Evaluation of the Effect of Staining Agents on the Color Stability of Composite Resins that Polished at Different Interval Times (an in Vitro Study).

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ABSTRACT

Aims: This study investigated the effect of polishing times (immediately, 1 hour and 24 hour after light curing) and staining solutions on the color stability of two composite resins. Materials and Methods: Nanocomposite (Filtek Z350) and microhybrid composite (Valux plus) were used. Sixty discs (8mm diameter×2mm height) were made for each composite, and divided into four groups: the first group was polished immediately after light curing; second group was polished after 1 h; third group was polished after 24 h of curing; and the fourth group was the control. Color was measured with spectrophotometer. After baseline color measurements, the specimens of each group divided into three subgroups; specimens of first subgroup were immersed in coffee; specimens of second subgroup were immersed in orange juice; specimens of third subgroup were immersed in distilled water, after 7 day of immersion, the color was measured again and ΔE were calculated. Statistical analysis: Two-way ANOVA, Duncan's Multiple Range test and t-test. Results: The influence of polishing times and staining solutions on color change was significant. Polishing 24 hours after curing presented lowest ΔE value. Coffee promoted more color change than orange juice and distilled water, Filtek Z350 showed lower ΔE than Valux plus. Conclusions: Immediate polishing negatively affect the color stability of composite resins, coffee had a significant influence on discoloration of composite resins. The nanocomposite showed higher color stability than microhybrid composite.

Key words: Color stability, Staining, Dental finishing/ polishing time

INTRODUCTION

In the era of the tooth-colored dental restorations, color stability has become one of the major requirements of restorative materials. It is well known that the original color of composite resins can be changed within a certain period of time (1). Discoloration of tooth-colored, resin-based materials may be caused by intrinsic and extrinsic factors. The intrinsic factors involve the discolora-
tion of the resin material itself, such as the alteration of the resin matrix and of the interface of matrix and fillers. Extrinsic factors for discoloration include staining by adsorption or absorption of colorants as a result of contamination from exogenous sources (2). The consumption of coffee and soft drinks, for example, has a high prevalence in the contemporary society, especially in industrialized countries. It has been demonstrated that surface discolorations in composite resins are related to hygiene, eating habits and smoking. The maintenance of the esthetics of a restoration is therefore related to the patients’ habits and lifestyle (3). Finishing refers to the reduction of the restoration to obtain the desired anatomy (4). Polishing refers to the reduction of the surface roughness of the restoration and scratches created by the finishing instruments (5). These procedures are commonly required after placement of direct composite resin restorations since they minimize the retention of plaque and stains and other problems resulting from the exposure of rough surfaces to the oral environment (6). In order to measure objectively the color alterations on composite resin restorations, some methods have been experienced, among them the spectrophotometry, which makes the study of several parameters related to color stability of composite resins possible. Vita Easyshade spectrophotometer can measure special sections of visible light spectrum based on the body light reflexion of wavelength specifics. By this method reflected wavelength by a body is changed in values expressed in ΔE* units (7). The organic matrix structure and the characteristics of fillers exert a direct influences on the surface roughness and staining susceptibility of composite resins (8). Due to the high staining index of coloring agents on composite resins, patients are recommended to avoid contact with coloring substances for at least 24 hour after completion of composite resin restorations (9).

MATERIALS AND METHODS

Two types of composite materials (shade A3) were selected in the study: nano-fill composite (Filtek™ Z350 XT, 3M™ ESPE, USA) and microhybrid composite (Valux Plus™, 3MTM ESPE, USA). Finishing and polishing system was Superfix (TDV Dental Ltda. Brasil). A brass mold (8 mm in diameter and 2 mm in height) was used to fabricate the discs Figure( 1).

Figure (1): The samples of composite resin after curing.
The mold was placed on a transparent matrix strip supported by a glass slide. The Composite resin was inserted into the mold in one increment. The mold and resin composite were covered by another matrix strip and glass slide. Light pressure was applied until the upper matrix strip and slide came into contact with the mold to expel excess material and to avoid air entrapment. Each specimen was photoactivated for 40 s using a LED light source (BEING foshan, China) with an intensity of 650 mW/cm² as measured with a radiometer (Cromatest 7041, Megaphysik, Germany). Sixty specimens were made from each composite resin (total=120 specimens). The specimens of each composite were divided into four groups according to the polishing time:

G1 the specimens was polished immediately after light curing (n=15),
G2 the specimens was polished 1 h after light curing (n=15, specimens were immersed for 1 hour in distilled water at 37°C then polished),
G3 the specimens was polished 24 h after light curing (n=15, specimens were immersed for 24 hour in distilled water at 37°C then polished)
G4 unpolished specimens served as a control (n=15).

The specimens were polished with Superfix aluminium oxide discs in the following order: coarse, medium, fine, and superfine grits. Both sides of the specimens were polished. Each disc was used in a circular motion with intermittent pressure for 20s with a low speed hand piece. The specimens were washed with an air/water spray to remove debris, air dried at each disc change. The discs were discarded after each use. Next, a mark was made on the bottom surface of the specimens to ensure that color reading would be done always at top surface. The specimens were stored in distilled water for 24 hours at 37°C. This was done to mimic the first day of service in the oral environment.

Baseline color measurements were performed using a spectrophotometer (Vita Easyshade; Vident, Brea, CA, USA) using CIE L*a*b* (Comission International l’Eclairage). The analyzed parameters were the values for L*, a* and b*. The L* represents the value (lightness or darkness). The a* value is a measure of redness or greenness. The b* value is a measure of yellowness or blueness. The spectrophotometer consisted of a base unit and a handpiece with a 5 mm diameter probe tip. The device must be calibrated each time when the unit was power up. Calibration is achieved by placing the probe against a calibration block housed within the machine,according to the manufacturer’s instructions. Color measurement was carried out by placing the probe tip on the central part of specimens, which was placed on a white background to prevent potential absorption effects on any of colors parameters (Figure 2).
All specimens were measured three times, and then the average values were calculated. After baseline color measurement, the specimens of each group were assigned to three subgroups according to the staining solutions.

First subgroup (n=5) was immersed in coffee prepared by dissolving 10 grams of coffee (Nescafé Matinal; Nestle, Brasil) in 1 Liter of boiling distilled water according to the manufacture instruction.

Second subgroup (n=5) was immersed in an fresh orange juice.

Third subgroup (n=5) was immersed in distilled water.

The solutions were replaced every day. After 7 days of immersion in the solutions(10), the specimens were rinsed with distilled water for 5 min and blotted dry with absorbent paper before the second color or measurement. Color of the specimens was measured after immersion in the different solutions by the spectrophotometer, as previously described. The following CIE formula was used to determine the total color difference (ΔE*):  

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

$$\Delta L^* = (L^*1 - L^*0), \Delta a^* = (a^*1 - a^*0), \text{ and } \Delta b^* = (b^*1 - b^*0)$$. Number 0 represents the baseline data, while no.1 represents the color data after staining.

RESULTS

The means, standard deviations of color change (ΔE*) in all groups of nanocomposite and microhybrid composite are presented in Table (1).
Table (1): Descriptive Statistics of color change of all groups for Nanocomposite and Microhybrid composite.

| Composite type       | Time              | Solution       | Mean   | SD       | No.   |
|----------------------|-------------------|----------------|--------|----------|-------|
| Control              | Distilled Water   | 1.276420       | 0.2294129 | 5        |
|                      | Coffee            | 7.905880       | 1.1525750 | 5        |
|                      | Orange            | 3.455980       | 0.0427567 | 5        |
|                      | Total             | 4.212760       | 2.9240538 | 15       |
|                      | Distilled Water   | 0.504920       | 0.0780859 | 5        |
| Immediately after curing | Coffee        | 5.080660       | 0.3247180 | 5        |
|                      | Orange            | 1.616520       | 0.3487245 | 5        |
|                      | Total             | 2.400700       | 2.0334369 | 15       |
| Nanocomposite        | Distilled Water   | 0.526940       | 0.1125318 | 5        |
| 1 Hour after curing  | Coffee            | 4.622700       | 0.8391944 | 5        |
|                      | Orange            | 1.330500       | 0.1724395 | 5        |
|                      | Total             | 2.160047       | 1.8914434 | 15       |
|                      | Distilled Water   | 0.498080       | 0.1430858 | 5        |
| 24 Hours after curing| Coffee            | 4.208780       | 0.4173674 | 5        |
|                      | Orange            | 0.979100       | 0.1960552 | 5        |
|                      | Total             | 1.895320       | 1.7248522 | 15       |
|                      | Distilled Water   | 0.701590       | 0.3679673 | 20       |
|                      | Coffee            | 5.454505       | 1.6417722 | 20       |
|                      | Orange            | 1.845525       | 1.0020032 | 20       |
|                      | Total             | 2.667207       | 2.3253158 | 60       |
|                      | Distilled Water   | 2.859780       | 0.0639759 | 5        |
|                      | Coffee            | 4.528760       | 0.1302711 | 5        |
|                      | Orange            | 3.479900       | 0.0715436 | 5        |
|                      | Total             | 3.622813       | 0.7182143 | 15       |
|                      | Distilled Water   | 2.794860       | 0.1062826 | 5        |
| Immediately after curing | Coffee        | 6.364180       | 0.3994648 | 5        |
|                      | Orange            | 3.442660       | 0.3263429 | 5        |
|                      | Total             | 4.200567       | 1.6315583 | 15       |
| Microhybrid composite| Distilled Water   | 2.230680       | 0.1731913 | 5        |
| 1 Hour after curing  | Coffee            | 5.232360       | 0.4286887 | 5        |
|                      | Orange            | 2.894620       | 0.2496410 | 5        |
|                      | Total             | 3.452553       | 1.3618328 | 15       |
|                      | Distilled Water   | 1.219340       | 0.1661084 | 5        |
| 24 Hours after curing| Coffee            | 3.912860       | 0.4863296 | 5        |
|                      | Orange            | 2.336620       | 0.1808762 | 5        |
|                      | Total             | 2.489607       | 1.1802076 | 15       |
|                      | Distilled Water   | 2.276165       | 0.6857535 | 20       |
|                      | Coffee            | 5.009540       | 0.9994763 | 20       |
|                      | Orange            | 3.038450       | 0.5223530 | 20       |
|                      | Total             | 3.441385       | 1.3819864 | 60       |

Two-way ANOVA, for the effect of polishing time, staining agent and their interactions of nanocomposite and microhybrid composite are shown in Table (2)
Table (2): Two way ANOVA, for the effect of polishing times, staining solutions and their interactions of Nanocomposite and Microhybrid composite.

| Composite type       | Source                | Type III Sum of Squares | df | Mean Square | F       | p–value |
|----------------------|-----------------------|--------------------------|----|-------------|---------|---------|
| Nanocomposite        | Corrected Model       | 308.635                  | 11 | 28.058      | 129.707 | 0.000   |
|                      | Intercept             | 426.839                  | 1  | 426.839     | 1973.224| 0.000   |
|                      | Time                  | 49.692                   | 3  | 16.564      | 76.573  | 0.000*  |
|                      | Solution              | 246.157                  | 2  | 123.078     | 568.976 | 0.000*  |
|                      | Time * Solution       | 12.787                   | 6  | 2.131       | 9.852   | 0.000*  |
|                      | Error                 | 10.383                   | 48 | 0.216       |         |         |
|                      | Total                 | 745.858                  | 60 |             |         |         |
| Microhybrid composite| Corrected Model       | 109.177                  | 11 | 9.925       | 135.890 | 0.000   |
|                      | Intercept             | 710.588                  | 1  | 710.588     | 9728.924| 0.000   |
|                      | Time                  | 22.729                   | 3  | 7.576       | 103.731 | 0.000*  |
|                      | Solution              | 79.584                   | 2  | 39.792      | 544.808 | 0.000*  |
|                      | Time * Solution       | 6.864                    | 6  | 1.144       | 15.663  | 0.000*  |
|                      | Error                 | 3.506                    | 48 | 0.073       |         |         |
|                      | Total                 | 823.271                  | 60 |             |         |         |

Corrected Total 319.019 59
Corrected Total 112.683 59

Show that there is a significant difference (P<0.05) in ΔE* among the groups with different polishing times, staining agents and their interactions.

Duncan's Multiple Range Test for comparing the effect of polishing times on the ΔE* of nanocomposite is presented in Table (3)

Table (3): Duncan's Multiple Range Test for comparing the effect of polishing times on the ΔE* of Nanocomposite and Microhybrid composite.

| Composite type       | Time                          | No. | Duncan's Grouping |
|----------------------|-------------------------------|-----|-------------------|
|                      |                               |     | A                 | B             | C         |
| Nanocomposite        | 24 Hours after curing         | 15  | 1.895320          |               |           |
|                      | 1 Hour after curing           | 15  | 2.160047          | 2.160047      |           |
|                      | Immediately after curing      | 15  | 2.400700          |               |           |
|                      | Control                        | 15  | 4.212760          |               |           |
|                      | 24 Hours after curing         | 15  | 2.489607          |               |           |
| Microhybrid composite| 1 Hour after curing           | 15  | 3.452553          |               |           |
|                      | Control                        | 15  | 3.622813          |               |           |
|                      | Immediately after curing      | 15  | 4.200567          |               |           |
Show that the groups which was polished 24 hours after curing had a lower statistically significant color change than the groups which was polished immediately after curing and the control group, while there was no significant color change between the groups which was polished 1 hour after curing and immediately after curing.

Duncan's Multiple Range Test for comparing the effect of polishing times on the $\Delta E^*$ of microhybrid composite is presented in Table (3), and show that the groups which was polished 24 hours after curing had a lower statistically significant color change than the other groups which was polished 1 hour, immediately after curing and the unpolished (control) group, while there was no significant color change between the group which was polished 1 hour after curing and the control group. The microhybrid composite color had a more significant color change when polished immediately after curing than the control and when polished 1 hour and 24 hour after curing did.

Duncan's Multiple Range Test for comparing the effect of staining agents on the $\Delta E^*$ of nanocomposite and microhybrid composite are presented in Table (4)

### Table (4): Duncan's Multiple Range Test for comparing the effect of staining solutions on the $\Delta E^*$ of Nanocomposite and Microhybrid composite.

| Composite type         | Solution   | No. | Duncan's Grouping | A       | B       | C       |
|------------------------|------------|-----|-------------------|---------|---------|---------|
| Nanocomposite          | Distilled  | 20  | 0.70159           |         |         |         |
|                        | Water      |     |                   |         |         |         |
|                        | Orange     | 20  | 1.845525          |         |         |         |
|                        | Coffee     | 20  | 5.454055          |         |         |         |
| Microhybrid composite  | Distilled  | 20  | 2.276165          |         |         |         |
|                        | Water      |     |                   |         |         |         |
|                        | Orange     | 20  | 3.038450          |         |         |         |
|                        | Coffee     | 20  | 5.009540          |         |         |         |

**Note:** Different letters mean significant differences.

Show that color change in groups immersed in coffee was significantly higher than that in groups immersed in other staining solutions tested in the study.

Student's t-test for comparing the effect of composite resin types on the $\Delta E^*$ is presented in Table (5).

### Table (5): Student's t–test for the effect of composite resin types on the $\Delta E^*$.

| Composite     | No. | Mean    | SD       | SE Mean  | T      | df    | p–value |
|---------------|-----|---------|----------|----------|--------|-------|---------|
| Microhybrid   | 60  | 3.441385| 1.3819864| 0.1784137| 2.217  | 118   | 0.029*  |
| Nanocomposite | 60  | 2.667207| 2.3253158| 0.3001970|        |       |         |
It was obvious that the nanocomposite showed significantly (P<0.05) less color change than microhybrid composite.

**DISCUSSION**

One of the disadvantages of Composite resin restorations is the color change with time, which is a major reason for replacing restorations. Restoration polishing is particularly important in order to delay the discoloration of the composite. Higher smoothness and less porosity reduce the adherence of agents responsible for changing the color of composites, such as dental biofilm, food colorants, tobacco, and others.

For both composites, polishing after 24 hours showed lowest color alteration than other groups. This result can be explained by the surface quality of composite resins and are related to roughness and staining. The delayed (24 hour) polishing may produce smoother surface and will retain less stains than rough surface. The probability of stain penetration into resin matrix is lower in smoother composite surface than in rough surfaces.

Some others showed that immediately finishing/polishing resulted in significantly higher roughness compared to delayed polished samples for hybrid and nanohybrid composites. ΔE values observed in study by certain others revealed that the lower the roughness after polishing, the greater the resistance to staining of composite resins.

The result showed that the specimens when polished immediately show higher ΔE* values than when polished 1 or 24 hours after curing. This result may be attributed to the incomplete maturity of the restorative material at the time of finishing/polishing. The composite polymerization reaction would not be complete prior to 24 hours. Studies showed that composite resins continue to polymerize after the initial set and they harden over a period of time and suggest that delaying finishing/polishing would allow the resin to become harder and then smoother surface may be made. Restorations that are not properly polymerized may result with a softer surface. During finishing/polishing, the matrix supporting inorganic filler particles might wear away leaving irregularities projecting from the surface.

For nanocomposite, the control group showed significantly higher color alteration than other groups. Finishing the composite surface with a celluloid strip can produce the smoothest composite surface, but the celluloid strip finished composite surface discolors more than a polished one. Since resin matrix emerges to the surface, which is highly rich in organic components. Moreover, resin matrices tend to absorb more water and are more prone to staining, once water is the vehicle for dyes penetration. Therefore, it is recommended that composite resins should be finished and polished in the appropriate time, because early finishing and polishing result in greater staining susceptibility.

According to individual ability of human eye to appreciate differences in colors, three different intervals were used to distinguish changes in color values: ΔE<1 - imperceptible by the human eye; 1.0 < ΔE < 3.3 - considerable appreciate only for skilled person, both clinically acceptable and, ΔE>3.3 - easily observed, these color changes values are not clinically acceptable. For specimens immersed in water, and orange juice, ΔE* values >3.3 were not detected. The specimens immersed in coffee, ΔE values > 3.3 were observed for all groups, this result indicate that the coffee solution has a stronger staining ability than other staining agents tested in this study. Coffee discolora-
tion originates from both mechanisms of adsorption of polar colorants onto the surface and absorption in the sub-surface layer, this adsorption and penetration of colorants into the organic phase of the materials being due to the compatibility of the polymeric phase with the yellow colorants of coffee\(^{(21)}\).

Based on the results (Table 5), microhybrid composite resin, exhibited more significant color changes than nanocomposite. Filtek Z350 presented color changes within the clinically acceptable levels (\(\Delta E<3.3\)). Staining susceptibility of resin-based materials may be related to resin filler type, type of resin matrix or type of the staining agent\(^{(22)}\). UDMA seems to be more stain resistant than bis-GMA due to its low sorption and water solubility characteristics\(^{(23)}\). The resin systems of Filtek Z350 consist of; Bis-GMA, UDMA, and TEGDMA and bis-EMA. The presence of UDMA makes this composite more color stable and more hydrophobic\(^{(24)}\). However, the resin system of Valux PlusTM consist of; Bis-GMA and TEGDMA.

The inferior color stability of Valux PlusTM materials has been attributed to its resin matrix and filler compositions. It has been noted that a composite with large filler particles are more prone to water aging discoloration than a composite with small filler particles, which is in line with the hydrolytic degradation matrix filler interfaces. Thus, a composite with large filler particles has more color permeability than a composite with small filler particles\(^{(21)}\). Accordingly, we can conclude that the microhybrid Valux PlusTM composite, in the presence of small-to-large filler particles (filler particle size range of 3.5 to 0.01 micron), with a BIS-GMA and TEGDMA resin base, is more prone to color discoloration.

**CONCLUSIONS**

Under the limitation of this study we found that the immediate polishing negatively affect the color stability of nanocomposite and microhybrid composites, so the polishing procedures should be delayed 24 hours after curing for best color stability. The nanocomposite showed higher color stability than microhybrid composite, so the practitioners should take into consideration the staining susceptibility of the resin composite. For the two composite resins, the immersion of specimens in coffee caused a significant color change.

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