Fluorescence of composite resins: A comparison among properties of commercial shades

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The aim of this study was to determine singular fluorescence qualities of different, commercially available resin composites shades. A total of 234 brand name colors including enamel, dentin and special shades were examined using a monochromator-based spectrophotometer. From the examined composites, Filtek™ Z250 (867±279) RFU and Supreme XT (dentin shades: (1,585±507) RFU; enamel shades: (4,473±330) RFU) are the only brands with a mean fluorescence maximum that resembles the fluorescence of natural samples. The shade types of the other brands showed a three to fifteen times higher mean maximum fluorescence (dentin shades: (10,351–47,774) RFU; enamel shades: (19,283–38,264) RFU; special shades: (35,934–60,001) RFU). The results of the present study supply for the first time individual fluorescence qualities of a vast sample of different composite shades, data needed not only for the development of new materials, but for diagnostic reasons in routine (re-)treatment, forensic and epidemiological endeavors.

Keywords: Fluorescence, Composite resin shades, Restoration, Diagnosis

INTRODUCTION

An aesthetic dental filling material shows optimal properties only when it reflects light or disperses fluorescent light in a similar way as the natural tooth.

In the past decade, the development and improvement needs of new composite tooth filling materials emitting fluorescence, and thus emulating the fluorescence behavior of the natural tooth, has become subject of research attention in dentistry. Fluorescence emitting substances release more perceptible light than the light falling on it, making it look brighter in contrast to non-fluorescent surrounding substances which, at best, can reflect no more than the visible light that is illuminating it¹. This luminescence phenomenon was already described in 1565 by the physician and botanist Nicolás Monardes². The term fluorescence was formulated later on by the physicist Sir George Gabriel Stokes in 1852³, Strübel stated in 1911 the diagnostic potential of tissue fluorescence and recognized that teeth fluoresced when illuminated with ultraviolet (UV) light⁴. Later observations published by Benedict in 1928 confirmed that dentine fluoresced much more strongly than enamel⁵ and in 1929 that initial carious areas could easily be detected in form of a shadow by the contrast created in relation to the surrounding unaffected tissue when illuminated under UV-light⁶. This fluorescence optical property of teeth makes them look whiter and brighter with an increased amount of UV component in the ambient light⁷.

Nowadays, the use of resin composites as amalgam alternative increases in posterior restorations, but certainly also in large anterior tooth restorations as a less invasive alternative to porcelain crowns. In recent years, the manufacturers of composites have been advertising their products possessing natural tooth-like fluorescence properties. Nevertheless, the fluorescent behavior of composites still does not seem identical to that of natural teeth⁷-¹⁰. Sound teeth generate maximum visible fluorescence emission in the blue-violet spectrum by an excitation wavelength of ultraviolet (UV) or near UV-light of approximately 400 nm. Observations have shown that changes in the UV component of artificial light and even mere daylight (i.e. direct and indirect sunlight) are able to influence the color of resin composites¹¹, as a result of an illuminant metameric failure. Because of this, the esthetic qualities of the chameleon effect of a restoration are affected. Thus, esthetic restorative materials should have comparable fluorescence properties to those of natural teeth¹²,¹³. Otherwise, the chameleon camouflage qualities of a restoration can significantly suffer depending on the illumination source. Consequently, composite restorations may match the color of a natural tooth when observed, for instance under daylight, by a dental unit lamp or by a combination of both, but may not match when the light source increases its UV or near UV-light excitation component. This way a mismatch of the composite filling with the adjacent natural tooth substance may clearly become apparent¹⁴,¹⁵.

On the other hand, the demand for better diagnostic methods detecting esthetic restorations is increasing nowadays. Restorations made with composite resins (today the most common used restorative material in dentistry) can represent an increasing diagnostic challenge, as clinical composite visualization can become
difficult or practically impossible under conventional clinical inspection procedures. To date diagnostic methods do not reach the desired diagnostic power needed, as past caries experience (i.e. the restored tooth) cannot be reliably assessed. This rather unobtrusive, unknown or ignored problem constitutes the current reality and is affecting the quality of clinical diagnosis not only in clinical practice, but also in forensic exploration and epidemiological research/analyses. Furthermore, there is a medical need for technological innovation in order to selectively remove these esthetic materials from teeth. Fluorescence inducing diagnostic devices could provide the possibility, owing to the optical properties of composites, to easily differentiate visually between restorative material and dental substance as composites generally fluoresce differently than the tooth itself.

Thus, new information on the fluorescence behavior of the different composite brands and also its numerous shades are currently needed. Nevertheless, fluorescence properties of sound and carious dental hard tissue have been measured in several studies, just few have evaluated the fluorescence properties of different commercially available dental composite resins materials, and there is a lack of studies analyzing the single properties of several different shades of one brand.

The general aim of this study was to determine the single fluorescence properties of a vast sample of currently available resin composite shades by a standardized procedure, in order to achieve truly comparable results.

**MATERIALS AND METHODS**

In the present study, 234 different colors of 16 different brands, representing a total number of 122 enamel shades, 80 dentin shades, and 32 other special light-curing resin composite shades were analyzed. The materials included 16 of the most commonly used brand name composites: Miris®2 (Coltène-Whaledent, Altstätten, Switzerland), Esthet-X® HD, Ceram-X® Duo, Spectrum® (Dentsply DeTrey, Konstanz, Germany), EcuSphere® (DMG Chemisch-Pharmazeutische Fabrik, Hamburg, Germany), ENAMEL Plus HFO/HRi® (GDF, Rosbach, Germany), Venus®, Venus® Diamond, Charisma® (Heraeus Kulzer, Hanau, Germany), Tetric EvoCeram®, IPS Empress® Direct (Ivoclar-Vivadent, Schaan, Lichtenstein), Filtek™ Supreme XT, Filtek™ Z250 (3M-Espe Dental Products, St. Paul, USA), Amaris® and Grandio® (VOCO, Cuxhaven, Germany).

**Method**

Based on a standardized procedure every composite shade of each single brand was individually packed (5 mm thick) and flattened with a microscope 3×1 inch glass slide (R. Langenbrinck, Emmendingen, Germany) into 96-well black microplates (Corning, Lowell, USA), and light-polymerized with a curing lamp (Bluephase®, Ivoclar Vivadent) for 40 s using soft start mode at a maximum output of (1,200±100) mW/cm². By doing so a reproducible, uniform, plan, shiny composite surface was achieved for every sample. Fluorescence measurements were done 24 h later at (37±0.5)°C using a monochromator-based multimode microplate reader (Synergy™ Mx, BioTek Instruments, Bad Friedrichshall, Germany) calibrated with a 0.001% fluorescein disodium salt solution. The probes were excited with a high energy xenon flash lamp and an angled approach to the well for excitation and emission beam from the top. The vertical offset was 4 mm and the column offset 0 mm. The fluorescence of each specimen was measured 1,880 times at different excitation/emission combinations (Fig. 1) with 5 nm steps and a bandwidth of 9 nm. The fluorescence reader independently repeated the measurement of each excitation/emission combination ten times and presented the result as the mean of the ten consecutive measurements. The delay between the measurements was 1 ms and the delay after plate movement 100 ms. Therefore, this study represents altogether no less than 4,399,200 separate fluorescence measurements.

![Fig. 1 Emission map depicting the peak-like shape of a representative composite sample (i.e. Enamel Plus HFO, shade: OBN).](image-url)
Data analysis
The tremendous amount of fluorescence data had to be reduced first to make comparison of the 234 different shades possible at all. Therefore, it was helpful that the fluorescence intensity function of the composite resins resembled a single mountain peak (Fig. 1), whereas for example the fluorescence of enamel and dentin shades resembled a broad mountain range. Thus, the fluorescence of a shade can be characterized by its fluorescence maximum, i.e., its magnitude in Relative Fluorescence Units (RFU) and its corresponding excitation and emission wavelength in nanometer. These three parameters were compared as shown in Fig. 2. Result calculations were performed using JMP® statistical software, version 10.0.0 (SAS Institute, Heidelberg, Germany). The normal distribution was tested with the Shapiro-Wilk test. Since the data was not normally distributed, the statistical significance of the differences was identified and values calculated using Kruskal-Wallis test and Wilcoxon rank sum test at the significance level of α=0.05. Results are shown as mean±standard error.

For comparison purposes, relevant data was adopted from an own previous study where properties of fluorescence from dentin and enamel of natural human teeth were confirmed by using exactly the same method of analysis.

RESULTS
The three parameters characterizing the fluorescence maximum of every single resin composite shade are presented in Table 1. Maximum fluorescence was achieved at a nearly comparable combination of excitation and emission wavelength between shades, but with strongly varying intensities.

Shade type comparison of brands with special shades
The dentin shades (n=48) showed a mean maximum fluorescence of (32,780±2,367) RFU, the enamel shades (n=98) of (27,998±1,230) RFU and the special shades (n=32) of (44,844±2,342) RFU (Fig. 3A, left side). The differences between special shades and dentin shades (p=0.0003) and between special shades and enamel shades (p<0.0001) are significant, the difference between enamel and dentin shades is not (p=0.1375).

The mean excitation wavelength at the fluorescence maximum was (397.9±0.7) nm for the dentin shades, (398.2±0.5) nm for the enamel shades and (401.3±0.6) nm for the special shades (Fig. 3B, left side). Again, the differences between special shades and dentin shades (p=0.0011) and between special shades and enamel shades (p<0.0007) are significant, while the difference between enamel and dentin shades is not (p=0.9982).

The mean emission wavelength at the emission maximum was (450.9±0.5) nm for the dentin shades, (450.3±0.5) nm for the enamel shades and (451±1) nm for the special shades (Fig. 3C, left part). All differences were not significant (p>0.05).

Shade type comparison of brands without special shades
The mean maximum fluorescence in this group was with
| Manufacturer       | Brand name            | Composition | Shade code | Shade type | Excitation Maximum (nm) | Emission Maximum (nm) | Fluorescence Maximum (RFU) |
|--------------------|-----------------------|-------------|------------|------------|-------------------------|-----------------------|---------------------------|
| 3M ESPE            | FiltekTM Supreme XT   | Nanoparticle| A3E        | Enamel     | 390                     | 455                   | 4,704                     |
|                    |                       |             | A1E        | Enamel     | 390                     | 455                   | 5,858                     |
|                    |                       |             | A2B        | Enamel     | 390                     | 455                   | 3,851                     |
|                    |                       |             | A2E        | Enamel     | 390                     | 460                   | 5,005                     |
|                    |                       |             | A3.5B      | Enamel     | 395                     | 465                   | 2,805                     |
|                    |                       |             | A3B        | Enamel     | 385                     | 460                   | 5,139                     |
|                    |                       |             | A3D        | Dentin     | 385                     | 455                   | 750                       |
|                    |                       |             | A4D        | Dentin     | 385                     | 460                   | 2,501                     |
|                    |                       |             | B2E        | Enamel     | 395                     | 465                   | 3,895                     |
|                    |                       |             | B3D        | Dentin     | 400                     | 470                   | 1,504                     |
|                    |                       |             | B2E        | Enamel     | 395                     | 465                   | 4,424                     |
|                    |                       |             | YT         | Special    | 415                     | 490                   | 581                       |
|                    | Filtek™ Z250 Microhybrid|            | A2        | Enamel     | 390                     | 455                   | 795                       |
|                    |                       |             | A3.5       | Enamel     | 390                     | 460                   | 290                       |
|                    |                       |             | A4        | Enamel     | 385                     | 450                   | 1,633                     |
|                    |                       |             | B3        | Enamel     | 390                     | 455                   | 750                       |
| Coltène-Whaledent  | Miris® 2 Nanohybrid   |             | D S0      | Dentin     | 400                     | 455                   | 23,954                    |
|                    |                       |             | D S1      | Dentin     | 400                     | 455                   | 21,761                    |
|                    |                       |             | D S2      | Dentin     | 395                     | 450                   | 18,168                    |
|                    |                       |             | D S3      | Dentin     | 395                     | 450                   | 16,802                    |
|                    |                       |             | D S4      | Dentin     | 395                     | 450                   | 12,512                    |
|                    |                       |             | D S5      | Dentin     | 395                     | 455                   | 11,984                    |
|                    |                       |             | D S6      | Dentin     | 395                     | 455                   | 9,489                     |
|                    |                       |             | D S7      | Dentin     | 390                     | 450                   | 8,342                     |
|                    |                       |             | E IR      | Enamel     | 395                     | 450                   | 21,467                    |
|                    |                       |             | E NR      | Enamel     | 395                     | 450                   | 28,314                    |
|                    |                       |             | E NT      | Enamel     | 395                     | 450                   | 25,711                    |
|                    |                       |             | E WB      | Enamel     | 395                     | 450                   | 27,659                    |
|                    |                       |             | E WR      | Enamel     | 395                     | 450                   | 30,120                    |
| Dentsply DeTrey    | Ceram-X® Duo Nanoparticle|            | D1        | Dentin     | 395                     | 450                   | 18,254                    |
|                    |                       |             | D2        | Dentin     | 395                     | 450                   | 13,449                    |
|                    |                       |             | D3        | Dentin     | 395                     | 450                   | 8,806                     |
|                    |                       |             | D4        | Dentin     | 395                     | 450                   | 6,721                     |
|                    |                       |             | E1        | Enamel     | 395                     | 450                   | 22,256                    |
|                    |                       |             | E2        | Enamel     | 395                     | 450                   | 20,195                    |
|                    |                       |             | E3        | Enamel     | 395                     | 450                   | 15,397                    |
| Dentsply DeTrey    | Esthet.X® HD Microhybrid|           | A1        | Enamel     | 395                     | 450                   | 29,724                    |
|                    |                       |             | A2        | Enamel     | 395                     | 450                   | 22,413                    |
|                    |                       |             | A2-O      | Dentin     | 395                     | 455                   | 14,645                    |
|                    |                       |             | A3        | Enamel     | 395                     | 450                   | 18,381                    |
|                    |                       |             | A3.5      | Enamel     | 395                     | 450                   | 9,966                     |
|                    |                       |             | A4        | Enamel     | 395                     | 450                   | 18,982                    |
|                    |                       |             | A4-O      | Dentin     | 395                     | 450                   | 16,118                    |
|                    |                       |             | AE        | Special    | 400                     | 450                   | 38,361                    |
|                    |                       |             | B1        | Enamel     | 395                     | 450                   | 26,535                    |
|                    |                       |             | B2        | Enamel     | 395                     | 450                   | 19,406                    |
|                    |                       |             | B2-O      | Dentin     | 395                     | 450                   | 16,145                    |
|                    |                       |             | B3        | Enamel     | 395                     | 450                   | 17,412                    |
|                    |                       |             | B5        | Enamel     | 395                     | 450                   | 11,844                    |
|                    |                       |             | CE        | Special    | 400                     | 450                   | 35,731                    |
|                    |                       |             | C1        | Enamel     | 395                     | 450                   | 26,457                    |
|                    |                       |             | C1-O      | Dentin     | 395                     | 450                   | 29,657                    |
|                    |                       |             | C2        | Enamel     | 395                     | 450                   | 21,848                    |
|                    |                       |             | C3        | Enamel     | 395                     | 450                   | 25,840                    |
| Manufacturer | Brand name           | Composition | Shade code | Shade type | Excitation Maximum (nm) | Emission Maximum (nm) | Fluorescence Maximum (RFU) |
|-------------|---------------------|-------------|------------|------------|--------------------------|------------------------|---------------------------|
| Dentsply DeTrey | Esthet.X® HD Microhybrid | C4          | Enamel     | C4         | 395                      | 450                    | 15,139                    |
|             |                     | C4-O        | Dentin     | C4-O       | 395                      | 450                    | 13,859                    |
|             |                     | C5          | Enamel     | C5         | 395                      | 450                    | 14,301                    |
|             |                     | D2          | Enamel     | D2         | 395                      | 450                    | 29,254                    |
|             |                     | D3          | Enamel     | D3         | 400                      | 450                    | 24,618                    |
|             |                     | D3-O        | Dentin     | D3-O       | 395                      | 450                    | 26,018                    |
|             |                     | GE          | Special    | GE         | 400                      | 450                    | 41,683                    |
|             |                     | U           | Enamel     | U          | 395                      | 450                    | 24,038                    |
|             |                     | W           | Special    | W          | 400                      | 450                    | 54,725                    |
|             |                     | WE          | Special    | WE         | 400                      | 450                    | 42,639                    |
|             |                     | WO          | Dentin     | WO         | 400                      | 450                    | 50,011                    |
|             |                     | XL          | Special    | XL         | 400                      | 450                    | 45,331                    |
|             |                     | YE          | Special    | YE         | 400                      | 450                    | 34,609                    |
|             | Spectrum TPH®3 Microhybrid | A1         | Enamel     | A1         | 400                      | 450                    | 34,279                    |
|             |                     | A2          | Enamel     | A2         | 395                      | 450                    | 23,756                    |
|             |                     | A3          | Enamel     | A3         | 395                      | 450                    | 26,191                    |
|             |                     | A3.5        | Enamel     | A3.5       | 395                      | 450                    | 20,055                    |
|             |                     | B1          | Dentin     | B1         | 400                      | 450                    | 36,094                    |
|             |                     | B2          | Dentin     | B2         | 395                      | 450                    | 24,083                    |
| DMG Chemisch-Pharmazeutische Fabrik | EcuSphere®- Carat Microhybrid | A          | Dentin     | A           | 400                      | 450                    | 18,532                    |
|             |                     | A1          | Dentin     | A1         | 400                      | 450                    | 34,581                    |
|             |                     | A2          | Dentin     | A2         | 400                      | 450                    | 19,611                    |
|             | EcuSphere®- Shape Microhybrid | A1         | Dentin     | A1         | 400                      | 450                    | 259                       |
|             |                     | A2          | Dentin     | A2         | 400                      | 450                    | 21,407                    |
|             |                     | A3          | Dentin     | A3         | 400                      | 450                    | 21,730                    |
|             |                     | A3.5        | Dentin     | A3.5       | 400                      | 450                    | 16,728                    |
|             |                     | A4          | Dentin     | A4         | 395                      | 465                    | 141                       |
|             |                     | B1          | Dentin     | B1         | 410                      | 495                    | 176                       |
|             |                     | B2          | Dentin     | B2         | 395                      | 460                    | 50                        |
|             |                     | B3          | Dentin     | B3         | 420                      | 520                    | 67                        |
|             |                     | C1          | Dentin     | C1         | 410                      | 510                    | 486                       |
|             |                     | C3          | Dentin     | C3         | 405                      | 445                    | 168                       |
|             |                     | D-A3        | Dentin     | D-A3       | 400                      | 450                    | 20,678                    |
|             |                     | D2          | Dentin     | D2         | 390                      | 455                    | 364                       |
| EcuSphere®- Shine Nanohybrid | A2          | Enamel     | A2         | 400                      | 450                    | 18,781                    |
|             |                     | A3          | Enamel     | A3         | 400                      | 450                    | 22,643                    |
|             |                     | TR          | Enamel     | TR         | 400                      | 450                    | 41,553                    |
| GDF - Gesellschaft für Dentale Forschung und Innovationen | Enamel Plus HFO® Microhybrid | GE1        | Enamel     | GE1        | 400                      | 450                    | 29,537                    |
|             |                     | GE2         | Enamel     | GE2        | 400                      | 450                    | 29,824                    |
|             |                     | GE3         | Enamel     | GE3        | 400                      | 450                    | 32,946                    |
|             |                     | IM          | Special    | IM         | 400                      | 450                    | 55,359                    |
|             |                     | IW          | Special    | IW         | 400                      | 450                    | 55,044                    |
|             |                     | OA          | Special    | OA         | 400                      | 450                    | 13,321                    |
|             |                     | OBN         | Special    | OBN        | 400                      | 445                    | 62,800                    |
|             |                     | OG          | Special    | OG         | 405                      | 450                    | 28,257                    |
|             |                     | OW          | Special    | OW         | 400                      | 450                    | 43,528                    |
|             |                     | UD1         | Dentin     | UD1        | 400                      | 450                    | 50,189                    |
|             |                     | UD2         | Dentin     | UD2        | 395                      | 450                    | 40,380                    |
|             |                     | UD3         | Dentin     | UD3        | 395                      | 450                    | 36,716                    |
|             |                     | UD4         | Dentin     | UD4        | 395                      | 450                    | 29,463                    |
|             |                     | UD5         | Dentin     | UD5        | 395                      | 450                    | 25,403                    |
|             |                     | UD6         | Dentin     | UD6        | 395                      | 450                    | 23,708                    |
| Manufacturer                      | Shade code | Shade type | Excitation Maximum (nm) | Emission Maximum (nm) | Fluorescence Maximum (RFU) |
|-----------------------------------|------------|------------|-------------------------|-----------------------|---------------------------|
| GDF - Gesellschaft für Dentale    | UD0        | Dentin     | 400                     | 450                   | 70,808                    |
| Forschung und Innovationen        | UD0.5      | Dentin     | 400                     | 450                   | 62,624                    |
|                                   | UD1        | Dentin     | 395                     | 450                   | 63,275                    |
|                                   | UD2        | Dentin     | 395                     | 450                   | 53,634                    |
|                                   | UD3        | Dentin     | 395                     | 450                   | 50,503                    |
|                                   | UD3.5      | Dentin     | 395                     | 450                   | 45,223                    |
|                                   | UD4        | Dentin     | 395                     | 450                   | 38,057                    |
|                                   | UD5        | Dentin     | 400                     | 450                   | 22,888                    |
|                                   | IWS        | Special    | 400                     | 450                   | 49,448                    |
|                                   | UD6        | Dentin     | 395                     | 450                   | 22,951                    |
|                                   | UE1        | Enamel     | 395                     | 430                   | 15,787                    |
|                                   | UE2        | Enamel     | 395                     | 430                   | 20,410                    |
|                                   | UE3        | Enamel     | 395                     | 430                   | 23,027                    |
| Charisma®                         | A1         | Enamel     | 405                     | 450                   | 44,784                    |
|                                   | A2         | Enamel     | 405                     | 450                   | 44,436                    |
|                                   | A3         | Enamel     | 405                     | 450                   | 38,754                    |
|                                   | A3.5       | Enamel     | 405                     | 450                   | 32,993                    |
|                                   | A4         | Enamel     | 405                     | 450                   | 34,918                    |
|                                   | B1         | Enamel     | 405                     | 450                   | 50,063                    |
|                                   | B2         | Enamel     | 405                     | 450                   | 34,396                    |
|                                   | B3         | Enamel     | 405                     | 450                   | 35,312                    |
|                                   | C2         | Enamel     | 405                     | 450                   | 36,811                    |
|                                   | C3         | Enamel     | 405                     | 450                   | 32,244                    |
|                                   | C4         | Enamel     | 405                     | 450                   | 26,362                    |
|                                   | D4         | Enamel     | 405                     | 450                   | 40,095                    |
|                                   | I          | Special    | 405                     | 450                   | 59,325                    |
|                                   | OA2        | Dentin     | 405                     | 450                   | 39,850                    |
|                                   | OA3        | Dentin     | 405                     | 450                   | 34,230                    |
|                                   | OA3.5      | Dentin     | 405                     | 455                   | 28,822                    |
|                                   | OB2        | Dentin     | 405                     | 450                   | 39,387                    |
|                                   | SL         | Special    | 405                     | 450                   | 61,564                    |
|                                   | SLO        | Special    | 405                     | 450                   | 59,129                    |
| Heraeus Kulzer                    | A1         | Enamel     | 405                     | 450                   | 46,230                    |
|                                   | A2         | Enamel     | 405                     | 450                   | 41,139                    |
|                                   | A3         | Enamel     | 405                     | 450                   | 36,231                    |
|                                   | A3.5       | Enamel     | 405                     | 450                   | 31,362                    |
|                                   | A4         | Enamel     | 395                     | 450                   | 22,222                    |
|                                   | B1         | Enamel     | 405                     | 450                   | 48,026                    |
|                                   | B2         | Enamel     | 405                     | 450                   | 39,632                    |
|                                   | B3         | Enamel     | 405                     | 450                   | 29,598                    |
|                                   | C2         | Enamel     | 405                     | 450                   | 36,575                    |
|                                   | C3         | Enamel     | 405                     | 450                   | 31,992                    |
|                                   | C4         | Enamel     | 405                     | 450                   | 25,597                    |
|                                   | D2         | Enamel     | 405                     | 450                   | 40,226                    |
|                                   | D3         | Enamel     | 405                     | 450                   | 33,304                    |
| Venus®                            | HKA2.5     | Enamel     | 405                     | 450                   | 39,004                    |
|                                   | HKA5       | Enamel     | 405                     | 450                   | 21,165                    |
|                                   | OA2        | Dentin     | 405                     | 450                   | 35,122                    |
|                                   | OA3        | Dentin     | 405                     | 450                   | 30,896                    |
|                                   | OA3.5      | Dentin     | 400                     | 450                   | 24,437                    |
|                                   | OB2        | Dentin     | 405                     | 450                   | 32,146                    |
|                                   | OC3        | Dentin     | 405                     | 450                   | 27,818                    |
|                                   | OD2        | Dentin     | 405                     | 450                   | 31,659                    |
|                                   | SB1        | Enamel     | 405                     | 450                   | 54,851                    |
|                                   | SB2        | Enamel     | 405                     | 450                   | 64,341                    |
|                                   | SBO        | Dentin     | 405                     | 450                   | 66,185                    |
| Manufacturer     | Brand name      | Composition | Shade code | Shade type | Excitation Maximum (nm) | Emission Maximum (nm) | Fluorescence Maximum (RFU) |
|------------------|-----------------|-------------|------------|------------|-------------------------|------------------------|---------------------------|
| Heraeus Kulzer   | Venus® Microhybrid | T1          | Special    | 405        | 450                     | 54,958                 |
|                  |                 | T2          | Special    | 405        | 450                     | 51,657                 |
|                  |                 | T3          | Special    | 405        | 450                     | 53,196                 |
|                  | Venus Diamond® Nanohybrid | A1          | Enamel    | 400        | 450                     | 42,795                 |
|                  |                 | A2          | Enamel    | 400        | 450                     | 40,565                 |
|                  |                 | A3          | Enamel    | 400        | 450                     | 37,436                 |
|                  |                 | A3.5        | Enamel    | 400        | 450                     | 38,058                 |
|                  |                 | A4          | Enamel    | 395        | 450                     | 33,535                 |
|                  |                 | AM          | Special    | 400        | 450                     | 38,341                 |
|                  |                 | B1          | Enamel    | 400        | 450                     | 45,838                 |
|                  |                 | B2          | Enamel    | 400        | 450                     | 38,793                 |
|                  |                 | B3          | Enamel    | 400        | 450                     | 35,656                 |
|                  |                 | BL          | Special    | 400        | 450                     | 51,372                 |
|                  |                 | BXL         | Special   | 400        | 450                     | 52,132                 |
|                  | IPS Empress® Direct Nanohybrid | C2          | Enamel    | 395        | 450                     | 35,329                 |
|                  |                 | C3          | Enamel    | 395        | 450                     | 32,024                 |
|                  |                 | CL          | Special    | 400        | 450                     | 39,093                 |
|                  |                 | CO          | Special    | 400        | 450                     | 49,365                 |
|                  |                 | D3          | Enamel    | 395        | 450                     | 36,609                 |
|                  |                 | HKA2.5      | Enamel    | 400        | 450                     | 38,249                 |
|                  |                 | HKA5        | Enamel    | 395        | 450                     | 32,366                 |
|                  |                 | OB          | Dentin    | 400        | 450                     | 55,101                 |
|                  |                 | OD          | Dentin    | 400        | 450                     | 35,683                 |
|                  |                 | OL          | Dentin    | 400        | 450                     | 39,273                 |
|                  |                 | OM          | Dentin    | 400        | 450                     | 40,824                 |
|                  |                 | YO          | Special    | 400        | 450                     | 54,133                 |
|                  | IPS Empress® Direct Nanohybrid | opal trans | Special  | 395        | 450                     | 46,476                 |
|                  |                 | 30 trans    | Special   | 400        | 450                     | 45,859                 |
|                  |                 | A1D         | Dentin    | 395        | 450                     | 45,993                 |
|                  |                 | A1E         | Enamel    | 395        | 450                     | 44,087                 |
|                  |                 | A2D         | Dentin    | 400        | 450                     | 33,505                 |
|                  |                 | A2E         | Enamel    | 395        | 450                     | 37,788                 |
|                  |                 | A3D         | Dentin    | 395        | 450                     | 32,643                 |
|                  |                 | A3E         | Enamel    | 395        | 450                     | 32,918                 |
| Ivoclar-Vivadent | Tetric EvoCeram® Nanohybrid | opal trans | Special  | 395        | 450                     | 36,760                 |
|                  |                 | 30 trans    | Special   | 400        | 450                     | 28,377                 |
|                  |                 | A1D         | Dentin    | 395        | 450                     | 26,188                 |
|                  |                 | A1E         | Enamel    | 395        | 450                     | 20,580                 |
|                  |                 | A2D         | Dentin    | 400        | 450                     | 17,856                 |
|                  |                 | A2E         | Enamel    | 395        | 450                     | 15,027                 |
|                  |                 | A3D         | Dentin    | 395        | 450                     | 14,985                 |
|                  |                 | A3E         | Enamel    | 395        | 450                     | 26,028                 |
|                  |                 | A1          | Enamel    | 395        | 450                     | 24,324                 |
|                  |                 | A2          | Enamel    | 395        | 450                     | 20,142                 |
|                  |                 | A3          | Enamel    | 395        | 450                     | 27,989                 |
|                  |                 | A3.5        | Enamel    | 395        | 450                     | 23,596                 |
|                  |                 | A3.5D       | Dentin    | 395        | 450                     | 24,628                 |
|                  |                 | A4          | Enamel    | 395        | 450                     | 35,934                 |
| VOCO             | Amaris® Microhybrid | O1          | Dentin    | 400        | 450                     | 34,373                 |
|                  |                 | O2          | Dentin    | 395        | 450                     | 29,034                 |
|                  |                 | O3          | Dentin    | 400        | 450                     | 21,842                 |
|                  |                 | O4          | Dentin    | 395        | 450                     | 15,780                 |
|                  |                 | O5          | Dentin    | 395        | 450                     | 12,832                 |
|                  |                 | TD          | Enamel    | 395        | 450                     | 23,728                 |
|                  |                 | TL          | Enamel    | 400        | 450                     | 44,382                 |
|                  |                 | TN          | Enamel    | 400        | 450                     | 31,713                 |
(13,721±1,799) RFU significantly lower ($p=0.0011$) for the dentin shades ($n=32$) than for the enamel shades ($n=24$) with (22,577±2,463) RFU (Fig. 3A, right side).

The mean excitation wavelength of the fluorescence maximum was (399±1) nm for the dentin shades and, therefore, significantly ($p=0.0429$) higher than for the enamel shades with (395.4±0.8) nm (Fig. 3B, right side).

There were no significant differences ($p=0.2098$) for the mean emission wavelength of the fluorescence maximum between the dentin shades with (458±3) nm and the enamel shades with (450.8±0.5) nm (Fig. 3C, right side).

**Comparison of the same shade type between the two groups**

The mean fluorescence maximum (Fig. 3A) of dentin shades from the brands without special shades was with (13,721±1,799) RFU significantly ($p<0.0001$) lower than of dentin shades from brands including special shades with (32,780±2,367) RFU. There was no significant difference ($p=0.0622$) between the enamel shades of the two brand types (brands with special shades: (27,998±1,230) RFU; brands without special shades: (22,577±2,463) RFU).

The mean excitation wavelength of the fluorescence maximum (Fig. 3B) showed no difference ($p=0.8411$) for the dentin shades (brands with special shades: (397.9±0.7) nm; brands without special shades: (399±1) nm), whereas the difference for the enamel shades was significantly ($p=0.0236$) different (brands with special shades: (398.1±0.5) nm; brands without special shades: (395.0±0.8) nm).

The mean emission wavelength at the fluorescence maximum showed no significant differences for the dentin shades ($p=0.0736$; brands with special shades: (450.9±0.5) nm; brands without special shades: (458±3) nm), and the enamel shades ($p=0.4560$; brands with special shades: (450.2±0.5) nm; brands without special shades: (450.0±0.5) nm).

Mean fluorescence maximum comparison of the different brands

Figure 4 depicts the mean fluorescence maxima differentiated according to the brands and shade types. The mean fluorescence data of natural dentin and enamel samples of a recent publication\(^{10}\) using the same method of analysis of the present study was added for comparison. These samples (including enamel and dentin samples) were excited at 400 nm (Fig. 3B) and the fluorescence detected at 450 nm (Fig. 3C).

Filtek™ Supreme XT was the brand with the lowest overall fluorescence and the only brand where the fluorescence of the special shade group was lower than that of dentin and enamel shades. Apart from Filtek™ Supreme XT, Filtek™ Z250 is the only brand with a fluorescence that resembles the fluorescence of the natural samples. The mean fluorescence of the shade types of the other brands was between 3 and 15 times higher than the natural fluorescence of enamel and dentin.

The special shade group of the other brands showed the highest mean fluorescence of the three shade groups. Nevertheless, five brands showed a higher mean fluorescence maximum for the enamel shades as for the dentin shades of the brand (Charisma\(^{\circledR}\),...
Fig. 3 A depicts boxplots of the maximum fluorescence in Relative Fluorescence Units (RFU), B of the excitation wavelength of the fluorescence maximum and C of the emission wavelength of the fluorescence maximum.

Fig. 4 The mean fluorescence maximum in Relative Fluorescence Units (RFU) of the assessed brands differentiated according to the shade type. At the right the mean fluorescence of dentin and enamel samples is depicted for comparison. These samples were excited at 400 nm and the fluorescence detected at 450 nm.

Grandio®, IPS Empress® Direct, Tetric EvoCeram® and Venus® showed a higher maximum for the dentin shades in comparison to the enamel shades of the brand (Enamel Plus HFO / HRi®, Esthet.X HD® and Venus® Dimond).

Due to the low number of shades in some groups (n<4), this data was not further analyzed statistically.

DISCUSSION

Aim of this study was to examine current composite materials with respect to their fluorescent properties. On account of the fact that the precise composition of composite materials —and especially the composition and concentration of the fluorophores used— is not published by manufacturing companies, the nature of this study can only be descriptive, and no causal relationship can be offered where the measured fluorescent properties are concerned.

The present study was carried out with a multi-mode monochromator-based device of the last generation that allows fluorescence measurements in a wavelength-range from 250 up to 900 nm in 1 nm increments with a repeatability of ±0.2 nm. This allowed a fast and sensitive measurement of a vast number of samples.

There is some published data assuming that fluorescence properties of resin composites may notably change as a consequence of aging. Storage conditions were kept equal for all samples and fluorescence measurements performed at a constant temperature.

By the method of analysis, it was assumed that the composition of specimens taken from a single batch is...
homogenous. Consequently inter-lot-variations were not addressed in this study.

For fluorescence analysis, resin composites were classified in three groups (i.e. enamel shades, dentin shades and special shades) according to their corresponding manufacturers indications in terms of multi-layer/shade-based restorations concepts30-33 for the use of translucent (i.e. enamel) and opaque (i.e. dentin) resin composite materials. The special shades group included composite masses used for reproducing characterizations, e.g. transparent, amber as well as intensive-white shades.

Although there are several studies analyzing the fluorescence qualities of dental resin composites6,9,22-27, no previous publication comparing the individual fluorescence properties of several different shades of one brand was found in the literature. Furthermore, since the objectives of these studies and methods employed differ, the results are not straightforwardly comparable. In a previous study10 using the same method only the shade codes of the conventional VITA shade guide were used.

In the present study some statistical differences were found in the corresponding optimal excitation wavelength between the shades, in order to ensure maximum fluorescence. However, even though some differences in the area of the wavelength are significant, the question arises as to what the clinical relevance of modifications in the illumination source might be. As of now it is not completely clear, if these statistical differences may have much real influence on the visual fluorescence behavior of composite restorations in the clinical situation. It might not be likely, as these excitation differences do not implicate major variations in the fluorescence inducing light spectrum of the illumination source. In any case, there is no doubt of the fact that there is a relatively broader peak when analyzing excitation compared to emission and that almost the maximum fluorescence induction can surely be reached for all the shades examined, if stimulated by an illumination source between 395 and 405 nm.

When analyzing emission, no statistical significant differences were found between the shades. The predominant detection optimum is at 450 nm.

The results show that the analyzed resin composites fluoresced with strong varying maximum fluorescence intensities. This evaluation disclosed the lack of standardized fluorescent properties between the different resin composite shades, even of those from one brand. The statistical differences between dentin shades and special shades as well as between enamel shades and special shades were found to be significant in contrast to the difference between enamel and dentin shades in the analysis of brands including the special shades. In this group, it is the special shades that fluoresce the most, whereas enamel and dentin shades fluoresce notably less and show no differences in their fluorescence intensity values. On the contrary, for the brands vended without special shades, the statistical differences analyzing maximum fluorescence intensities between dentin and enamel shades were significant. In this group, the enamel shades fluoresce the most. Consequently, the shades used on or just below the surface (in accordance with the multi-layer/shade-based restoration technique) have a higher capacity to fluoresce. This is in contrast to the natural tooth, in which dentin exhibits more fluorescence than enamel. When comparing a brand with or without special shades, a statistical significant difference was found between the dentin shades in regard to maximum fluorescence intensities. By comparing the fluorescence maximum of different brand shades types (Fig. 4), it furthermore became evident how heterogeneous the analysis of fluorescence intensities can be, depending on which composite brand and/or shade type was being analyzed. The results demonstrate that the analyzed composite brand shade types reached their maximum fluorescence with distinctive varying fluorescence intensities. Furthermore, the special shades showed a tendency to increase the maximum fluorescence intensities, which possibly is counterproductive from an esthetic point of view, since precisely these shades are commonly used for the external composite layer of a filling, making it most susceptible to an illuminant metameric fluorescence failure in contrast to the adjacent natural tooth substance. Consequently, for developing a more natural fluorescence in composites, intensities have to be reduced and fluorophores need to be incorporated additionally, which fluoresce at lesser excitation wavelengths. On the other hand, from an epidemiological and forensic point of view it is beneficial that composites do not have natural fluorescence. Because, once natural fluorescence is perfectly imitated, composites can no longer be distinguished from the tooth by fluorescence diagnostic lamps. In this case the addition of fluorophores emitting in the infrared range could be considered in future research, however, necessitating the development of a detection device, since these fillings were neither visible to the human eye nor detectable by fluorescence measurements.

Differences in fluorescence intensities between composites already are described in previous analyses6,9,21,25. However, none of them depicted the individual maximum fluorescence intensity properties attainable for the different composite shades masses of several different brands. Nevertheless, some previous studies27,25 simply assumed having reproduced the fluorescence intensity properties of a brand by solely analyzing one of its several shades. As shown, this may not reflect the actual properties of the different shades of a brand. Moreover, this behavior seems not to be influenced much by other physical properties of the material—like the composite filler properties characterizing a specific composite brand—, since differences in fluorescence intensities also occurred in the analysis of composites of one brand (Fig. 4 and Table 1).

Results also show that the great minority (<20%) of the composite shades achieved maximum RFU emission value, comparable with the results of a previous work analyzing biological samples of enamel and dentin10.
As shown in the results, Filtek™ Supreme XT and Filtek™ Z250 were the only brands in which different shades achieved fluorescence values comparable to those of natural samples. The mean fluorescence of the other type brand shades was much higher than that of biological samples. Nevertheless, it is not yet known which implications these findings may have on the fluorescence properties of a restoration matching tooth substance in the clinical situation. Studies analyzing the clinical fluorescence behavior of filings using several composite shades of the same brand are missing, and previous studies assuming to reproduce the fluorescence clinical performance of a brand by solely analyzing one of its several shades are inappropriate, due to the presented heterogeneous fluorescence properties of resin composites. Shade combinations might lead to undesirable changes in the fluorescence properties of the restored tooth. Studies simulating the clinical situation using a vast number of shade combination samples of resin composites will require extensive work, as not only different brands, but also their different shades need to be included, and fillings in differing thickness layer combinations need to be performed, in order to get further understanding about how fluorescence of current resin composite filling materials behave de facto in the clinical situation. Nevertheless, as demonstrated in the present findings, the momentary reality of fluorescence discrepancies between composite shades makes it evident that new improved materials are needed, if only for esthetic purposes. Owing to the results, it seems that the majority of the analyzed composite shades might achieve a stronger visual fluorescence clinically, when illuminated by a light source of the right wavelength in comparison to the biological tooth substance. Owing to the presented heterogeneous properties, large restorations with comparable fluorescence qualities to those of adjacent natural tooth substances should be difficult or impossible to be performed with the analyzed composites. As soon as multiple shade-layers combinations need to be applied for an esthetic outcome of fillings, the fluorescence optical appearance can —from an esthetic point of view— be negatively and unpredictably influenced. Since the fluorescence properties among the composite shades and those of natural hard tooth substance drastically differ, the presented fluorescence differences among the composite shades of one brand seem, from a metameric and esthetic point of view, still suboptimal for the great majority of the analyzed composite shades types. It seems that metameric fluorescence failure in multi-layer/shade-based composite restorations remains, to some extent, an illuminant-depending esthetic problem. Unfortunately, this problem cannot be fully avoided under varying lighting conditions, as this seemed to be mainly dependent on the fluorescence behavior of the resin composite type. Consequently, in large composite restorations performed by the present analyzed resin composite shades, the clinical esthetic optical behavior of composite restorations will most likely suffer when exposed to fluorescence inducing light, making them to fluoresce visually much stronger than the contiguous natural tooth substance.

It has been stated in previous studies that the visual fluorescence intensity of composite fillings may be regulated essentially by the exterior composite layer26, even if the layer is thin30. However, this observation did not analyze vast composite brands or the fluorescence behavior by combining their several shades. In contrast, it seems that the fluorescence clinical appearance depends on a much more complex interrelation, in which not only the thickness of the last composite layer, but also several secondary factors like the brand and/or its shades may play a crucial role. This dynamic multi-factorial phenomenon influencing visual fluorescence and its clinical manifestation will depend on the physical interaction of three primary factors: the substance (composite/tooth), the light source (illuminant wavelength and intensity) and the observer (human eye). Changes in the qualities of each of these primary factors alone can influence fluorescence visual perception (Fig. 5). For instance, variations in the translucency/opacity of the last composite layer of a composite filling alone can influence fluorescence visualization, as the fluorescence qualities of non-superficial composite layers can become visually more or less evident, disturbing the chameleon effect of a filling.

Nonetheless, this optical fluorescence metameric failure, attainable by the majority of the composite shades analyzed, allows the particular advantage of identifying the composite as a restoration and/or as a persistent rest of it —in case of retreatment for instance— allowing a previously incomparable level of diagnostic accuracy in the clinical situation. This could help to define better diagnostic standards for achieving much more accurate procedures necessary for principles.

Fig. 5 Primary factors involved in fluorescence etiology.
of “good clinical practice” involving ethical and forensic aspects.

Summing up, the results demonstrate that the analyzed composite brand shade types reached their maximum fluorescence at about the same excitation and at equal emission wavelengths, but with distinctive varying fluorescence intensities. The results provide useful individual fluorescence data for the single shades of internationally known brands of resin composites. Further development of fluorescence qualities of resin composites is needed to find equally comparable fluorescence properties between shades and brands of present resin composites, not only for esthetic reasons, but also for the development of reliable methods for composite filling identification in restorative dentistry that may replace conventional diagnostic tools in the near future. This may well account for the efforts of contributing to the development of more consistent and application-oriented materials compatible with the present needs of these materials.

DISCLOSURE STATEMENT

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