Comparative Study Regarding the Colorimetric Changes of Two Composite Resins after Immersion in Several Beverages and One Antibacterial Mouthwash

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*The aim of the study was to assess the colorimetric changes of two composite resins immersed in five solutions as follows: saliva (control), saliva and coffee, saliva and red wine, saliva and coca-cola, saliva and Corsodyl. The study group included 200 samples of composite resins, divided in two groups (Filtek Ultimate and Filtek Z250, 3M ESPE Dental Products, St. Paul, USA), each sample with 20mm diameter and 1,5mm thickness. The pH of immersion solutions was assessed. The samples were divided in five study groups, each sample was immersed in one of the immersion solutions, at 37° C. For each group, 10 specimens have been immersed for 24 h in the storage solution and the other ten specimens have been stored for seven days. The colorimetric measurements were performed, using Lovibond Reflectance Tintometer RT, Lovibond Brand UK. The colorimetric differences (ΔE) defined by system CIE L*a*b*, between the control group and the study group, were assessed for each immersion period. The test U Mann-Whitney was applied to compare the study groups with the control group, and test Kruskal Wallis was applied for ΔE values. The assessment of colorimetric changes (ΔE) have shown differences between the control group (saliva) and the study groups as follows: Control<Corsodyl<Coca Cola<coffee<red wine, for both composite resins (ΔE>1). The test U Mann-Whitney showed statistically significant differences (p = 0.0001) between the control group and the study groups for both composite resins. The test Kruskal Wallis also showed statistically significant differences for values ΔE (p< 0.05). The composite resins Filtek Ultimate and Filtek Z250 presented visible colorimetric changes after immersion in tested solutions. The most obvious changes were observed after immersion in Coca cola, coffee and red wine. Filtek Ultimate was more stable comparing with Filtek Z250 regarding colorimetric changes.*

Keywords: color stability, composite resins, acidic beverages, antibacterial mouthwash

The clinical success in operative dentistry with composite resins depends on the performance regarding shape, surface and color choice. The colorimetric stability of direct coronal restorations can be influenced by various intrinsic and extrinsic factors.

Internal color changes depend upon the composite photoinitiator system [1-3]. Intrinsic discoloration is permanent and is related to polymer quality, type, and quantity of inorganic filler and type of accelerator added to the photo initiator system [4, 5].

The extrinsic factors that influence colorimetric changes of esthetic restorations are determined by retention and adsorption of colored substances and depend on diet, type of beverages and hygiene. The extrinsic discolorations are also related with the chemical properties of composite resins and surface roughness of composite resin restoration.

A number of studies have shown the discoloring effect of various stains like, coffee, wine, fruit juices/soft drinks, and the effect of turmeric and tobacco (chewable form) [6].

The assessment and quantification of the color changes of dental materials requires the understanding of color space and differential colorimetry. Current photometric and colorimetric instruments provide reliably quantification of the color of acrylic resin specimens. [7]. Photometric and colorimetric instruments measure color and express it in terms of three coordinate values (L^*, a^*, b^*), which locate the object color within the CIELAB color space [8-15]. The L^* coordinate represents the brightness of an object, the a^* value represents the red or green chroma, and the b^* value represents the yellow or blue chroma. The color difference (ΔE) of two objects can then be determined by comparing the differences between respective coordinate values for each object. The formula used for calculating the color differences in this system is $\Delta E = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$ where L^*, a^*, and b^* are the differences of the color parameters between the two specimens measured for comparison. Numeric description of color permits precise definition of the magnitude of the colour difference between objects.

The aim of the study is to assess the colorimetric stability of two composite resins (Filtek Ultimate and Filtek Z250) after exposure to the action of three popular beverages and one antiseptic mouthwash with potential to produce colorimetric changes.

Experimental part

Materials and Methods

The study groups included 100 samples of two composite resins (Filtek Ultimate and Filtek Z250 3M ESPE Dental Products, St.Paul, USA), shade A_2 enamel, divided in two groups (table 1). The dimensions of each sample were 20 mm diameter and 1.5 mm thickness. For each

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Product	Category	Composition	Manufacturer
Filtek Ultimate	Nanohybrid composite	Bis-GMA, UDMA,TEGDMA,PEGDMA, BIS-EMA, zirconium/silica cluster filler (20nm silica filler and 4 to 11 nm zirconium filler) 55.6% by volume	3M ESPE Dental Products, St.Paul, USA
Filtek Z250	Universal Microhybrid Composite	Bis-GMA, Bis-EMA, UDMA (0,01-3.5 μm zirconium/silica filler) 60% by volume	3M ESPE Dental Products, St.Paul, USA

Table 1
TESTED COMPOSITE MATERIALS

Solutions	pH
Red wine	3.66
Coca-cola	2.70
Corsodyl	4.94
Coffee (Nescafe original, 3g at 100ml water)	4.80

Table 2
TESTED SOLUTIONS AND THEIR *p*H

Solutions	Ingredients	pH
Solution 1 (control)	Saliva; 990 ml (NaCl 0.70g/l, KCl 1.2g/l; $Na_2HPO_4H_2O$ 0.26g/l; $NaHCO_3$ 1.5g/l; KSCN 0.33g/l; uree 1.35g/l	7.73
Solution 2	Saliva 660 ml + Coca-Cola (330 ml)	5.45
Solution 3	Saliva 660 ml + Corsodyl (330ml)	7.71
Solution 4	Saliva 660 ml + Coffee (330 ml)	6.69
Solution 5	Saliva 660 ml + Red wine (330 ml)	4.31

Table 3
COMPOSITION OF THE TESTED SOLUTIONS AND THEIR *p*H

sample, material was introduced between two glass plates covered with celluloid matrixes for a flat surface. For each sample a 40 s photopolymerisation was performed using a photocuring lamp LEDidition (Ivoclar Vivadent clinical, Austria). Each sample was polymerized on each surface for a homogeneously light polymerization. The samples were immersed in distilled water at 37° C for 24 h, then were introduced in the test solutions.

The products used in this study were AFNOR saliva [16], coffee Nescafe Brasero (Nestle, Spain), dry red wine (13.5% alcohol) Trockener Rotwein Schwaben Wein-Merlot/Cabernet Sauvignon (Cramele Recas SA Romania), Coca-Cola (Coca-Cola HBC Romania SRL), mouthwash Corsodyl (Glaxo SmithKline Consumer Healthcare, UK). The initial *p*H was measured for each product, using pH-meter Hanna Instruments pH210 (SUA). The calibration was performed in three points at *p*H 4.01; 7.01 and 10.01 at 25° C (table 2).

The study solutions were prepared and their *p*H was measured using the same pH-meter (table 3).

Ten samples of materials were immersed in the study solutions at 37° C, in darkness for 24 h i.e. 7 days. The solutions were not changed during test period. The colour measurement was performed using a colorimeter Lovibond Reflectance Tintometer RT, (Lovibond Brand UK) at 24 h and 7 days. Before each measurement, the samples were washed with distilled water and dried with an air-spray.

The colorimetric differences (ΔE) between control group and study groups were defined using system CIE L*a*b*, using non-parametric statistical test U Mann-Whitney for variables L, a, b. This test is the non-parametric equivalent of test *t* for independent specimens.

The null hypothesis considers that between the control group and study group there are no statistically significant differences. The research hypothesis considers that between the control group and study group there are statistically significant differences.

Results and discussions

The tables 4 and 5 present the mean values of parameters L*a*b*, (ΔE), and standard deviation after one day and 7 days for each study group.

In our study, the values ΔE were higher than 1, value considered perceivable by human eye.

The colorimetric analysis found color changes (ΔE), as follows: saliva (control)< Corsodyl<cola<coffee<red wine, for both composite resins after 24 h and 7 days.

For composite resin Filtek Ultimate the values ΔE were very high, after 24 h and 7 days, for solution 5 (saliva-red wine) 22.34/22.04 and for solution 4 (saliva-cofee) 12.24/ 14.00. The values ΔE for solution 3 (saliva-Corsodyl) and solution 2 (saliva-Coca-cola) were lower but still perceptible.

For composite resin Filtek Z250 the values ΔE were also very high, after 24 h and 7 days, for solution 5 (saliva-red wine) 16.82/20.37 and for solution 4 7.99/12.25. For samples of Filtek Z250, immersed in solutions 2 and 3, the values ΔE were lower, comparing with solutions 4 and 5, but higher comparing with solution 1.

The test *U* Mann Whitney find statistically significant differences between study groups and control group, for parameters L, a, b (**p** = 0.0001 < 0.05), both for measurements performed after 24 h and 7 days.

The test Kruskal Wallis applied ΔE also found statistically significant changes (p = 0.0001).

Table 4
COLOUR DEVIATION FOR THE COMPOSITES AFTER 24 h OF IMMERSION IN SOLUTIONS

Composite/ Solution	Average value	Standard deviation	Composite/ Solution	Average value	Standard deviation
Filtek Ultimate - solution 1			Filtek Z250- solution 1		
L*	79.87	.07706	L*	77.39	.57857
a*	0.78	.02908	a*	0.22	.08386
b*	12.90	.05744	b*	16.79	.27700
Filtek Ultimate- solution 2			Filtek Z250- solution 2		
L*	77.88	.16560	L*	76.12	.04290
a*	0.99	.01947	a*	1.00	.02003
b*	17.19	.06717	b*	18.55	.02514
ΔE*	4.73		ΔE*	2.31	
Filtek Ultimate- solution 3			Filtek Z250- solution 3		
L*	81.65	.06443	L*	78.86	.39434
a*	0.85	.01751	a*	0.43	.03134
b*	11.35	.02406	b*	16.85	.11662
ΔE*	2.36		ΔE*	1.49	
Filtek Ultimate- solution 4			Filtek Z250- solution 4		
L*	72.39	.16387	L*	70.40	.11641
a*	3.04	.02601	a*	2.72	.02098
b*	22.32	.19312	b*	19.75	.08138
ΔE*	12.24		ΔE*	7.99	
Filtek Ultimate- solution 5			Filtek Z250- solution 5		
L*	57.85	.03401	L*	61.04	.09343
a*	-2.31	.04050	a*	-0.92	.02150
b*	10.71	.44410	b*	13.03	.04968
ΔE*	22.34		ΔE*	16.82	

Table 5
COLOUR DEVIATION FOR THE COMPOSITES AFTER 7 DAYS OF IMMERSION IN SOLUTIONS

Composite/ Solution	Average value	Standard deviation	Composite/ Solution	Average value	Standard deviation
Filtek Ultimate - solution 1			Filtek Z250- solution 1		
L*	79.59	.04392	L*	80.74	.13969
a*	0.46	.01135	a*	0.24	.01912
b*	15.05	.01370	b*	16.79	.06408
Filtek Ultimate- solution 2			Filtek Z250- solution 2		
L*	74.13	.03866	L*	74.36	.02486
a*	2.15	.01853	a*	1.66	.02406
b*	17.71	.11542	b*	18.98	.02214
ΔE*	6.30		ΔE*	7.62	
Filtek Ultimate- solution 3			Filtek Z250- solution 3		
L*	76.9	.12563	L*	77.72	.04111
a*	1.1	.03864	a*	0.93	.02633
b*	14.66	.10023	b*	17.39	.12931
ΔE*	2.79		ΔE*	3.88	
Filtek Ultimate- solution 4			Filtek Z250- solution 4		
L*	66.78	.05739	L*	70.29	.08647
a*	4.63	.01633	a*	3.15	.04725
b*	18.85	.04581	b*	20.76	.11633
ΔE*	14.00		ΔE*	12.25	
Filtek Ultimate- solution 5			Filtek Z250- solution 5		
L*	58.11	.18786	L*	60.53	.10651
a*	2.73	.04742	a*	2.73	.02000
b*	19.43	.49840	b*	15.72	.21844
ΔE*	22.04		ΔE*	20.37	

SD- standard deviation

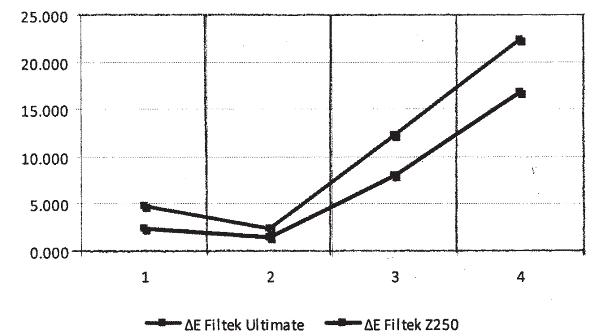

Fig. 1. Colorimetric changes for composite resins immersed in study solutions after 24 h

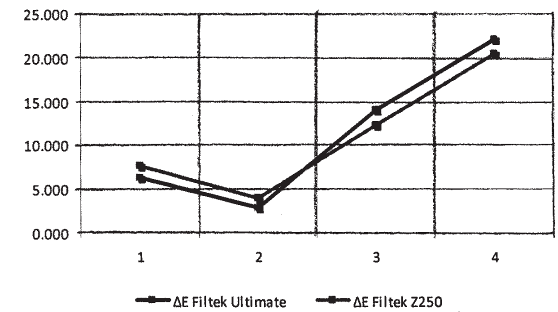

Fig.2. Colorimetric changes for composite resins immersed in study solutions after 7 days

The value chi-square for test Kruskal Wallis applied ΔE on study groups was statistically significant (p = 0,0001), comparing with control group.

The test Mann-Whitney was applied in order to estimate an accurate difference between study groups (table 7).

For both composites, the differences were statistically significant between the tested solutions, according to Mann-Whitney test ($p < 0.05$).

The growing demand for esthetics leads to premature replacement of restorations, as one of the main causes of composite restoration replacement in anterior teeth is material discoloration [17-21].

Discoloration can be caused by intrinsic or extrinsic factors. Extrinsic factors are directly related to the patient diet [22-24]. The impact of a beverage on composite properties can be related to quantity and frequency of ingestion.

In this study, we have tested three of the most frequently consumed beverages (coffee, red wine and cola) and one of the most common mouthwash used for oral hygiene (Corsodyl).

Ertas and colleagues related that a 24-h storage time simulates about 1 month of coffee consumption [25, 5]. The authors simulated the colouring of composite restorations within 7 months of coffee consume.

In vivo, composite resins can be intermittently or continuously exposed to chemical agents found in saliva, food, and beverages. The chemicals of the oral environment could have an appreciable influence on the degradation of composite restoratives [26].

Significant color shifts occurred in each test group in each staining solution over time. Moreover, the color shift in different test groups was significantly different when the magnitudes of color differences within the test groups were compared after 24 h and after 7 day (p <0.05).

The colorimetric analyze have shown color changes (ΔE), which increased in the following order: saliva (control) > Corsodyl > cola > coffee > red wine, for both composites Filtek Ultimate 2.36/2.79 > 4.73/6.30 > 12.24/ 14.00 > 22.34/22.04 and Filtek Z250 1.49/3.88 > 2.31/7.62 > 7.99/12.25 > 16.82/20.37 (one day/seven days).

	ΔE* Filtek Ultimate 24h	ΔE* Filtek Ultimate 7 days	ΔE* Filetk Z250 24 h	ΔE* Filetk Z250 7 days
Chi-Square	36.592	36.599	35.387	36.596
df	3	3	3	3
Asymp. Sig.	**.000**	**.000**	**.000**	**.000**

a.Kruskal Wallis Test b. Grouping Variable: comp

Table 6
KRUSKAL WALIS TEST ΔE* FOR THE STUDIED SOLUTION. TEST STATISTICS[a,b]

Test Statistics[b]	ΔE* Filtek Ultimate 24 hours	ΔE* Filtek Ultimate 7 days	ΔE* Filtek Z250 24 hours	ΔE* Filtek Z250 7 days
Cola-Corsodyl				
Z	-3.782	-3.782	-3.099	-3.780
Asymp. Sig. (2-tailed)	**.000**	**.000**	**.002**	**.000**
Exact Sig. [2*(1-tailed Sig.)]	.000[a]	.000[a]	.001[a]	.000[a]
Cola- Coffee				
Z	-3.782	-3.784	-3.780	-3.781
Asymp. Sig. (2-tailed)	**.000**	**.000**	**.000**	**.000**
Exact Sig. [2*(1-tailed Sig.)]	.000[a]	.000[a]	.000[a]	.000[a]
Cola- Red Wine				
Z	-3.782	-3.781	-3.780	-3.782
Asymp. Sig. (2-tailed)	**.000**	**.000**	**.000**	**.000**
Exact Sig. [2*(1-tailed Sig.)]	.000[a]	.000[a]	.000[a]	.000[a]
Corsodyl –Red Wine				
Z	-3.780	-3.781	-3.780	-3.782
Asymp. Sig. (2-tailed)	**.000**	**.000**	**.000**	**.000**
Exact Sig. [2*(1-tailed Sig.)]	.000[a]	.000[a]	.000[a]	.000[a]
Coffee- Red Wine				
Z	-3.780	-3.782	-3.780	-3.784
Asymp. Sig. (2-tailed)	**.000**	**.000**	**.000**	**.000**
Exact Sig. [2*(1-tailed Sig.)]	.000[a]	.000[a]	.000[a]	.000[a]

a. Not corrected for ties. b. Grouping Variable: comp

Table 7
MANN-WHITNEY STATISTIC TEST RESULTS

Test Mann-Whitney applied in order to estimate differences between parameters L, a, b of control and study group composite resin samples, showed statistically significant changes (p=0.0001) for all cases.

The colorimetric changes are more obvious for samples immersed in solutions saliva-coffee and saliva-red wine comparing with solutions saliva-Coca-Cola and saliva-Corsodyl, for both study periods.

The values ΔE for Filtek Ultimate in the solutions coffee and red wine were higher, comparing with Filtek Z250.

The denture base acrylic resins have been reported to be critical in color stability, which is affected by different alcohol concentrations and *p*H solutions. Several studies have reported that alcohol facilitates staining by softening the resin matrix [22]. Therefore, it could be possible that the alcohol component in wine (10% alcohol by volume) roughened the denture base resin surfaces, thereby resulting in increased staining. Since the *p*H of red wine is about 3.7, it is possible that the acidic *p*H may have had an effect on the structure of the materials [27].

In our investigation, the color differences for Filtek Z250 were higher after 7 days of storage in red wine compared with the first immersion period. It seems that time influences the color change, probably by the cumulative effect of surface degradation promoted by alcohol (13.5%) and *p*H (3.66) and the subsequent adsorption of natural and artificial red wine colorants.

The coloring degree of samples with coffee is lower comparing with red wine. The tannic acid has been proved especially responsible for the staining of this beverage [28].

Filtek Ultimate contains Bis-GMA, UDMA, TEGDMA, PEGDMA, BIS-EMA, zirconium/silica cluster filler (20 nm silica filler and 4 to 11 nm zirconium filler) 55.6% by volume. Filtek Z250 contains Bis-GMA, Bis-EMA, UDMA (0.01-3.5μm zirconium/silica filler) 60% by volume. This different composition between the two composite resins explains the higher values ΔE for Filtek Ultimate in coffee and red wine comparing with Filtek Z250.

Similar results have been reported by Villalta and colleagues and Yazici and colleagues. Villalta and colleagues investigated the effects of coffee or red wine staining solutions on Filtek Supreme (nanohybrid resin based composite) (ΔE* ≈ 12.0) and Esthet X (microhybrid resin based composite) (ΔE* ≈ 3.9) [29]. Following storage in coffee or red wine solutions, the nanohybrid composite changed colour more than the microhybrid material. Yazici and colleagues observed the effects of two staining solutions (tea or coffee) on the colour stability of a hybrid RBC (ΔE* ≈ 4.4) and a nanohybrid RBC (ΔE* ≈ 8.0) after different immersion periods [30].

Results showed hybrid resin based materials to be more color stable than the nanohybrid materials to coffee and tea stains and that stainability is resin-material dependent.

The observed colour change (ΔE*) could be different and less obvious in vivo. A longer staining period may be required in vivo to cause a clinically perceptible colour change. This can be attributed to the cleansing action of saliva in the oral activity, and the daily rinsing and brushing action for oral hygiene maintenance [31, 32].

M.T. Radu et al. [33] found that the erosion of composite resins surface (Filtek Z250 and Charisma) is related to acidity level of the immersing solution. In the case of Coca Cola, *p*H= 2.6. Such a low acidity is given by the phosphoric acid, added to make the drink tastier, and also to the carbon dioxide, which is transformed into carbonic acid. The *p*H of the soft drink Nestea is 3.6. Composition of the Corsodyl solution for oral rinsing contains 96% ethanol, castor oil, sorbytol, mint oil, purified water, the *p*H recording a value of 5.6. For both types of composite resins under analysis, the most aggressive medium was that of Coca Cola, followed, in decreasing order, by Nestea Lemon, Listerine and Corsodyl.

Food or beverages (such as soft or alcoholic drinks or some other food derivatives) may affect the behaviour of the restoration materials [34, 35].

Several soft drinks have a *p*H = 3 or even lower, which means that intakes for long periods may cause erosion of both enamel and composite resins. Some previous studies have demonstrated that the microfiller particles tend to be eliminated from the resin, while the components of the organic matrix get decomposed when occurring in a medium with a low *p*H (4,5) [36-38].

The surfaces changes can be considered as a degradation and erosion process of polymeric matrix. The acidity mechanism at the level of the composite resin may be explained by the hydrolysis of the ester radicals present at the level of dimethylacrylate monomers (Bis-GMA, Bis-EMA, UDMA, TEGDMA)

An extremely important part is also played by the coupling agent that, to some extent, protects the filler against degradation through hydrolysis [39].

Another possible cause of the degradation of composites surface is due to the fact that the fillers and the matrix are too weakly linked, which may be correlated with an insufficient treatment of the filler surface with silan, leading to filler erosion [40, 41].

Conclusions

The effects of solutions with coloring potential were perceptible for human eyes($\Delta E>1$).

The colorimetric variations for all study solutions, for both composite resins (Filtek Ultimate si Filtek Z250) were statistically significant.

The colorimetric changes are more obvious for samples immersed in solutions saliva-coffee and saliva-red wine comparing with solutions saliva-Coca-Cola and saliva-Corsodyl, for both study periods.

Regarding colorimetric stability, the composite resin Filtek Ultimate seems to be more stable comparing with Filtek Z250.

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