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THE EFFECT OF A NATURAL EXTRACT-BASED EXPERIMENTAL BLEACHING GEL UPON THE COLOUR AND SURFACE ROUGHNESS OF A COMPOSITE RESIN - AN IN VITRO STUDY

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ABSTRACT. *Objective:* To evaluate the bleaching effect and the changing in the surface roughness of one experimental natural-based bleaching gel and of three commercial bleaching gels, namely: Opalescence PF 16%, Ultradent, Opalescence Quick PF 45%, Ultradent (based on carbamide peroxide CP) and Philips Zoom DayWhite 6% HP light activated (based on hydrogen peroxide HP) upon A3 body shade Filtek Supreme, 3M Espe nanocomposite. *Material and methods*: Thirty disks of the A3 body shade Filtek Supreme, 3M Espe, were immersed in a coffee solution and further divided into 6 groups: group 1 was kept as control, while groups 2-6 underwent a certain bleaching protocol, with different materials and exposure time, as follows: group 2: experimental gel, five sessions, 6 hours each; group 3: Opalescence PF 16 % (Ultradent), five sessions 6 hours each; group 4: Opalescence Quick PF 45% (Ultradent), 2 sessions, 30 min each; group 5: Opalescence Quick PF 45% - 2 applications/30 min and Opalescence PF 16% - 5 applications/6h and group 6 was bleached using an in office gel containing HP 6% (Philips Zoom DayWhite 6% HP light-activated 2 applications/30min).

CIE L*a*b* parameters and surface roughness were recorded before and after bleaching. Data were analyzed using one-way repeated measures ANOVA, and multiple comparisons were adjusted using the Bonferroni method ($p<0.05$). Results: The average colour differences at the end of the bleaching protocols were as follows: 1:$\Delta E^*=1.40$, 2:$\Delta E^*=3.76$, 3:$\Delta E^*=5.13$, 4:$\Delta E^*=4.79$, 5:$\Delta E^*=5.44$, and 6:$\Delta E^*=6.83$. Overall, a significant statistical difference was found between the groups ($p<0.05$). However, multiple comparisons showed

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no significant difference among the groups based on carbamide peroxide (CP) (3, 4, and 5) ($p>0.05$). The results of the statistical test indicated a significant effect of the bleaching gels only for groups 5 and 6 ($p<0.05$). For these groups, surface roughness significantly decreased after the bleaching protocol. For the experimental group, no significant modifications of the surface were observed. Conclusions: The most effective protocol was the "in office" bleaching method based on hydrogen peroxide (HP) with light activation. The natural extract-based gel was less efficient than CP and HP groups; however, ΔE* was above the acceptability threshold. The natural extract-based experimental bleaching gel showed no significant change upon surface roughness.

Keywords: *nanocomposite, colour stability, bleaching, surface roughness, natural extract*

INTRODUCTION

Photo activated resin composites remain even today the dental restorative materials most frequently used in the treatment of both anterior and posterior teeth. Nowadays, patients seek better colour matching restorations and composite resins to satisfy their needs [1].

Discolouration is a significant aesthetic problem for direct tooth-coloured restorations. However, restoration longevity and the aesthetic appearance greatly depend on the quality of the finishing and polishing techniques employed [2-3]. An unacceptable colour match is a major reason for composite restoration replacement. Intrinsic factors - due to changes in the filler, matrix, or silane coating - or extrinsic factors - such as stain absorption - may cause the discolouration of the aesthetic materials [2]. Experimental studies have shown that the colouring effect also depends on the staining solutions used [4].

Tooth bleaching has gained considerable acceptance among dentists and patients, as it is a simple, effective and safe procedure to lighten discoloured teeth. Since its introduction by Haywood and Heymann in 1989 [5] tooth whitening has become one of the most popular aesthetic procedures offered by dentists.

There are several types of bleaching methods, all of them based on the principle of peroxide degradation into hydrogen peroxide (HP) or its compounds - carbamide peroxide (CP), unstable free radicals, which are further decomposed into large pigmented molecules - either through oxidation or through a reduction reaction [6]. The bleaching methods are: the so called "in office bleaching" based on 16%, 35%, 40%, 45% of either HP or CP, for 30 to 45 minutes, with or without light or laser activation, or the "at home bleaching", under the supervision of the dentist, with lower concentrations of HP or CP, applied with strips or custom trays.

Considerable research has been carried out in order to find out the effects bleaching has on tooth surface and on the dental restorative materials. In as far as the dental surface is concerned, the effects included alterations in the morphology, as well as in the chemical and physical properties [7]. Scanning electron microscope findings have shown microscopic changes in the tooth structure, namely: an increased porosity, depression and surface irregularities [8], an increased surface roughness (SR) [9], a decrease in hardness [10] as well as in fracture resistance [7]. SR suffered alterations during or after treatment, depending on the HP concentration [11]. Researchers believe that SR leads to an increased susceptibility to bacterial adhesion and staining [10,12,13]. After bleaching, pigments adhere to the rough surface, especially to that of the enamel, far easier than to the original tooth surface, which causes more discolouration [14,15].

Many studies have examined the changes induced by bleaching in the characteristics of composite resins, such as colour, surface hardness and roughness, staining susceptibility, microleakage and elution [16]. Using a spectrophotometer, Li et al. [17] found significant changes in the colour of nanohybrid and packable composite resins after bleaching with 15% CP. Another study [18] showed that this difference was especially noticeable when a high peroxide concentration (35% HP) was used on low-density resins, such as microfilled composite resins. The authors associated these results with the resin matrix volume and the filler type. Both studies reported that the colour change was clinically acceptable. A recent study by Wattanapayungkul et al. [19] demonstrated that the treatment of composite resins with a low peroxide concentration significantly increased surface roughness.

A few studies assessed the influence of bleaching gels upon glass ionomer cements. A recent *in situ* study by Li et al. [16] showed a significant difference in the colour of a conventional glass ionomer cement restoration after four weeks of bleaching with 15% CP, when compared to that noted before the treatment. However, two weeks after the completion of the whitening treatment, the colour returned to that recorded before the treatment, which means that the bleaching did not alter the colour of the glass ionomer cement.

Moreover, scanning electron microscopy (SEM) revealed a slight surface dissolution. Similarly, another study [20] found alterations, such as cracks and pits, in the surface of the glass ionomer cement, which were explained by the capacity of the bleaching agent to alter the surface properties of the material. The authors also found that bleached glass ionomer restorations were more susceptible to different staining solutions with a pH from 3.73 to 6.25, such as red wine, herbal tea, Coca-Cola and coffee [20].

These conflicting reports encourage further investigation into the effects bleaching agents have on composite resin colour and SR.

Our study aims to analyze: a). the bleaching effect of an experimental bleaching gel based on natural extracts in comparison with that of commercial bleaching agents based on CP or HP used in different protocols (simulating "at home" and "in office" applications); and b). the effect of the same bleaching agents on the surface roughness of the A3 body shade Filtek Supreme, 3M Espe nanocomposite.

The null hypothesis is that both surface roughness and the colour of the A3 body shade Filtek Supreme, 3M Espe nanocomposite do not change after the application of the bleaching gels.

RESULTS AND DISCUSSION

CIE L*a*b* parameters were recorded as follows: at the beginning of the bleaching protocol (Baseline), at the end of the bleaching protocol (Final), and at intermediate phases, after each bleaching session, depending on the protocol used. Colour difference ($\Delta E_{ab}{}^{*\,*}$) was calculated using the equation [21]:

$$\Delta E_{ab}{}^{*} = [\,(\Delta L^{*})^{2} + (\Delta a^{*})^{2} + (\Delta b^{*})^{2}\,]^{1/2}$$

where $\Delta L^{*}=L_2^{*}- L_1^{*}$, $\Delta a^{*}=a_2^{*}- a_1^{*}$, $\Delta b^{*}=b_2^{*}- b_1^{*}$; L_1^{*}, a_1^{*}, b_1^{*} correspond to the colour parameters before bleaching and L_2^{*}, a_2^{*}, b_2^{*} are the colour parameters of the specimens at the end of each bleaching session, depending on the protocol used.

At the end of the bleaching protocol, colour variation - expressed as colour difference (Final $\Delta E_{ab}{}^{*}$) calculated for the commercial and the experimental bleaching materials used in the different protocols - was, as follows: Group 1 : $\Delta E_{ab}{}^{*}=1.40$, Group 2: $\Delta E_{ab}{}^{*}=3.76$, Group 3: $\Delta E_{ab}{}^{*}=5.13$, Group 4: $\Delta E_{ab}{}^{*}=4.76$, Group 5: $\Delta E_{ab}{}^{*}=5.44$, and Group 6: $\Delta E_{ab}{}^{*}=6.83$, as shown in Table 1. Overall, a significant statistical difference was found between the groups ($p<0.05$). However, multiple comparisons showed no significant difference between the groups based on CP (3, 4, and 5) ($p>0.05$).

Univariate ANOVA was conducted to test the null hypothesis that surface roughness of composite resins does not change after the application of the bleaching gels for none of the tested products. The results of the statistical test indicated a significant effect of the bleaching gels only for groups 5 and 6 ($p<0.05$), which provided enough evidence to reject the null hypothesis. For these groups, surface roughness significantly decreased after the bleaching protocol, while no significant difference was observed in the experimental group.

Choosing the bleaching agent can be a challenge, since best whitening results, with lack of side-effects are desired.

In the case of restorative materials, the aim is to have a bleaching result that is similar to the one gained for the teeth, as well as to prevent any changes in the surface texture and composition following the bleaching.

Our study has focused on both effects on dental composites – namely: colour change and the preservation of the initial structure. We have used the A3 body shade Filtek Supreme, 3M Espe, which is a light activated composite, for both anterior and posterior teeth.

Table 1. ΔE^*_{ab} values calculated for each group at different intervals
(N/A- not applicable- where the protocols involved a limited number of measurements)

Groups	ΔE_{ab}* 1	ΔE_{ab}* 2	ΔE_{ab}* 3	ΔE_{ab}* 4	Final ΔE_{ab}*
1. Control	0.95	1.13	1.07	1.35	1.40
2. Experimental	1.44	3.04	3.59	3.80	3.76
3. Opalescence 16%	4.17	4.48	4.90	4.90	5.13
4. Opalescence 45%	3.79	N/A	N/A	N/A	4.76
5. Opalescence 45%+16%	4.46	4.77	5.15	5.27	5.44
6. Zoom	6.41	N/A	N/A	N/A	6.83

Table 2. Mean surface roughness values and standard deviations recorded for each group

Time	Group	Mean surface roughness (Ra)	Std. Deviation (+/-)
Before bleaching	Control	0.452	0.063
	Experimental	0.412	0.054
	Opalescence 16%	0.406	0.034
	Opalescence 45%	0.372	0.028
	Opalescence 16%+45%	0.429	0.041
	HP 35% light activated (Zoom)	0.417	0.024
After bleaching	Control	0.430	0.033
	Experimental	0.407	0.029
	Opalescence 16%	0.413	0.026
	Opalescence 45%	0.384	0.024
	Opalescence 16%+45%	0.397	0.045
	HP 35% light activated (Zoom)	0.371	0.032

In as far as discolouration is concerned, the null hypothesis was rejected, since different degrees of colour variation were obtained following the bleaching protocols: five times bleaching for 6 hours, with colour measurements between the various bleaching sessions, for groups 1, 2, 3, and 5; and two times bleaching for 30 minutes for groups 4 and 6 with colour measurements between the various bleaching sessions.

Bleaching, either “at home” or “in-office”, is one of the most popular treatments, appealing to both patients and dentists, as it is considered to be a simple and effective way of lightening discoloured teeth. Bleaching agents may result in a colour change of a restoration different from the one gained for the teeth, a situation that may not be accepted by the patient. If a restorative composite had a perfect colour match before the bleaching treatment, this may no longer be the case after bleaching.

Within the limits of this study, it has been noticed that even a low concentration of bleaching agent had an effect on the colour of the restorative materials.

The results of our study showed that a significant colour difference was obtained for group five, that was bleached with Opalescence 45%+16%, and group six, bleached with HP 35% light activated (Zoom), whereas the colour of the composite resin was least affected when the experimental natural-based bleaching gel was used.

In our study, colour differences were calculated using the CIE L*a*b* colour space. CIE (Commission Internationale de L’ Eclairage) L*a*b* colour parameters are used to quantify the optical properties of natural dental structures and dental materials [21]. In order to quantify the differences between two coloured samples, colour difference thresholds have been introduced in dentistry. Paravina et al. conducted a multicenter study upon visual thresholds in dentistry; they have reported that values smaller than $\Delta E_{ab}^* = 1.2$ are not perceptible, while values greater than $\Delta E_{ab}^* = 2.7$ [22] are considered to be clinically unacceptable. In our study, at the end of the bleaching protocol, all test groups showed colour difference values above the acceptability threshold, thus suggesting that the bleaching effect was significant and visually noticeable.

The second effect we observed was that of the modifications in surface roughness.

According to Paravina et al., the apparent colour difference is related to the surfaces, which are a result of the polishing techniques of the composites [23]. Polishing the composite up to Grit-1200 greatly helps to stimulate the clinical circumstances.

A large number of previous studies have demonstrated that bleaching agents affect the restorative materials by modifying hardness, roughness, colour and surface morphology [24]. The active ingredients of the most commonly used bleaching materials in different bleaching methods are carbamide peroxide and hydrogen peroxide. Carbamide peroxide degrades into approximately one-third of hydrogen peroxide and two-thirds of urea [25]. The free radicals that are formed eventually combine to form molecular oxygen and water. Some aspects of this chemical process might accelerate the hydrolytic degradation of the restorative materials, as described by Soderholm et al. [26]. Chemical softening of the restorative materials might also occur if the bleaching products have a high concentration of hydrogen peroxide [27].

It is known that nanoresin composites followed by microhybrid and hybrid composites showed least surface roughness (Ra) and colour change when exposed to different staining solutions [28]. Roman et al. [29] reported that unpolished composites presented higher Ra values than those that were polished.

The results of our study indicated a significant effect of the bleaching gels only in the case of groups 5 and 6 ($p<0.05$). For these groups, surface roughness significantly decreased after the bleaching protocol. No significant difference was found in the experimental group.

CONCLUSIONS

1. Both the commercial and the natural-based extract bleaching gels induced noticeable colour changes upon the tested composite resin; however, the most important colour change was noticed in the case of the HP 35% light activated Zoom bleaching gel.
2. The natural extract based gel was less efficient than the CP and HP groups; however, ΔE^* was above the acceptability threshold.
3. The surface roughness of the tested composite resin was significantly altered only when high concentrations of CP or HP were used.
4. The natural extract-based experimental bleaching gel showed no significant changes in the surface roughness.

EXPERIMENTAL SECTION

1. Disk preparation, staining and bleaching protocols

Thirty nanocomposite resin disks (n=30) were fabricated (A3 body shade Filtek Supreme, 3M Espe). The composition of the resin matrices and fillers of this composite resin is listed in Table 3.

The specimens were polished to a uniform thickness using abrasive sandpaper (2mm thickness, 10mm diameter) and were eventually immersed into a coffee solution (5g/100ml, Jacobs Kronung, Kraft Foods, Germany) for 8 hours/day on three consecutive days.

During the staining process, the specimens were stored in distilled water at 37°C. The specimens were further divided into 6 groups: one control group, and 5 test groups.

Group 2 underwent a bleaching protocol using an experimental gel based on natural extracts (5 applications/6h).

For test groups 3 and 4 two commercial gels were used, one for “at home” use (Opalescence CP16%, Ultradent – 5 applications/6h) and one for “in office” use respectively (Opalescence Quick CP45%, Ultradent – 2 applications/ 30 min).

Group 5 underwent a combined bleaching protocol (CP45% - 2 applications/ 30 min and CP16% - 5 applications/6h).

Group 6 was bleached using an “in office” gel containing HP 6% (Philips Zoom DayWhite 6% HP light-activated 2 applications/30min.

Table 3. Composition of the resin matrices and fillers of the composite resin

Material	Shade	Description	Composition	The resin system
Filtek™ Ultimate 3M™ ESPE™	A3B (Body)	Universal restorative material	The filler is a combination of a non-agglomerated/non-aggregated, 20nm nanosilica filler, and loosely bound agglomerated zirconia/silica nanocluster, consisting of agglomerates of primary zirconia/silica particles with size of 5-20 nm fillers. The cluster particle size range is 0.6 to 1.4 microns. The filler loading is 78.5% by weight.	BIS-GMA, BIS-EMA (6), UDMA with small amounts of TEGDMA.

2. Colour evaluation

Measurements were performed using a dental spectrophotometer (VITA Easyshade® Advance 4.0., VITA, Bad Sackingen, Germany) according to the CIE L*a*b* colour system. L* refers to lightness; its value ranges from 0 (black) to 100 (white), while a* and b* are measures of greens or redness and respectively blueness or yellowness of an object. The a* and b* chromaticity coordinates reach 0 for neutral colours and increase in magnitude for saturated or intense colours [30]. Five measurements were taken for each sample at a time.

CIE L*a*b* parameters were recorded as follows: at the beginning of the bleaching protocol (Baseline), at the end of the bleaching protocol (Final), and at intermediate phases, after each bleaching session, depending on the protocol used. Colour difference ($\Delta E_{ab}^{*\,*}$) was calculated using the equation [21]:

$$\Delta E_{ab}^* = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$$

where $\Delta L^*=L_2^* - L_1^*$, $\Delta a^*=a_2^* - a_1^*$, $\Delta b^*=b_2^* - b_1^*$; L_1^*, a_1^*, b_1^* correspond to the colour parameters before bleaching and L_2^*, a_2^*, b_2^* are the colour parameters of the specimens at different intervals of the bleaching protocol.

In order to measure the same colour during the staining and bleaching process, a thermoformed plastic positioning device was used, perforated according to the probe tip of the VITA EasyShade. Between the sessions the disks were stored in distilled water at room temperature.

Data were analyzed using one-way repeated measures ANOVA was and multiple comparisons were adjusted using the Bonferroni method ($p<0.05$).

3. Surface roughness

Surface changes were identified by surface roughness measurements, using the Mitutoyo SJ 210 Surftest. Mean arithmetic deviation of the profile (Ra) was measured in 5 points of each sample, at the beginning and at the end of each bleaching protocol, for each group. Measurements were made with the profile method using a standard stylus device. The measuring speed, pin diameter, and pin top angle of the tool were 0.25mm/s, 5 μm, and 90°, respectively. The measuring force of the scanning arm on the surfaces was 4 mN, which did not cause any significant damage to the surface according to Mitutoyo Surftest SJ-201 user manual. Measurements were performed at room temperature, and the pin was calibrated before the tests. Surface roughness was evaluated for each specimen at the beginning and at the end of each bleaching protocol. The recorded data were statistically analyzed using Univariate ANOVA ($\alpha=0.05$).

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CONFLICTS OF INTEREST

There are no conflicts of interests

R E F E R E N C E S

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