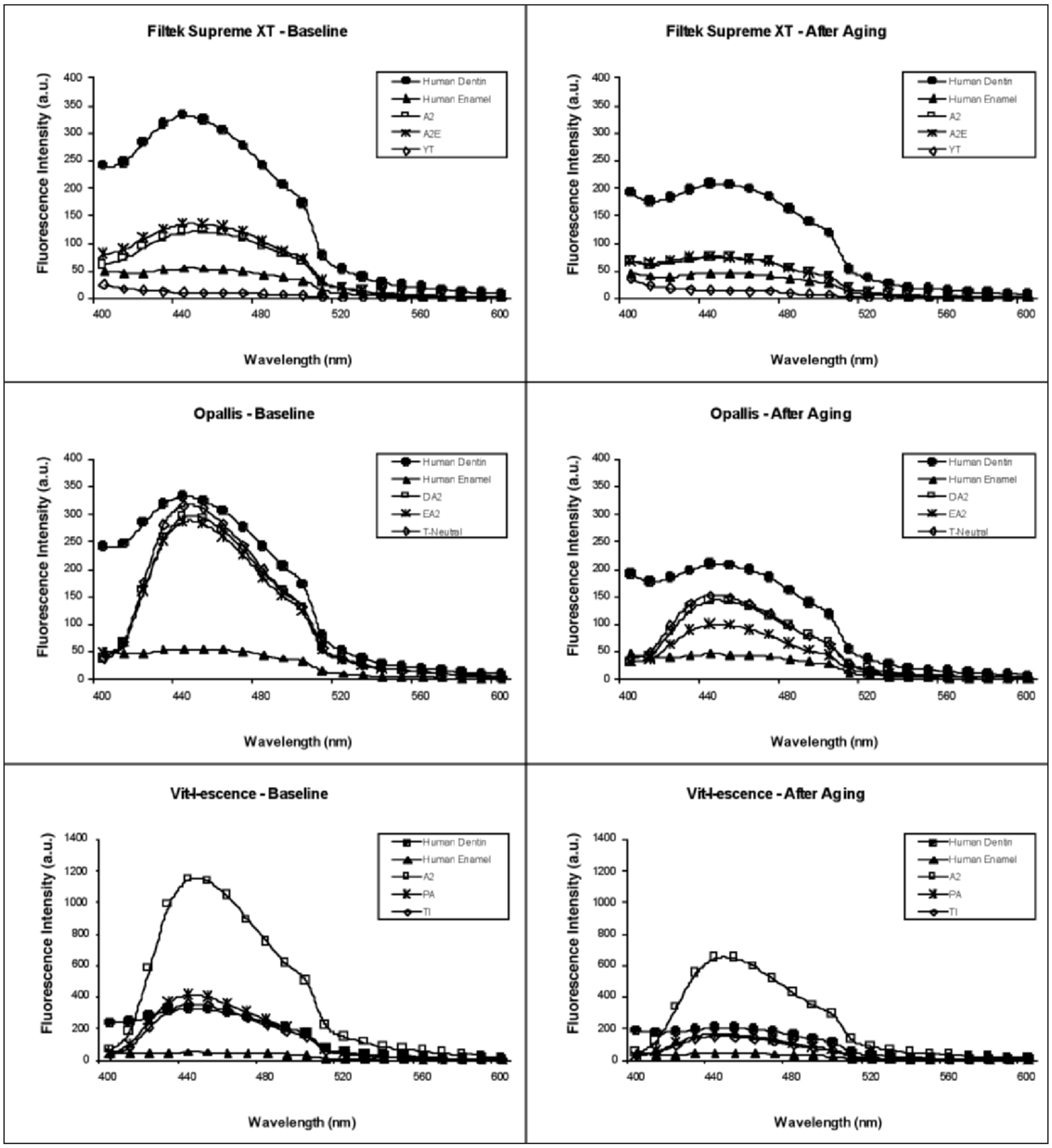

Figure 2. *Fluorescence intensity spectra of Filtek Supreme XT, Opalis and Vit-l-escence, with the controls enamel and dentin, ranging from 400 nm to 600 nm.*

ature and the length of time for which heat was applied. In the current study, however, the fluorescence of dentin was found to decrease following aging. Nevertheless, the accelerated aging process should not be compared with physiological aging, in which several factors that lead to the induction and production of fluorescent substances occur over time. In accelerated aging, which was carried out in this study, temperature may have acted as a catalyst, speeding up photocleavage of organic bonds.[29]

The fluorescence intensities of human enamel and translucent Filtek Supreme XT were extremely low, even at baseline. After aging, changes in the fluorescence of these materials were not significant. The fluorescence of enamel is mainly attributed to its organic components, which account for less than 2% of its total composition. Because of the weak fluorescence of enamel, some studies have evaluated the optical properties of teeth using only dentin specimens[2] or the flat surfaces of bovine or human teeth.[7,13-14] In the current study, enamel and dentin were assessed separately in order to determine their fluorescence intensities before and after accelerated aging of both dental tissues.

CONCLUSIONS

Within the limitations of this study, the null hypothesis of this investigation was rejected, as the fluorescence intensities of the composites tested were not only different, but were also different from those of the controls.

Before aging, the resin composites Esthet-X for dentin, Vit-l-escence for enamel, Vit-l-escence translucent and all the shades of Opallis and Charisma had fluorescent intensities comparable to that of human dentin.

The alternative hypothesis was accepted, except for human enamel and translucent Filtek Supreme XT, which were not affected by accelerated aging, and the fluorescence intensity of all the other materials tested had decreased. Thus, when subjected to accelerated aging, most of the materials tested had poor fluorescence stability.

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