

# Difficulties in measuring the colour variations of the composite materials

M. IONAȘ<sup>a</sup>, M. MOLDOVAN<sup>b</sup>, L. SILAGHI DUMITRESCU<sup>b</sup>, T. HORAȚIU IONAȘ<sup>c</sup>, G. BOȚA<sup>a</sup>

<sup>a</sup>“Lucian Blaga” University of Sibiu, Romania, Faculty of Medicine, 2A Lucian Blaga Str., 550169, Sibiu, Romania

<sup>b</sup>Babes Bolyai University - Raluca Ripan Chemistry Research Institute, 30 Fantanele Str, 400294, Cluj-Napoca, Romania

<sup>c</sup>DMD, private dental office AMIC Ltd, 26, 9 Mai Str., 550201, Sibiu, Romania

Spectrophotometers are used in dental practice in order to determine colour changes of dental composites as accurate as possible. The aim of this study is to find out if the VITA Easyshade dental spectrophotometer and the laboratory UNICAM 4 UV-VIS spectrophotometer have similar efficiency in measuring the colour of dental composites regardless of their inorganic filling. The results indicate that dental spectrophotometers have difficulties in detecting colour changes of dental nanocomposites.

(Received September 19, 2015; accepted October 28, 2015)

*Keywords:* Nanocomposite, Spectrophotometer, Colour variation

## 1. Introduction

The study of colour is an important part of aesthetic dentistry. Among the most important objectives of both dentist and patient is to imitate the colour of the dental tissues by means of restorative materials [1, 2]. The most aesthetically desired among the materials for direct restoration are the composite materials [3, 4]. Regarding their colour, not only the choice of the initial colour is important, but also the colour modifications that occur in time [5, 6, 7].

The colour modifications in time of dental composites can occur due to intrinsic and extrinsic factors. Intrinsic factors are represented by the chemical stability of resin matrix and matrix/particles interface, and extrinsic factors are related to the absorption of staining solutions.

There are two ways of measuring colour in dentistry, one of them is subjective, using shade guides, and the other one objective, using mainly spectrophotometers [1].

To permanently choose an accurate shade match is difficult. Several electronic shade-matching devices for dentistry have been marketed in the past years [9]. Even though dental spectrophotometers have been initially created to determine the colour of dental tissues, [10] more and more studies use them to determine the colour of dental composites [11, 12, 13, 17, 18]. In European dental schools the Vita Easyshade, Advance spectrophotometer is the most frequently used digital system [14].

Vita Easyshade is a portable system for tooth shade determination in the mouth. The spectrometer measures the intensity of the light received in the form of a wavelength in the range of 400–700 nm. The  $L^*a^*b^*$  and  $C^*h^*$  colour space coordinates of the shade are calculated using a D65 illuminant at an observer angle of 2° [26].

The laboratory spectrophotometers can be successfully used to determine colour variations of dental composites [19, 20, 21]. Unicam UV VIS is a compact laboratory spectrophotometer capable of measuring the light radiation with great accuracy in a wavelength interval of 200-900 nm. Starting from the light intensity on different wavelengths are calculated the colour parameters  $L^*a^*b^*$  of the studied samples. The laboratory and the dental spectrophotometers have been designed to be used in different environments. They should be able to measure a sample identically. Dental spectrophotometers must face very high demands regarding hygiene/ disinfection, they must furnish correct information regarding colour and must require a minimum of calibration. The laboratory spectrophotometers enable very precise measurements, require however a controlled work environment, which is not compatible with the clinical use.

The question is, whether the two spectrophotometers have similar capacities to measure the colour of the dental composites no matter of their inorganic filling.

Null hypotheses:

[1] The null hypothesis, that there are no differences between the measurements of the two equipments. The two equipments will measure the parameters which do not differ statistically. It shows that the equipment measure identically.

[2] The null hypothesis, that there are no differences of  $\Delta E^*$  between the two devices. The parameters can be statistically different between the devices, but the calculated colour variation is not much different statistically. It indicates that the devices measure  $\Delta E^*$  identically even if there are differences between the  $L^*a^*b^*$  parameters.

[3] The null hypothesis, that the difference between  $\Delta E^*$  calculated by means of the furnished parameters and

by the two devices, the same material, the same time interval, will not differ from the difference between the samples immersed in wine, for the immersion in water. This means that the difference between  $\Delta E^*$  water spectrophotometer UNICAM 4 UV-VIS-water Easyshade will be similar to the difference between  $\Delta E^*$  wine UNICAM UVVIS-wine Easyshade. A constant interval is preserved between the two  $\Delta E^*$ , no matter of the measurement solution. It shows that the two devices measure  $\Delta E^*$  differently but the difference between them remains constant. Knowing this difference we can make the necessary corrections for the  $\Delta E^*$  values.

## 2. Material and method

We took into study the microhybrid composites Valux Plus shade A2 and an experimental composite produced in the Raluca Ripan Chemistry Research Institute shade A2, as well as a nanocomposite, Filtek Ultimate A2 Body. The samples were realized with a mould having a diameter of 30mm and a thickness of 2mm. These dimensions are necessary so as to be able to be read with the UNICAM UV-VIS spectrophotometer [19]. The composite was inserted and compressed with the spatula in the mould, afterwards a polyester film was applied and pressed with a 1mm thick glass stab. The polymerization of the materials was performed from several directions, 20 seconds for each exposure, on a single surface with a photopolymerization lamp LED Elipar Freelight 2, 3M ESPE, guide Ø 8 mm, 1000mW/cm<sup>2</sup>. Total exposure time was 120 sec, The surface was finished during several steps with abrasive paper with increasing granulation up to 1600 grits. The final thickness of the samples was of 2mm ( $\pm 5\%$ ). The checking of the thickness has been performed with a digital micrometre [21].

We chose to immerse the composites in water, because water determines the slightest modifications of colour and in wine, because wine determines the greatest modifications of colour [22, 23, 24].

The samples were immersed in water and red wine at 37 C. The samples were subsequently removed from the solutions, dried, and then measured after 24 h, 7 days and 28 days with the UNICAM 4 UV-VIS spectrometer and with the Easy Shade spectrophotometer. The measurement was performed on a white background. For every composite material were realised 10 samples, divided randomly in two groups, 5 in each group, each sample being read for three times by means of the spectrophotometer UNICAM 4 UV-VIS and 5 times by means of the spectrophotometer Vita Easyshade advance. The CIE L\*a\*b\* system was chosen to evaluate the colour

variation (DE) because it is appropriate for the determination of small colour changes and has advantages such as repeatability, sensitivity and objectivity [25].

Starting from the device readings, we calculated for each sample the mean L\*a\*b\* parameters of a specific time moment, the immersion solution and the measurement device.  $\Delta E^*$  for a certain material, the same solution of immersion, the same measurement device and different moments in time were calculated according to the formula

$$\Delta E^* = \sqrt{(L_m - L_n)^2 + (a_m - a_n)^2 + (b_m - b_n)^2}$$

Where L, a and b are mean CIE L\*a\*b\* parameters for the time moments *m* and *n*.

Next we calculated the difference for the same material, solution of immersion and time interval between  $\Delta E^*$  calculated with the spectrophotometer UNICAM 4 UV-VIS and  $\Delta E^*$  calculated with the Easyshade. The difference of  $\Delta E^*$  for the same time interval, different measuring devices, was calculated according to the formula:

$$dif\Delta E_{water} = \Delta E_{water}^{uvvis} - \Delta E_{water}^{easyshade}, \text{ respectively } dif\Delta E_{wine} = \Delta E_{wine}^{uvvis} - \Delta E_{wine}^{easyshade}$$

The L\*a\*b\* and  $\Delta E^*$  values were studied separately for every material and immersion solution. The statistical test used for the values L\*a\*b\* and  $\Delta E^*$  was paired t-test.

The calculated values *dif* $\Delta E$  were grouped according to the immersion solution. If the values *dif* $\Delta E$  respect the null hypothesis no 3 they will measure a constant interval between the two  $\Delta E^*$  no matter of the measuring solution and of the material of the sample. Thus we grouped *dif* $\Delta E$  in two groups according to the solution of immersion and we applied a statistical test of independent type sample t test.

## 3. Results

### Null hypothesis 1

First we compared the L\*a\*b\* values measured by the two equipments. In Table 1 one can observe that 14 out of 18 statistical tests revealed significant differences between the values offered by UNICAM 4 UV-VIS and VITA Easyshade Advance. The maximum number of differences was seen at the Filtek material (6 out of 6) while the minimum number was seen at the experimental composite (3 out of 6).

Table 1. Paired sample *t* test results between  $L^*a^*b^*$  measured values with UVVIS equipment and  $L^*a^*b^*$  values measured with Easyshade dental spectrophotometer at different moments in time and different solutions

Tested parameters		$L^*$	$a^*$	$b^*$	$L^*$	$a^*$	$b^*$
Material	Solution	Paired samples correlations			Statistical test results significant for $p < 0.05$ (2-tailed)		
Experimental composite	Water	0.013	0.002	0.000	0.000	0.191	0.000
	Wine	0.004	0.147	0.615	0.179	0.063	0.000
Filtek	Water	0.190	0.071	0.254	0.000	0.000	0.000
	Wine	0.000	0.000	0.000	0.000	0.037	0.000
Valux	Water	0.002	0.325	0.000	0.000	0.000	0.000
	Wine	0.000	0.000	0.102	0.000	0.000	0.175

### Null hypothesis 2

We compared the values of  $\Delta E^*$  same material/same solution of immersion, calculated from the average parameters provided by the two measuring devices. In Table 2 one can observe that the devices determined similar values of  $\Delta E^*$  only for Valux and the experimental composite immersed in wine. As regards Filtek significant statistic differences of  $\Delta E^*$  were observed both for the samples immersed in water and for the samples immersed in wine.

Table 2. Paired sample *t* test results between  $\Delta E^*$  values calculated using measurements with the UVVIS equipment and  $\Delta E^*$  values calculated using measurements with the Easyshade dental spectrophotometer

Material	Solution	Paired samples correlations	$\Delta E^*$ , statistical test results significant for $p < 0.05$ (2-tailed)
Experimental composite	Water	0.019	0.000
	Wine	0.021	0.058
Filtek	Water	0.663	0.008
	Wine	0.000	0.002
Valux	Water	0.000	0.000
	Wine	0.000	0.281

### Null hypothesis 3

The normal distribution of the values  $dif\Delta E$  has been tested with the Kolmogorov-Smirnov test. The conclusion was that both groups have normal distribution with  $p = 0.349$  For water and  $p = 0.353$  for wine.

During the independent *t*-test sample we tested the variance equality between the two groups with the Levene's Test for Equality of Variances. Because the result of this test was rather significant ( $F = 15.266$ ,  $p = 0.000$ ) we decided to report the result for equal variances not assumed,  $t = -1.926$ ,  $p = 0.061$  (two-tailed).

Considering the fact that the result is not statistically significant, this means that the null hypothesis is

confirmed but the obtained value is very close to the critical value, we decided to perform a test for each material with the aim to identify possible measuring problems at some materials (Table 3).

Table 3. Test result for  $dif\Delta E$  for each individual material samples immersed in water versus samples immersed in wine

Material	Levene's Test for Equality of Variances	$dif\Delta E$ , statistical test results significant for $p < 0.05$ (2-tailed)
Experimental composite	$F = 6.250$ , $p = 0.020$	$t = 0.884$ , $p = 0.394$
Filtek	$F = 5.934$ , $p = 0.023$	$t = 3.030$ , $p = 0.010$
Valux	$F = 7.439$ , $p = 0.012$	$t = -0.219$ , $p = 0.830$

## 4. Discussions

The spectrophotometers can exactly determine the optical characteristics of some materials but the offered results depend on the constructive particularities of the devices and on the details of conception of the experiments. Our study aims to examine if two spectrophotometers of different construction will provide comparable results in determining the colour variation ( $\Delta E^*$ ) of some dental composites. We also introduced intentionally in our study two solutions of immersion to measure both the minimum variations (water) and the maximum ones (wine).

The quality of the VITA Easyshade tool for measuring the CIE  $L^*a^*b^*$  parameters of composite materials has been often demonstrated by using it in numerous studies in vitro and in vivo [4, 17, 27, 28]. The results with VITA Easyshade are not 100% precise, they indicate predictable shade values from repeated measurements with a reliability of 96.4% and an accuracy of 92.6% [12].

We compared the measured values of  $L^*a^*b^*$  and found out that there are statistically significant differences at over 75% of the measured sets of values (14 out of 18). We consider that this result indicates that the two devices

measure the colour parameters of the studied materials differently and thus the first null hypothesis is infirmed. It is very probable that this fact is caused by the technical measuring characteristics of the devices.

Considering that the first null hypothesis has been infirmed, it would have been desirable that at least  $\Delta E^*$  calculated on the basis of the  $L^*a^*b^*$  be similar between the two devices. We observed that again there are statistically significant differences for more than 60% of the  $\Delta E^*$  values (4 out of 6). Considering this fact, the second null hypothesis of our study is infirmed; too, the spectrometers used to perform measurements in our study will lead to different  $\Delta E^*$  values according to the device used.

The most interesting result is the one from the third null hypothesis, which examines if the two devices determine  $\Delta E^*$  differently and maintain a constant difference. In the first and second null hypothesis the solution of immersion and the time were constant values (paired t-test using the material, the solution of immersion and the time as pairing elements). By calculating the  $dif\Delta E$  we eliminated the measuring device and the material and thus we were able to compare if the differences between  $\Delta E^*$  for water and wine are similar. As we mentioned in the results, the statistical tests indicate that, the results of the global test were not significant, which means that, between the  $\Delta E^*$  values determined with the UVVIS and the  $\Delta E^*$  values determined with Easyshade similar differences will be preserved regardless of the solution of immersion. Being aware of this difference we were able to compare the values of  $\Delta E^*$  obtained from the studies performed with different devices and apply the necessary correction. In our study the results indicate insignificant differences between the two spectrophotometers, with regard to the hybrid composites and significant results only in the case of the nanocomposites. A possible explanation could be the way in which the structure of the composite material handles the light

Dental composites are basically composed of three chemically different materials: the organic matrix or the organic phase; the inorganic matrix, which is the filler or disperse phase; and an organosilane, which is a coupling agent which binds the filler to the organic resin. The current trend towards minimizing the filler size in order to achieve great optical qualities and towards maximizing the filler loading is an attempt to improve all of the requirements for dental composites. Contemporary composites may contain large quantities of sub-micron and even nano-particles. The filler particles, the size and number, are mainly responsible for spreading the light in composite materials. (29) In nanocomposites, nanofillers are added and distributed in a dispersed form or as clusters. (30, 31, 32) Dental nanocomposites have particle dimensions that are in the wave length spectrum of visible light, thus their capacity to absorb or spread the light is modified compared to the larger particles. In clinical applications this can be an advantage because the aesthetic aspect. The size and volume fraction of fillers in resin composites should be controlled for the best colour

reproduction, considering the refractive indices of filler and resin matrix [31, 32].

## 5. Conclusion

The current trend in dental medicine is to reduce the size of inorganic filling particles and to increase their number so as to improve the mechanical and aesthetic quality of the composite materials. The dental spectrophotometers can detect modifications triggered by immersion factors but there can occur problems in the presence of very small particles of inorganic filling.

## References

- [1] C. Gómez-Polo, M. Gómez-Polo, A. Celemin-Viñuela, J. A. Martínez Vázquez De Parga, J. Dent. **42**(6), 742 (2014).
- [2] S. M. Mayekar, Dent Clin North Am., **45**(1), 155 (2001).
- [3] H. Lu, I. Roeder, I. Lei, J. Power, J. Esthet. Restor. Dent., **17** (2), 102 (2005).
- [6] S. T. Fontes, M. R. Fernandez, C. M. De Mura, S. S. Meireles, J. Appl. Oral Sci., **17**(5), 388 (2009).
- [7] I. Nasim, P. Neelakantan, R. Sujeer, C. V. Subbarao, J. Dent., **38** (Suppl. 2)e137 (2010).
- [8] R. Janda, J-F. Roulet, M. Kaminsky, G. Steffin, M. Latta, Eur J Oral Sci., **112**(3), 280 (2004).
- [9] N. M. Ayad. Eur. J. Esthet. Dent., **2**(2), 236 (2007).
- [10] M. S. Festuccia, Lda F. Garcia, D. R. Cruvinel, Fde C. Pires-De-Souza, J Appl Oral Sci. **20**(2), 200 (2012).
- [11] W. D. Browning, D. C. Chan, J. S. Blalock, M. G. Brackett, Oper. Dent., **34**(3), 337 (2009).
- [12] H. O. Elamin, N. H. Abubakr, Y. E. Ibrahim, Eur. J. Dent. **9**(2), 213 (2015).
- [13] E. U. Çelik, A. Aladağ, L. Ş. Türkün, G. Yılmaz, J Esthet Restor Dent. **23**(3), 179 (2011).
- [14] S. Kim-Pusateri, J. D. Brewer, E. L. Davis, A. G. Wee, J Prosthet Dent. **101**(3), 193 (2009).
- [15] M. Ionaş, S. Oancea, M. M. Cernuşcă-Miţariu, Optoelectron. Adv. Mater. –Rapid Comm., **7**(11-12), 922 (2013).
- [16] A. Dozic, A. K. Kharbanda, H. Kamell, J. D. Brewer, J Dent Res **39** (Suppl.3), 23 (2011).
- [17] C. Llana, E. Lozano, J. Amengual, L. Forner, J. Contemp Dent Pract. **12**(1), 19 (2011).
- [18] A. Dozić, C. J. Kleverlaan, A. El-Zohairy, A. J. Feilzer, G. Khashayar, J Prosthodont., **16**(2), 93 (2007).
- [19] E. Sarkis, The Saudi Dent. J. **24** (2), 85 (2012).
- [20] Y. F. Ren, L. Feng, D. Serban, H. S. Malmstrom, J. Dent., **40** (Suppl 1), 48 (2012).
- [21] M. Moldovan, L. Silaghi Dumitrescu, C. Prejmerean, D. Dudea, V. Popescu, S. Boboia, D. Prodan I. Cojocar, Mat. Plast., **47**(4), 421 (2010).
- [22] H. Yu, X. Pan, Y. Lin, Q. Li, M. Hussain, Y. Wang, Oper Dent., **34**(1), 72 (2009).

- [23] M. Ionaş, M. E. Badea, L. Silaghi Dumitrescu, T. Ionaş, M. Moldovan, *Mat. Plast.* **51**(4), 435 (2014).
- [24] F. T. Topcu, G. Sahinkesen, K. Yamanel, U. Erdemir, E. A. Oktay, S. Ersahan, *Eur. J. Dent.*, **3**(1), 50(2009).
- [25] E. Ertaş, A. U. Güler, A. C. Yücel, H. Köprülü, E. Güler, *Dent Mater J.*, **25**(2), 371 (2006).
- [26] F. Falkensammer, G. V. Ametzt, A. Wildburger, J. Freudenthaler, *J.Prosthet. Dent.*, **109**(6), 378 (2013).
- [27] P. Gürdal, B. G. Akdeniz, B. Hakan Sen, *J Oral Rehabil.* **29**(9), 895 (2002).
- [28] C. Olms, J. M. Setz, *Clin Oral Investig.*, **17**(4), 1161 (2013).
- [29] E. U. Çelik, A. Aladağ, L. S. Turkun, G.Yilmaz, J. *Esthet. Restor. Dent.*, **23** (3), 179, 188 (2011).
- [30] Z. Ergücü, L. S. Türkün, A. Aladag, *Oper. Dent.*, **33** (4), 413 (2008).
- [31] Y. K. Lee, *Dent Mater.* **24**(9), 1243 (2008).
- [32] M. H. Chen, *J. Dent. Res.*, **89**(6), 549 (2010).
- [33] J. L. Ferracane, *Crit Rev Oral Biol Med*, **6**(4), 302 (1995).
- [34] S. B. Mitra, D. Wu, B. N. Holmes, *JADA*, **134**(10), 1382 (2003).

---

\*Corresponding author: tibiionas@yahoo.com