Optical properties stability of light-cured versus pre-cured CAD-CAM composites

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Abstract - The aim of this study was to evaluate the optical properties changes after staining of pre-cured (PC) and light-cured (LC) composites. Specimens were prepared using different LC composites (GrandioSO – Voco, Filtek Z350 – 3M/ESPE, Opallis – FGM and Kalore – GC) and four PC composites (Grandio Blos – Voco, Lava Ultimate – 3M ESPE, Brava Block – FGM and Cerasmart – GC) (n=20). Baseline color, gloss, translucency, and fluorescence were evaluated. Staining protocol was performed for 15 days and the final optical properties were reevaluated. The changes on each property were calculate (∆Gloss, ∆Translucency, ∆Fluorescence, ∆E*00). Data were analyzed by ANOVA and Tukey’s test (α=5%). Changes on all properties were observed after staining for all materials, with darkening and reduction of gloss, fluorescence and translucency. Non-significant differences were observed between the light-cured and pre-cured materials of the same manufacturer for the ∆G and ∆T, but significant differences existed for ∆E and ∆E*00. For ∆F, the only significant differences were observed between Brava Block and Opallis (smaller). For ∆E*00, only the light-cured composites GrandioSO and Z350 showed significant less change than the corresponding blocks. Pre-cured composites were affected the same way than light-cured ones by the staining in relation to the reduction of gloss and translucency. A higher reduction of fluorescence was observed only for one brand of block and was similar for the others. Two brands the light-cured materials showed less staining, while for the others was similar. The effects of staining vary according to the composite formulation.

Keywords—Color, Composite, Fluorescence, Gloss, Staining, Translucency

I. INTRODUCTION

A frequent cause of composite restoration replacement is the change of their optical properties during time. These changes may lead to an unacceptable difference between the remaining of tooth structure and the restoration [1,2]. The most important optical properties concerning the esthetic outcome of composite restorations is the color matching [3,4]. However, the proper translucency to simulate the enamel and dentin tissues [5] and a surface gloss similar to the neighbor enamel surface [6] are also very important. In addition, when exposed to an environment containing mainly UV light, the fluorescence of the restorative material in relation to the tooth is extremely relevant [7,8]. An adequate composite resin shade and translucency must be selected before the restoration is performed, allowing to create undetectable restorations [9]. However, the restorative material suffer different alterations in the oral environment during the years, resulting on loss of the desired optical characteristics [10]. Color alteration can occur inside or on the surface of the material. In the first case, it can be caused by chemical alteration of the composite matrix and polymerization initiators, or by absorption of substances available in the oral environment [11–17]. Besides, the insufficient polymerization caused for inadequate irradiation from the light curing units increase the residual monomers content, increasing water sorption as well of staining molecules [18,19]. The use of higher concentration of photoinitiator and tertiary amine on the material’s formulation can also increase color alteration [20].

The color change can also result from surface adsorption of different substances, which is mainly associated to the material surface roughness [21]. Some studies also showed that staining substances can produce degradation and softening of the composite polymer, reducing microhardness and increasing roughness [22]. A rougher surface loss its glossy aspect, affecting the esthetics of the restoration [23]. The softening of the organic matrix increases the wear exposure of inorganic fillers and its displacement, creating pores that is increase biofilm accumulation and staining [24,25]. Examples of these most common agents are coffee, wine, tobacco, ethanol and different kinds of oils available in the regular human diet [26,27]. With the development and improvement of the CAD/CAM technology, the use of pre-cured composite blocks is becoming more popular. On this case, the material’s polymerization is performed by the industry, using a chemical curing process, under pressure and heating, which increases the degree of conversion and physical properties [28-30]. This improved polymerization is expected to increase mechanical properties and stability when exposed to the oral environment [31-33]. Studies have shown that polymerized composites outside the oral cavity, with additional pressures and also different treatments, such as
thermal, can improve the physical properties and degree of conversion of composite resins, increasing restoration longevity[34-36].

Besides the curing process, the composition of a composite may have a great influence on the color stability of the material, after exposure to different staining substances [37]. The mostly frequent used composites have in their organic composition some monomers: bisphenol A glycidyl methacrylate (Bis-GMA), triethylene glycol dimethacrylate (TEGDMA), dimethacrylate urethane (UDMA) and Bisphenol A ethoxylated methacrylate, Bis-EMA [38]. Although the basic components are the same, each manufacturer choose a specific blend of resin monomers, which present different levels of water sorption that can interfere on composite staining. Some studies showed that TEGDMA presented the higher level of water sorption, Bis-GMA and UDMA were intermediate and Bis-EMA the lowest. One of the reason was the presence of hydroxyl groups on Bis-GMA and UDMA, while Bis-EMA has a central phenyl ring core stiff[37-39]. Another reason could be the lower degree of conversion and consequent unreacted residual monomers for certain blends [40]. The TEGDMA differences might be associated to their distinct physical structure [35]. TEGDMA network is more heterogenic than others, resulting on larger micro-porous space between the polymeric agglomerates, that can be related to a higher water sorption [36]. Although there are no hydroxyl groups on TEGDMA, it presents water affinity because of the ether linkage binding structure, which is compatible with water [37]. Therefore, the ether linkages structures can increase composite’s water sorption.

Considering the differences between the conventional light-cured composites and the pre-cured CAD-CAM blocks, and their different formulations from various manufactures, the aim of the present in vitro study was to evaluate the effect of staining substances, generally available on the oral cavity, over color, translucency, fluorescence and gloss of different composite materials. The first null hypothesis tested was that optical properties of conventional and CAD-CAM blocks composites do not differ from each other. The second and third null hypothesis were that the alteration of optical properties after immersion in staining solution is not related to the material (light-cured vs pre-cured CAD-CAM) or the composite configuration.

II. MATERIAL AND METHODS

A. Specimens’ preparation

Cylindrical specimens of 6mm of diameter and 1,1mm height were prepared, using four light-cured composites and four pre-cured CAD-CAM blocks (n=20). The technical information about the material’s tested are shown on Table 1. All materials were shade A2. For the light-cured ones the chromatic enamel shade was used, while for the composite blocks the level of translucency used was the LT (low translucence).

The light-cured composite specimens were fabricated using a silicone matrix mold. The composite was applied in a single increment and light-cured for 20s with a LED light-curing unit (Valo Cordless, Ultradent, Salt Lake City, Utah, USA) with a radiant emittance of 1000 mW/cm². For the pre-cured composites, cylinders were first obtained from the pre-cured CAD-CAM blocks using a diamond trephine mill. In order to obtain specimens with the same thickness of the light-cured ones, the cylinders were sliced using a diamond disc on a low-speed cutting machine (Labcut, Extec, Enfield, CT, USA). The surface of all specimens was polished using silicon-carbide abrasive papers (grit #1200, #2400 and #4000, Extec Corp, Enfield, CT, USA) in polishing machine under water cooling, for 30, 60 and 120 s, respectively. After polishing all specimens presented 1mm height.

B. Color, gloss, translucency and fluorescence

Color and translucency measurements were performed by a colorimetric spectrophotometer (CM 2600d, Konica Minolta, Osaka, Japan), adjusted for small area view (SAV), D65 standard illuminant, 100% UV included, observer angle of 2° and specular component included (SCI). The reflectance data were converted to the chromatic coordinates L*, a*, and b*, using the Spectramagic NX software (Konica Minolta, Osaka, Japan). For analysis of translucency, the translucency parameter (TP) was calculated as color difference between L*, a*, b* coordinates obtained placing the specimens over the white and black standard backgrounds [41].

Gloss measurement was performed by a gloss meter device (Novo-Curve, Rhopoint, Queensway, England), which presented a 2x2mm reading area and 60° of light incidence. The results were expressed in Gloss Units (GU).

For the fluorescence, a spectrofluorophotometer (RF-5301 PC, Shimadzu Corp., Kyoto, Japan) was used. This measurement was performed by a 365nm wavelength for excitation [39] and a detection spectrum of 400-600nm. The wavelength and intensity emission of each specimen were obtained.

C. Staining protocol

A staining broth was prepared based on the American Dental Association (ADA) recommendation for laboratory testing, containing some common dental staining substances [41]. The broth was prepared with instant coffee, black tee, gastric mucin, FD&C red, FD&C yellow 5, red wine and distilled water. Specimens were immersed in the broth for 15 days at 37°C, with daily changes [37]. After that, the optical properties of the specimens were evaluated again. The changes of gloss (ΔG), translucency (ΔT) and fluorescence (ΔF) were calculated subtracting the final value from the respective baseline one. The color change was calculated using the ΔE*00 formula, according to the Commission International the L’Eclairage (CIE) [42].

D. Statistical analyses

To test the difference in optical properties between groups, either the baseline data and the data of changes on gloss (ΔG), translucency (ΔT), fluorescence (ΔF) and color (ΔE*00) were submitted to one-way ANOVA and post-hoc Tukey’s test. For all analysis, a significance level of 5% was adopted and the Statistica for Windows software (StatSoft, Tulsa, OK, USA) was used.

III. RESULTS

Mean values (±SD) of gloss (GU), fluorescence (I) and translucency (TP) obtained with different materials before immersion in staining broth are displayed in Table 1. One-way ANOVA (p=0.000) showed that GrandioSO, Opallis and Brava Block are less glossy than other tested materials, while
performing a restoration, in order to select the proper properties of enamel and dentin. The optical properties of pre-cured CAD-CAM materials from the same manufacturers and GrandioSO showed less change than Cerasmart, while showed less change than Lava and Kalore. For ΔF, Opallis differences were observed for the ΔG. For the ΔT, Opallis corresponding blocks.

The absolute means of all optical parameters before and after staining are displayed on Figs 1-3, while means changes translucent than the light-cured composites. In relation to fluorescence, all blocks showed higher values than the light-cured version for all manufacturers. In relation to translucency, significant differences were observed only for FGM and GC manufacturer, with the CAD-CAM blocks being less translucent than the light-cured composites.

The present study showed significant differences among the groups for the changes on translucency (ΔT) (p=0.0076), fluorescence (ΔF) (p=0.0001) and color (ΔE*) (p=0.0001). Non-significant differences were observed on gloss parameter (ΔG) (p=0.0930). When the light-cured and pre-cured CAD-CAM materials from the same manufacturers were compared, Grandio Blocs showed higher values than the light-cured GrandioSO, while non-significant differences were observed for the other manufacturers. In relation to fluorescence, all blocks showed higher values than the light-cured version for all manufacturers. In relation to translucency, significant differences were observed only for FGM and GC manufacturer, with the CAD-CAM blocks being less translucent than the light-cured composites.

One-way ANOVA highlighted significant differences among the groups for the changes on translucency (ΔT) (p=0.0076), fluorescence (ΔF) (p=0.0001) and color (ΔE*) (p=0.0001). Non-significant differences were observed on gloss parameter (ΔG) (p=0.0930). When the light-cured and pre-cured CAD-CAM materials from the same manufacturers were compared, non-significant differences were observed for ΔG and ΔT. For ΔF, the only significant differences were observed between Brava Block and Opallis from FGM Company. For ΔE*, only the light-cured composites GrandioSO and Z350 showed significant less change than the corresponding blocks.

Comparing all materials at the same time, non-significant differences were observed for the ΔG. For the ΔT, Opallis showed less change than Lava and Kalore. For ΔF, Opallis and GrandioSO showed less change than Cerasmart, while Brava showed the higher change than all the others. For ΔE*, Opallis and GrandioSO showed less change than Grandio Blocs, while Lava exhibited the higher color change than all the other materials.

**IV. DISCUSSION**

Composite resins are restorative materials used for aesthetic procedures due to their optical properties close to dental structure [43]. However, the color variability of human teeth justifies the need of composite systems which includes different opacities and translucencies in order to let the clinician mimic as much as possible the complex optical properties of enamel and dentin. The optical properties of a restorative material are determined by several parameters, such as gloss, translucency, fluorescence and color, which globally contribute to the overall aesthetic appearance of a material.

The present study showed significant differences among the tested materials in relation to all optical properties evaluated, thus rejecting the first null hypothesis. Therefore, the clinician must consider those differences before performing a restoration, in order to select the proper composite which best matches optical characteristics of the involved teeth. For instance, in a clinical case where the dental enamel is highly translucent, materials with smaller translucency, such as GrandioSO, Grandio Blocs and Brava Block (Table 2) would probably result in poor esthetics. On the other hand, a highly smooth labial surface of incisors, typical of middle and old age patients, would be better replicated with materials with higher gloss, such as Z350, Lava, Kalore and Cerasmart.

### Table I. Mean values (±SD) and results Tukey’s test considering the absolute values of gloss, fluorescence and translucency before staining.

| Composite / Manufacturer | Gloss** | Fluorescence** | Translucency** |
|--------------------------|---------|---------------|---------------|
| GrandioSO / Voco         | 49.03 (11.26)a | 59.02 (3.72)a | 12.38 (0.51)a |
| Grandio Blocs LT / Voco  | 60.07 (10.62)b<sup>c</sup> | 128.47 (8.24)c | 11.87 (0.55)a |
| Filtek Z350 / 3M ESPE    | 73.39 (14.20)d | 70.38 (8.13)a | 14.76 (1.22)b |
| Lava Ultimate LT / 3M ESPE | 70.10 (6.50)d | 95.58 (4.56)b | 15.07 (0.78)c |
| Opallis / FGM            | 47.13 (7.73)a | 63.42 (4.88)a | 14.36 (0.75)b |
| Brava Block LT / FGM     | 55.66 (13.11)b<sup>c</sup> | 625.98 (65.74)f<sup>c</sup> | 11.71 (0.82)a<sup>c</sup> |
| Kalore / GC              | 68.65 (6.58)kd<sup>c</sup> | 157.65 (14.08)kd<sup>c</sup> | 16.33 (0.71)kd<sup>c</sup> |
| Cerasmart LT / GC        | 73.43 (6.53)kd | 185.85 (19.69)kd<sup>c</sup> | 14.15 (1.05)kd<sup>c</sup> |

* Significant differences on the columns. **Groups flowed by different letter on columns present significant differences.

Gloss and surface roughness are usually linked together, and the relationship between the two has been illustrated in previous studies [44,45]. Tunac et al. [27] showed how the filler size, distribution, geometry and volume fraction could influence the polishing ability of composites, improving with smaller particle size and higher filler loading. The present study showed significant differences between light-cured composite, with GrandioSO and Opallis being less gloss than Z350 and Kalore. Z350 was the only pure nanoparticle light-cured composite tested in the present study, which could help to explain the higher glossy surface than other tested composites. On the other hand, Kalore contains prepolymerized silica filler, which can be easily wear by abrasive particles and produce a smooth surface [46-48].

Regarding CAD-CAM blocks, gloss was in general higher than light-cured counterparts even if only Grandio Blocs showed higher gloss than GrandioSO, which may be related either to its smaller filler content (3% less), either to the monomer combination (UDMA instead of Bis-EMA). The high-pressure/high polymerization process for CAD-CAM blocks fabrication should lead to a less porous material with a better filler distribution [49,50].

In addition, the manufacturing process led to a conversion degree of CAD-CAM blocks higher than the light-curing composites, which could affect the effect of polishing and final gloss[27,51]. Another possible explanation of obtained results could be related to the monomer itself: generally, blocks contains UDMA monomer instead of Bis-EMA, which is present on the light cured material. The Bis-EMA monomer previously presented a twice higher solubility level in comparison to UDMA [31], probably due to the crosslinking pattern and the organic matrix strength or even be related to the filler-resin bond. Finally, although the kind of fillers are the same, CAD-CAM blocks probably have a higher ratio of silicon dioxide nanoparticles in relation to the glass ceramic, but this information is not provided by the manufacturer.
TABLE II. MEANS (±SD) OF COLOR CHANGE AND RESULTS OF TUKYÉ’S TEST.

| Composite          | ΔG  | ΔT  | ΔF  | ΔE* | ANOVA |
|--------------------|-----|-----|-----|-----|-------|
| GrandioSO-Voco     | -2.40 | 0.82 | -16.14 | 2.16 | 0.0930 |
|                    | (10.50)a | (0.52)c | (6.31)e | (1.12)a |       |
| Grandio Bloxs-Voco | -2.13 | 0.96 | 33.44 | 3.39 | 0.0076* |
|                    | (17.44)a | (0.57)ab | (16.03)bc | (0.55)b |       |
| Filtek Z350-3M ESPE| 10.43 | 0.77 | -26.99 | 2.69 | 0.0001* |
|                    | (11.97)a | (0.34)ab | (7.67)bc | (0.56)ab |       |
| Lava Ultimate-3M ESPE| 11.23 | -1.30 | 25.58 | 5.15 | 0.001* |
|                    | (15.79)a | (0.69)a | (10.80)bc | (2.16)c |       |
| Opallis-FGM        | -3.53 | -0.42 | -16.28 | 2.20 | 0.0001* |
|                    | (6.53)a | (1.09)b | (7.25)c | (0.95)a |       |
| Brava Block-FGM    | -4.93 | 0.90 | 167.11 | 2.51 | 0.0001* |
|                    | (14.32)a | (0.37)ab | (90.99)bc | (0.69)ab |       |
| Kalore-GC          | -8.63 | -1.09 | 45.97 | 2.76 | 0.0001* |
|                    | (14.13)a | (0.77)a | (15.88)bc | (1.47)ab |       |
| Cerasmart-GC       | 13.33 | 0.90 | -50.48 | 2.95 | 0.0001* |
|                    | (12.60)a | (0.65)ab | (16.57)b | (1.31)ab |       |

* Significant differences on the columns.

Concerning the fluorescence, which was significantly higher in CAD-CAM blocks than light-cured composites of the same manufacturer, it would impair the esthetics of a smile in an environment rich in UV light, such as in night clubs. The reason because the blocks from all manufacturers showed higher fluorescence than the light-cured materials is hard to explain. Probably the ingredient responsible for the fluorescence is higher in the blocks than in the light-cured materials, or the curing process in some way is able to influence the fluorescence emission due to an unknown mechanism.

Translucency is one of the primary factors in evaluating dental esthetics [52]. The CAD-CAM blocks from FGM and GC showed significant less translucency than the light-cured versions. That was expected, since the blocks are generally provided in two translucency levels, which are low translucency (LT) and high translucency (HT), and the one tested was the LT version. The blocks were compared with enamel shade composites, which are more translucent options for direct restorations. However, concerning the other manufacturers, the level of translucency of the LT version were similar to the direct enamel shades.

Manufacturers, the level of translucency of the LT version were similar to the direct enamel shades. Therefore, it is expected that the HT versions of those blocks would be higher than the enamel color of the corresponding direct materials.

Alterations of the optical properties over years is one of the reasons for restoration replacement, above all in the anterior [4,31]. The present study results showed that the staining solution lead to changes in optical properties, as darkening, brightness reduction, fluorescence and translucency. However, non-significant differences were found between light-cured and pre-cured CAD-CAM blocks regarding ΔG, although significant differences were observed in ΔT, ΔF and ΔE. Thus, the second null hypothesis was rejected. The composite staining can be related to their water sorption degree and to the resin matrix hydrophobic effect. If a composite absorbs water, it also absorbs other liquids, such as drinks with dark pigments that result in staining. The present study showed that aesthetic alterations on composite does not happen by a single reason or just with some composite type [53].

The resin matrix liquid sorption can lead to silane hydrolysis and microcracks formations, which allows staining solution penetration between fillers and matrix resulting in color change and consequent optical properties alteration. This process could also impact on the restoration outcome by expanding and plasticizing the resin component [54]. On the other hand, the reflectivity of light is related to the size of the filler and the filler-matrix homogeneity. The lower the filler-matrix homogeneity, the lower the reflectivity of the light [55].

Fig. 1. Means (SD) of gloss (GL) for all groups before and after staining.

Fig. 2. Means (SD) of translucency (TP) for all groups before and after staining.

Fig. 3. Means (SD) of fluorescence for all groups before and after staining.
Regarding ΔT results, the different amount of inorganic components in some tested materials could impact in translucency worsening after immersion. Opallis showed less change than Lava and Kalore for the ΔT: the resin with the least amount of inorganic content in the resin composition showed the least translucency changes, as stated by Salgado et al. [56]. In fact, Opallis presents in its composition 78.5 to 79.5% of organic content, while Lava has 80% and Kalore has 82%. Lee et al. [57] analyzed the translucency parameters of some composites, and the results showed a change after storage in water. The composite resins absorb water at the matrix-filler interface and undergo hydrolytic degradation, changing the pattern of translucency and light diffusion. Although the difference in inorganic amount is small, it could help to explain the difference found between the tested composite resins.

The high-pressure/high polymerization process which CAD-CAM blocks are submitted can also affect some optical properties changes, as observed in the present study where ΔT of Grandio blocs and Lava Ultimate was greater than light-cured counterparts. Previous works showed that the refraction index of the composite matrix changes with the curing process [56,57]. Gloss is a visual attribute of the geometric distribution of the reflected light from a surface and is an indicator of surface smoothness. The higher the gloss, the higher is the surface luster, and it is known that higher gloss can reduce the color difference effect, since the reflected light is predominant rather than the light reflected from the underlying composite material [58].

Based on the present study results, the third null hypothesis was rejected since the composite formulation did not influence the effect of staining on optical properties. Composites are exposed to oral environment, saliva, food and beverage components, in addition to drugs and other external habits [11]. Water or liquid sorption occurs through absorption in the composite resin matrix [11]. Therefore, the water sorption amount depends on the filler content of the composite and the bond quality between resin and filler. Another relevant point is when excessive water sorption occurs, decreasing the composite quality, by expanding and plasticizing the formulation components.

The decrease of composite quality cause silane hydrolyzation and microcracks formation which enhance composite staining and discoloration [11]. After immersion in staining broths, GrandioSO exhibited less color change than Grandio Blocs, while the pre-cured Lava Ultimate compound exhibited the greatest color change. The polymeric matrix composition (BisGMA, UDMA and TEGDMA) of these two types of resins may be the reason for this difference. GrandioSO composite does not contain UDMA, BisGMA. Under regular polymerization conditions, UDMA showed less water sorption than BisGMA, despite little difference [50], what might be the difference between the founded results.

V. CONCLUSION

It can be concluded the staining process similarly affected the light-cured and the pre-cured CAD-CAM composites tested, when related to gloss and translucency reduction. A higher fluorescence reduction was observed only for one brand of pre-cured block and was similar for the others. For two manufactures, the light-cured materials showed less staining than the blocks, while for the others it was similar.

The staining effect vary according to the composite formulation.

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