# **CLAUDIA BATITUCCI DOS SANTOS DAROZ**

# INFLUÊNCIA DAS FONTES E TEMPOS DE FOTOATIVAÇÃO SOBRE A PIGMENTAÇÃO E PROPRIEDADES FÍSICO-QUÍMICA E MORFOLÓGICA DE DIFERENTES TIPOS DE RESINA COMPOSTA

Tese apresentada à Faculdade de Odontologia de Piracicaba, da Universidade Estadual de Campinas, para obtenção do Título de Doutor em Clínica Odontológica, Área de Concentração Dentística.

Orientadora: Profa. Dra. Giselle Maria Marchi Baron Co-orientador: Prof. Dr. Flávio Henrique Baggio de Aguiar Prof. Dr. William Michael Johnston

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#### RESUMO

O objetivo deste estudo foi avaliar o efeito da fonte de luz (FL) e tempo de fotoativação sobre a pigmentação, grau de conversão (GC), microdureza (KHN) e rugosidade de superfície (Ra) de diferentes tipos de resinas compostas. Espécimes (2,0 mm de altura e 5,0 mm de diâmetro) foram confeccionados a partir de diferentes tipos de resinas compostas (RC) [Nanoparticuladas: Filtek Supreme Plus A2D and Filtek Supreme XT A2E (3M ESPE); Microparticulada: Durafill A2 (Heraeus Kulzer); Microhíbridas: Filtek Z250 A2 (3M ESPE) e Venus A2 (Heraeus Kulzer)], utilizando-se dois tipos de fontes de luz, halógena (Optilux 501 -Demetron) e LED (Elipar FreeLight 2 - 3M ESPE) e com diferentes tempos de fotoativação (metade do tempo, dobro do tempo e tempo recomendado pelo fabricante). Após 24h, realizou-se acabamento e polimento. GC e cor inicial foram averiguados nas superfícies de topo (T) e base (B). Posteriormente, os espécimes foram armazenados em vinho tinto. Avaliação da alteração da cor (Sistema CIE L\*a\*b\*) foi determinada após 1, 2, 7, 14, 20 e 30 dias no vinho. KHN e Ra foram determinados nas superfícies de topo e base antes e após armazenagem em vinho por 30 dias. Todos os fatores estudados foram estatisticamente significantes (ANOVA / Stepdown Bonferroni / Bonferroni Correction), havendo interações quádrupla (Resina X Luz X Superfície X Dias; p = 0,042) na avaliação da alteração de cor; e triplas, para GC (Luz X Resina X Tempo; p = 0,05; Luz X Resina X Superfície; p = 0,02), KHN (Luz X Resina X Superfície; p = 0,05; Luz X Resina X Vinho; p < 0,0001; Resina X Tempo X Superfície; p = 0,008) e Ra (Luz X Resina X Superfície; p = 0,045; Luz X Tempo X Vinho; p = 0,0008; Resina X Tempo X Superfície; p < 0,0001; Resina X Tempo X Vinho; p = 0,047; Resina X Vinho X Superfície; p = 0,022). A pigmentação da superfície de base foi significantemente maior que da superfície de topo para todos os tipos de RC e FL. A polimerização pelo dobro do tempo reduziu a pigmentação da base de dois tipos de RC. Observou-se diferença significante no GC entre T e B e a dureza do T foi maior que da B. Após armazenamento no vinho, houve diminuição da dureza do T. Não

se observou diferença significante no Ra entre T e B; no entanto, após estocagem em vinho, o Ra da base da RC microparticulada foi significantemente aumentado, independentemente do tempo de fotoativação e FL utilizada. A fotopolimerização pelo dobro do tempo recomendado parece influenciar no grau de pigmentação da base de alguns materiais resinosos, porém, não foi capaz de manter a dureza da superfície de topo após armazenagem no vinho. Diferenças entre as FL depende do tipo de resina composta e tempo de fotoativação utilizado.

Palavras-chave: fontes de luz, pigmentação, tempo de fotoativação, rugosidade, dureza, grau de conversão

#### ABSTRACT

The objective of the present study was to evaluate the effect of LCU and light curing time on the discoloration, degree of conversion (DC), microhardness (KHN) and surface roughness (Ra) of different types of composite resins. Specimens (2.0 mm of height; 5.0 mm of diameter) were fabricated from different types of composite resins (CR) [Nanofilled: Filtek Supreme Plus A2D and Filtek Supreme XT A2E (3M ESPE); Microfilled: Durafill A2 (Heraeus Kulzer); Microhybrid: Filtek Z250 A2 (3M ESPE) and Venus A2 (Heraeus Kulzer)], using two types of LCUs; halogen (Optilux 501 - Demetron) and LED (Elipar FreeLight 2 -3M ESPE) with different light curing times (half of-, double of- and the time recommended by the manufacturer). After 24h, specimens were finished and polished. DC and the initial color were evaluated at both the top (T) and bottom (B) surfaces. Further, the specimens were stored in red wine. The color change (CIE L\*a\*b\* System) was established after 1, 2, 7, 14, 20 e 30 days in the wine. KHN and Ra were evaluated at both the top and the bottom surfaces before and after the storage in the wine for 30 days. All factors studied were a statistically significant factor (ANOVA / Stepdown Bonferroni / Bonferroni Correction), with quadruple interaction (Resin X Light X Surface X Days; p = 0.042) for the color change evaluation; and triple interaction for DC (Light X Resin X Time; p = 0.05; Light X Resin X Surface; p = 0.02), KHN (Light X Resin X Surface; p = 0.05; Light X Resin X Wine; p < 0.0001; Resin X Time X Surface; p = 0.008) and Ra (Light X Resin X Surface; p = 0.045; Light X Time X Wine; p = 0.0008; Resin X Time X Surface; p < 0.0001; Resin X Time X Wine; p = 0.047; Resin X Wine X Surface; p = 0.022). The discoloration of the bottom surface was significantly higher than the top surface for all types of CR and LCU. The light curing for double of the recommended time reduced the discoloration from the bottom surface of two types of CRs. Significant difference was observed between the DC of the T and B; and the hardness of T was higher than B. After the wine storage, the hardness of T was diminished. No significant difference in Ra between T and B was observed;

however, after the storage in the wine, the Ra from the bottom surface of the microfilled composite was significantly higher, regardless the light curing time and LCU used. The lightcuring for double of the recommended time seems to influence on the degree of color change of the bottom surface of some composite materials, however, it was not capable to keep the hardness of the top surface after storage in the wine. The differences between the LCUs are dependent on the type of composite resin and light curing time used.

Key Words: light curing units, discoloration, ligth curing time, roughness, hardness, degree of conversion

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## INTRODUÇÃO

Na Odontologia atual, a busca por restaurações estéticas que mimetizam a cor dos dentes naturais tem ganhado cada vez mais destaque entre os procedimentos restauradores. Um material restaurador estético ideal deve apresentar propriedades físicas e mecânicas suficientes para resistir aos desafios encontrados no meio bucal, como também, a capacidade de manutenção da sua cor original ao longo do tempo.

Dentre os materiais restauradores estéticos disponíveis para uso direto, as resinas compostas fotopolimerizáveis recebem grande evidência, devido as suas propriedades estéticas, adesivas e de polimerização relativamente rápida (Bayne *et al.*, 1994; Franco & Lopes, 2003). Uma polimerização adequada é essencial para obtenção e manutenção de uma restauração com propriedades ideais (Yoon *et al.*, 2002; Price *et al.*, 2003; Vandewalle *et al.*, 2004). Desta forma, o tipo de fonte de luz e densidade de energia, conferida pela relação entre irradiância e tempo de fotoativação, ganham grande importância durante a realização de uma restauração em resina composta.

A fotopolimerização das resinas compostas é iniciada por meio de fotoiniciadores capazes de absorver luz em determinados comprimentos de onda (Burgess *et al.*, 2002). O fotoiniciador comumente usado é a canforoquinona, que absorve energia dentro da faixa azul do espectro de luz visível, com pico de absorbância em 468nm (Nomoto, 1997). A formação de radicais livres desencadeada pela excitação da canforoquinona, permite a conversão dos monômeros resinosos livres em cadeias poliméricas complexas (Rueggeberg, 1999). Quanto maior o número de monômeros resinosos convertidos em polímeros, melhores serão as propriedades físico-mecânicas do material formado (Feilzer *et al.*, 1995; Yoon *et al.*, 2002; Price *et al.*, 2003). Além disso, mais resistente será o material contra degradação hidrolítica, lixiviação de monômeros resinosos não reagidos e alteração de cor com o tempo (Ferracane, 1985; Janda *et al.*, 2005; Silva *et al.*, 2008). Ainda, uma resina composta insuficientemente polimerizada contem moléculas de

canforoquinona não reagidas, que conferem uma cor mais amarelada ao material restaurador. Além disso, essas moléculas de canforoquinona não reagidas podem reagir e provocar o "clareamento" do material, em conseqüência da sua própria reação química de polimerização (Janda *et al.,* 2004; Janda *et al.,* 2005). Isso tudo compromete a cor final e a própria estabilidade de cor da restauração em resina composta.

O tempo de foto emissão, a densidade de potência e o comprimento de onda da fonte de luz utilizada para a polimerização do material resinoso são fatores importantes que determinam seu grau de conversão, e conseqüentemente, a manutenção das propriedades mecânicas e estéticas do material (Feilzer *et al.*, 1995; Christensen *et al.*, 1999; Rueggeberg, 1999; Burgess *et al.*, 2002; Yoon *et al.*, 2002; Leonard *et al.*, 2002; Price *et al.*, 2003). Há uma grande variedade no mercado de unidades fotoativadoras, com intensidades e espectros de emissão luminosa variados (Small, 2001; Franco & Lopes, 2003). Atualmente, os dois tipos de aparelhos fotoativadores mais utilizados são de lâmpadas com filamento de quartzo-tungstênio em ambiente de gás halógeno (QTH) e os diodos emissores de luz (LED).

Os aparelhos QTH emitem luz branca, a qual é filtrada para redução das radiações infravermelha e ultravioleta; assim como para restrição do feixe de luz visível, somente passando a faixa de luz azul onde a absorbância do fotoiniciador é máxima (Rueggeberg, 1999; Burgess *et al.*, 2002). Estes aparelhos apresentam maior tempo de uso no mercado; porém, com o uso, apresentam diminuição gradual da intensidade de luz emitida, devido, principalmente, à degradação do bulbo e seu refletor, rompimento do filtro e danos às pontas de fibra óptica (Burgess *et al.*, 2002; Franco & Lopes, 2003). Desta forma, requerem manutenção constante para utilização adequada.

Os aparelhos LED utilizam semicondutores à base de nitrato de gálio para geração de luz azul (Yoon *et al.*, 2002). O espectro de luz produzido é estreito, mas com o pico próximo do ideal, em torno de 470 nm, para ativação dos materiais que empregam a canforoquinona (Price *et al.*, 2003). Estes aparelhos requerem menos energia para operar, não se degradam com o tempo e nem

necessitam de refletores e filtros (Burgess *et al.*, 2002; Leonard *et al.*, 2002; Franco & Lopes, 2003). Estes aparelhos vem apresentando bons desempenhos, substituindo muitas vezes os aparelhos QTH.

Quando uma resina composta recebe uma quantidade inadequada de energia total, a dureza é adversamente afetada, independente da intensidade emitida pela fonte de luz (Price *et al.*, 2003; Vandewalle *et al.*, 2004). Ou seja, mesmo utilizando um aparelho que emita uma alta intensidade de luz, porém com tempo de foto emissão reduzido, a luz não é capaz de penetrar nas regiões mais profundas correspondentes a base da restauração. Nessas áreas mais profundas da restauração de resina composta, a atenuação da intensidade de luz pode levar a uma diminuição do grau de polimerização do material, fazendo com que a resina apresente propriedades mecânicas inferiores em relação ao topo da restauração. A base da restauração, insuficientemente polimerizada, pode contribuir para deterioração da integridade marginal entre dente e restauração ao longo do tempo em conseqüência da ação repetida de tensões cíclicas – térmicas e mecânicas – e da ação contínua da saliva e solventes orgânicos, pelo fenômeno de sorção e solubilidade do material resinoso (Larsen & Munksgaard, 1991; Ferracane, 1994; Lucena-Martin *et al.*, 2001; Vandewalle *et al.*, 2004; Yap *et al.*, 2004).

Em relação a manutenção da cor da restauração em resina composta, vários fatores podem contribuir para a alteração de cor do material ao longo do tempo. Dentre estes temos: o grau de polimerização do material resinoso; o tipo, tamanho e concentração das partículas de carga; forma de ativação da reação, se química ou física; tipo de fotoiniciador; a composição da matriz orgânica, se mais hidrófoba ou hidrófila; a lisura ou rugosidade de superfície, conforme o acabamento e polimento do material restaurador, tipo de fonte de luz e tempo de fotoativação; hábitos alimentares, de higiene, e fumo do indivíduo, entre outros (Dietschi *et al.*, 1994; Uchida *et al.*, 1998; Stober *et al.*, 2001; Schulze *et al.*, 2003; Janda *et al.*, 2004; Patel *et al.*, 2006; Sarac *et al.*, 2006; Brackett *et al.*, 2007; Lee *et al.*, 2007; Lee & Powers, 2007; Sarafianou *et al.*, 2007; Ruttermann *et al.*, 2008).

Conforme a natureza orgânica do material resinoso, este pode ser mais susceptível a absorção de líquidos, e consequentemente dos pigmentos presentes no meio bucal (Dietschi *et al.*, 1994). Uma resina composta com maior quantidade de matéria orgânica, como no caso das resinas microparticuladas, ou que apresente maior porcentagem de monômeros hidrófilos, tende apresentar maior alteração de cor com o passar do tempo. Da mesma forma um material resinoso com maior rugosidade de superfície, o qual tende a acumular mais pigmentos e biofilme sobre sua superfície provocando alteração da sua cor inicial (Patel *et al.*, 2004). Materiais resinosos pobremente polimerizados também sofrem maior alteração de cor, tanto por apresentarem uma rede polimérica mal formada, quanto por apresentarem muitas moléculas fotoiniciadoras sem reagir ainda presentes no seu interior. Estas moléculas podem sofrer oxidação com o tempo, provocando o "clareamento" do material ou o seu escurecimento, devido aos subprodutos formados após reação da amina terciária (Janda *et al.*, 2004; Janda *et al.*, 2005), outra molécula também considerada como co-iniciadora da reação de fotopolimerização.

Em vista do grande número de materiais resinosos existentes no mercado, cada um com suas peculiaridades quanto ao tipo de matriz e tamanho da partícula de carga; associado ao tipo de fonte de luz, mais recente ou com maior base literária; e tendo como referência que a efetividade de polimerização é um fator fundamental para obtenção e, consequentemente, manutenção das propriedades mecânicas e estéticas do material restaurador resinoso ao longo do tempo; o conhecimento da influência das fontes de luz QTH e LED e tempo de fotoativação sobre a pigmentação de diversos tipos de resinas compostas, assim como, a determinação do grau de conversão e o comportamento dos materiais resinosos por meio da dureza e rugosidade de superfície frente à ação química de uma solução pigmentadora, são essenciais para obtenção de indícios sobre a longevidade de restaurações estéticas em resina composta.

# **OBJETIVOS**

#### **Objetivo Central**

Avaliar *in vitro* o efeito de diferentes fontes de luz, bem como, de diferentes tempos de fotoativação sobre as propriedades óticas, físico-químicas, mecânicas e morfológicas de uma variedade de resinas compostas para restaurações diretas.

#### **Objetivos Específicos**

• **Capítulo 1**- verificar a influência das fontes fotoativadoras (Halógena e LED) e tempos de fotoativação (metade do tempo, dobro do tempo e tempo recomendado pelo fabricante) sobre a pigmentação das superfícies de topo e base de três tipos de resina composta (nanoparticulada, microparticulada e microhíbrida) após estocagem em vinho tinto por 30 dias.

 Capítulo 2 – verificar, através do grau de conversão e do teste de microdureza Knoop, a efetividade dessas fontes de luz e tempos de polimerização, assim como o efeito da solução de vinho tinto sobre a dureza e rugosidade das superfícies de topo e base dos mesmos tipos de resina composta após período de 30 dias.

# CAPÍTULO 1

# COMPOSITE RESIN DISCOLORATION: INFLUENCE OF LIGHT CURING UNIT AND LIGHT CURING TIME

#### Enviado para Revista Dental Materials

**Objectives:** To evaluate the effect of light curing units (LCUs) and light curing times on composite resins discoloration over a period of 30 days immersed in red wine.

**Material and Methods:** Composite discs 2 mm thick x 5 mm diameter were fabricated from four composite resins (CR): Filtek Supreme Plus A2D (3M ESPE), Durafill A2 (Heraeus Kulzer), Filtek Z250 A2 (3M ESPE) and Venus A2 (Heraeus Kulzer). The LCUs used were Halogen – Optilux 501 (Demetron) and Blue LED – Elipar FreeLight 2 (3M ESPE). The light curing times were half of, full of and double of the time recommended by the manufacturers. After 24 h in distilled water at 37°C, the specimens were polished and baseline measurements of the CIE-L\*a\*b\* color were obtained using a spectroradiometer and an external light source with a noncontacting 45-degree illumination and 0-degree observation optical configuration. Specimens were stored in red wine (37°C) and color measurements of both top and bottom surfaces were obtained after 1-, 2-, 7-, 14-, 20- and 30-days in the wine. The red wine was renewed every week and its pH was ~3.23. Statistical analysis was performed using Repeated Measures ANOVA and stepdown Bonferroni for non related pairs and Bonferroni corrected t-test. for related pairs (p < 0.05).

**Results:** A quadruple interaction (Resin X Light X Surface X Days; p = 0.042) was observed for the color change evaluation. The discoloration of the bottom surface was significantly higher than the top surface for all types of CR and LCUs. The lightcuring for double of the recommended time reduced the discoloration from the bottom surface of Supreme A2D and Venus.

**Conclusions:** The lightcuring for double of the recommended time seems to influence on the degree of color change of the bottom surface of some

composite materials, although the effects of changes in curing time on discoloration by red wine vary significantly by the composite resin being stained, as do the effects of the LCU.

**Key Words:** Composite resin; Light curing unit; Light curing time; Color change; Food staining

#### Introduction

Color stability of composite materials may be directly related to the type of composite matrix, the size and volume of the inorganic fillers, the type of photoinitiator, the type of light curing unit (LCU), and to the amount of light curing exposure time (Dietschi et al., 1994; Janda et al., 2005; Kolbeck et al., 2006; Brackett et al., 2007; Ruttermann et al., 2008). A composite material highly compounded of organic matrix or with a higher percentage of hydrophilic monomers, is more susceptible to color change overtime. In the same direction, insufficiently polymerized composite materials show unreacted monomers and photoinitiator molecules that remain entrapped into the unstable network. The unreacted photoiniator molecules, such as canphorquinone may oxide leading to the photobleaching of the composite material. On the other hand, byproducts from tertiary amine darken the composite material overtime (Janda et al., 2004; Janda et al., 2005). Therefore, an adequate polymerization of the composite resin material is essential to maintaining its color stability overtime.

Many changes have occurred on composite's matrix composition over the years. Mostly all light curing composite resins are made of BisGMA monomers (Peutzfeldt, 1997). To be able to add fillers to the organic matrix, dimetacrilate monomers are necessary to reduce the viscosity of the BisGMA matrix. TEGDMA is one of the dimetacrilate monomers used to dilute the BisGMA matrix, however these monomers are of polar nature and more hydrophilic (Peutzfeldt, 1997; Rawls & Esquivel-Upshaw, 2005). To reduce the water sorption by composite materials, new monomers of hydrophobic nature were created by removing the hydroxyl groups from the BisGMA molecules. Ester (BisEMA) and urethanes (UDMA) monomers may improve the mechanical properties of composite material exposed to water overtime (Peutzfeldt, 1997).

The most common inorganic particles used to enhance the mechanical and physical properties of composite materials are quartz, glass, and colloidal or amorphous silica. Inorganic fillers are also responsible for reducing the composites' shrinkage during polymerization, reduce water sorption and discoloration of the material overtime. However, the bonding between the inorganic particle and the resinous matrix must be strong so that the composite material can render its maximum performance (Rawls & Esquivel-Upshaw, 2005).

Many factors can interfere with composite's polymerization, such as, the amount and color of composite material increments, the size and volume of inorganic fillers, the light intensity and wavelength bandwidth emitted by the LCU, the distance between the end of the light tip and the composite increment, as well as the type of LCU and light curing time (Rueggeberg & Jordan, 1993; Rueggeberg et al., 1994; Christensen et al., 1999; Price et al, 2000; Gagliani et al, 2002; Yoon et al, 2002). The longevity of composite restorations may be highly influenced by the degree of composites polymerization; therefore, the objectives of the present study were to evaluate the effect of LCUs and exposure times on composite resins discolorations over a period of 30 days immersed in red wine. The hypotheses tested were that the different LCU units influence the composites final discoloration and that a longer exposure time renders a composite more resistant to color change.

## **Material and Methods**

#### Specimens Fabrication

Seventy two disk specimens (n = 3) of composite resins were fabricated using a stainless steel metallic mould of 2 mm of height and 5 mm of diameter. A polyester matrix was placed on the bottom and top surfaces of the stainless steel mould, before and after the placement of the composite materials, respectively. A 500 g load was placed on top of the polyester matrix, composite material and stainless steel mould set for 30 seconds to guarantee the proper accommodation of the composite resin prior to polymerization. The type, color, composition, and light curing time recommended by the manufacturer of each composite material used in this study are shown on Table 1.

The composite disks were lightcured for different times using a conventional quartz-tungsten-halogen (HAL) light (Optilux 501, Demetron/Kerr Corp., Orange, CA, USA) and a second-generation blue light emitting diode (LED) (Elipar FreeLight 2, 3M ESPE, Seefeld, Germany). Light intensity measurements of both LCUs were made periodically throughout the specimens' fabrication (HAL: 1000 mw/cm<sup>2</sup>; LED: 990 mw/cm<sup>2</sup>), and the battery-operated LED unit was charged between each exposure. The curing times were half of-, double of- and the time recommended by the manufacturer of each composite resin. During polymerization, the end tip of the LCU was placed over the polyester matrix on top of the stainless steel mould/composite resin set. Right after polymerization, the specimens were placed in plastic containers filled with distilled water and stored at 37°C for 24 hours.

After 24 h of storage, the top surface of each specimen was ground with abrasive paper (LECO Corp., St. Joseph, MI, USA) with increasing grit size (400; 600 and 1,200) to remove the incompletely polymerized oxygen-inhibited layers. The top surfaces were polished using felt wheels (LECO Corp., St. Joseph, MI, USA) with diamond pastes of 3 and 1 micrometer. The specimens were ultrasonically cleaned between polishing felt wheels and for another 20 minutes further. The specimens were stored in distilled water for 24 h at 37° C, until being tested.

Brand Name (Batch #)	Classification	Shade	Composition*	Recommended time	Manufacturer
Filtek Supreme Plus (5028A2D- 7BC)	Nanofilled	A2D	Resin: Bis-GMA, UDMA, TEGDMA, bis- EMA	40 s	3M ESPE, St. Paul, MN, US
			Fillers: SiO <sub>2</sub> nanofiller (20 nm), ZrO <sub>2</sub> /SiO <sub>2</sub> nanoclusters (0.6-1.4 µm size), 78.5% wt, 59% v		
Durafill VS	Microfilled	A2	Resin: UDMA	20	Heraeus Kulzer GmbH, Gruner Weg, Hanau, Germany
(010210)			Fillers: highly disperse SiO <sub>2</sub> (0.02-0.07 μm), splinter polymer (10-20 μm)		
Venus (010139)	Microhybrid	A2	Resin: Bis-GMA, TEGDMA	20	Heraeus Kulzer GmbH, Gruner Weg, Hanau, Germany
			Fillers: Barium Aluminium Fluoride Glass $(0.7 - 2 \ \mu m)$ , highly disperse SiO <sub>2</sub> $(0.04 \ \mu m)$ , 61% v		
Filtek 250 (6020A2-7BP)	Microhybrid	A2	Resin: Bis-GMA, UDMA, bis-EMA	20	3M ESPE, St. Paul, MN, US
			Fillers: ZrO <sub>2</sub> /SiO <sub>2</sub> (0.01-3.5 μm size), 60% v		

#### Table 1. Composition of the materials tested

\*Source: Manufacturer instructions. BisGMA: Bisphenol-A-diglycidyl methacrylate, UDMA: urethane dimethacrylate, TEGDMA: triethylene glycol dimethacrylate, bis-EMA: ethoxylated bisphenol-A. dimethacrylate

# Color Measurement

After 24 h of storage in distilled water, specimens were blot dried and a baseline measurement of the CIE L\*a\*b\* color of the top and bottom surfaces of the specimens was obtained using a reflection spectroradiometer (SpectraScan

PR705 Photo Research, Thermo Oriel Instruments, Stratford, CT, USA) and an external light source with a noncontacting 45-degree illumination and 0-degree observation optical configuration. Before every measurement, a white standard illuminant D65 was used to calibrate the spectroradiometer machine. Specimens were, then, stored in red wine ( $37^{\circ}$ C) and color measurements of both top and bottom surfaces were obtained after 1-, 2-, 7-, 14-, 20- and 30-days in wine. The red wine (Carbenet Sauvignon, Oak Leaf Vineyards, Ripon, CA, USA; Contains sulfites, Alcohol 12.5% by vol., L00183A) with a pH of ~3.23 ± 0.02 was renewed every week. Before any measurement, the specimens were rinsed with water and blot dried.

All measurements were repeated four times against a black background and mean values for the L\*, a\*, and b\* values were calculated. The color change of each specimen was calculated using the CIE 2000 color difference formula (Luo et al., 2001) relative to the color before immersion in wine. The color differences at days 7 to 30 were fit to the following formula which would specify the maximum color change of each composite material at infinite time:

$$\Delta E_{00}^* = \Delta E_{\max}^* \left( 1 - \exp\left[\frac{-D}{\tau}\right] \right)$$

where  $\Delta E_{00}^*$  is the CIE 2000 color difference,  $\Delta E_{max}^*$  is the value that  $\Delta E_{00}^*$  would approach if left in the wine for a long time, D is the number of days in the wine, and  $\tau$  is the time constant in days which describes how fast the color change occurs.

## Statistical Analysis

Statistical analysis was performed using Repeated Measures ANOVA and stepdown Bonferroni correction on t-test probability for non related pairs and Bonferroni correction for related pairs at significant level of 5%.

#### Results

For all three color directions (L\*, a\*, and b\*) and for the color change following wine submersion, a quadruple interaction (Resin X Light X Surface X Days; p = 0.042) was observed.

Although there was found a significant effect (P = 0.042) on the color change for the interaction between the LCU, Resin, Time in the wine and Surface, no statistically significant difference in the average color difference after wine submersion could be observed between the two LCUs for any one combination of Resin, Time in the wine and Surface studied (Fig. 1-4). Differences between the  $\Delta E^*$ max from the top and the bottom surfaces could be observed for all light curing times and composites resins, except for Venus when light cured for double of the recommended time (Fig.3). In general,  $\Delta E^*$ max from the bottom surface was higher than from the top surface.

#### Days in red wine comparisons (D1, D2, $\Delta E^*$ max)

The composite resins Durafill and Z250 showed significant differences between the maximum color change ( $\Delta E^*max$ ) and the color change from Day 1 and Day 2 for all light curing times and for both surfaces and LCUs. When looking at the Supreme group, the difference between  $\Delta E^*max$  and Day 1 and Day 2 could only be observed when LED was used, regardless of the light curing time; and when HAL was used for 20 s (half of the recommended time) (Fig.1). When HAL was used with the recommended and double of the time, 40 and 80 s respectively, no difference could be observed between the maximum color change and color change from Day 1 and Day 2 at the top surface.

Similar result was observed on the Venus group when looking at the top surface that was light cured with HAL for half of (10 s) and with LED for half of (10 s) and double of (40 s) the recommended time. However, when light cured for 20 (recommended time) and 40 s using the HAL LCU and for 20 s using LED LCU, significant difference could be observed between  $\Delta E^*$ max and Day 1 and Day 2 for both top and bottom surfaces of Venus (Fig.3).





Figure 2. Mean color change ( $\Delta$ E2000) from Day 1, Day 2 and  $\Delta$ E\*Max of Durafill.



Figure 3. Mean color change (ΔΕ2000) from Day 1, Day 2 and ΔΕ\*Max of Venus.



Figure 4. Mean color change ( $\Delta$ E2000) from Day 1, Day 2 and  $\Delta$ E\*Max of Z250.

#### Composite resins comparisons

Taking into consideration the color change from Day 1 and Day 2, significant differences among the different composite resins could only be observed at the bottom surface. However, no differences between Durafill and Venus; between Durafill and Z250; and between Supreme and Z250 were observed for either LCUs or light curing times.

When  $\Delta E^*$ max is taken into account, significant differences among all composite resins can be observed at both top and bottom surfaces. Nevertheless, differences between Supreme and Z250 are only observed at the bottom surface for both LCU and with all light curing times; Supreme showed higher values of  $\Delta E^*$ max. The same occurred for Durafill and Z250 when HAL was used. In this case, Z250 had shown higher values of  $\Delta E^*$ max.

Significant difference between the color change from Day 1 and Day 2 are only observed at the bottom surface of Supreme composite resin light cured with HAL for half of the recommended time (20 s) (Fig. 1). Supreme is also the only composite resin that had shown significant differences between the color change of the top and of the bottom surfaces at Day 1 and Day 2 with both LCUs and with all light curing times. This difference between the color change of top and bottom surfaces could also be observed with Z250 light cured with HAL and with LED for 10 s (half the recommended time) at Day 1 and Day 2, respectively; and with HAL for 10, 20 and 40 s at Day 2 (Fig. 4).

## Light curing exposure times comparisons

Significant differences among the light curing times were only observed for Supreme and Venus at the bottom surface and at the maximum color change ( $\Delta$ E\*max). When Supreme was light cured for double of the time recommended by the manufacturer compared to half of the time, less color change was observed at the bottom surface, regardless of the LCU used (Fig.1). The same as for Venus when light cured with LED. However, when Venus was light cured with HAL, less color change was observed for double of the recommended time compared to the time recommended by the manufacturer (Fig. 3).

#### Discussion

The effects of LCUs and different light curing times on composites discolored by red wine were evaluated in the present study. Even though, LCUs and light curing times play a significant role on composites final properties and longevity, many other factors, such as resin composition, are similarly important to describe the behavior of composite restorations under a staining regimen (Topcu et al., 2009).

The possibility of comparing the color change of both top and bottom surfaces of the composites specimens may sound clinically irrelevant in some aspects, as usually the bottom surface may be directly in contact with the dental surface and indirectly protected from the oral fluids. However, the discoloration of the restorations' margins, especially at the gingival margin of Class II restorations, may be due to an inadequate polymerization of the bottom surface of the first increments of the composite material. Therefore, the main reason for comparing the color change between the top and bottom surfaces was to be able to indirectly visualize the difference on the depth of cure between the two surfaces. It is expected that the bottom surface is less polymerized than the top surface of a composite restoration because of the distance between the end of the light curing tip and the composite increment (Ernst et al, 2004), as well as, because of the attenuation of light intensity across the composite mass (Rueggeberg et al., 1994). Therefore the bottom surface would be more prone to staining than the top surface. In fact, the results show that the maximum color change ( $\Delta E^*$ max) of the bottom surfaces was significant higher than of the top surfaces for all composite resins and all light curing times, except for Venus when light cured for double of the time recommended by the manufacturer. This result shows that even light curing a composite increment for double of the time recommended by the manufacturer, this may be insufficient to prevent a higher discoloration of the bottom surface compared to the top surface of composite restorations. Subsequently, other mechanical properties, such as hardness and flexural strength, may be impaired as well. Additionally, a longer exposure time could have a negative effect over the pulpal condition due to the rising of temperature during the light curing of composite material (Duray et al., 2008). Consequently, the first composite increment should be less than 2 mm to be sufficiently cured, especially in deep cavities such as Class II where the bottom surface of the restoration is directly in contact with oral fluids.

A less polymerized composite surface may be more prone to the effect of the low pH and high water and ethanol content from the wine solution (Vandewalle et al., 2004). The ethanol and water are considered organic solvents that can soften the resinous matrix of composite materials, contributing to the penetration within the composite body of the pigments present at the wine. In some aspect, Venus may have been benefited from a longer exposure time. When light cured for 40 s with either LCU, no significant difference in the  $\Delta E^*$ max could be observed between the top and the bottom surfaces. The longer exposure time may have contributed to improve the degree of polymerization of the bottom surface, rendering a more homogeneous composite restoration; however, this should be proven by other test methods such as hardness or transformation infrared spectroscopy.

The composite resins used in this study were selected according to their chemical composition (monomer content) and type and size of filler particles. One type of nanofilled and of microfilled composite resins, and two types of microhybrids resins were selected. The exact composition of those composite resins is not known, as the information is not fully provided by manufacturers. Because of its less hydrophobic nature, the TEGDMA present in the composition of Supreme may be responsible for a higher water intake, and consequently discoloration of this composite material, especially at its bottom surface (Peutzfeldt, 1997; Rawls & Esquivel-Upshaw, 2005; Güler et al., 2009). The higher resin matrix content of microfilled composite materials is another fact that may contribute to a more liquid sorption from the environment by this type of composite resin, although UDMA, present in the composition of Durafill, is considered a hydrophobic monomer (Peutzfeldt, 1997). One limitation from the present study may be related
to the higher amount of organic matrix that may have been accumulated at the bottom surfaces of the composite specimens, what may also have contributed to a higher discoloration of these surfaces.

Studies have shown that a  $\Delta E < 1.0$  is not visually perceptible by the human vision, however a  $1.0 \le \Delta E \le 3.0$  although is visually perceptible, it is considered acceptable. On the other hand, a  $\Delta E > 3.3$  is visually perceptible and not acceptable, it demands the restoration substitution (Ruyter et al., 1987; Kolbeck et al., 2006). Those values are based on the CIELAB of 1976. For the CIELAB of 2000, the predicted values of  $\Delta E00$  would be equal to 0.93 and 2.88 for a corresponded value of  $\Delta E76$  equals to 1.0 and 3.3, respectively. In the present study, when considering the top surface of composite samples at Day 1, all  $\Delta E00$  values were below 3.3, however they were always above 1.0. This shows that the color changes were already visually perceptible by the human vision. This fact shows how a staining solution like red wine can affect the composite surface and rapidly discolor it after only 1 day of storage. Although this experiment does not well simulate a clinical situation, it shows the high susceptibility of composite materials to water sorption in the early stages after polymerization.

The color data from days 7, 14, 20 and 30 enabled the construction of a formula to calculate the maximum color change at an infinite time, which was called  $\Delta E^*$ max. The values of  $\Delta E^*$ max would represent the final color change of either top or bottom surface of each composite material that was light cured for half, double or the recommended time by the manufacturer. Significant differences among the light curing times were only observed at the bottom surfaces on two types of composite materials, Supreme and Venus. A longer exposure time resulted in a composite less susceptible to color change at the bottom surface, in other words, more resistant to liquid sorption; although many other factors may contribute to this phenomenon, such as the material's chemical composition (Dietschi et al., 1994; Kolbeck et al, 2006). Nevertheless, the  $\Delta E^*$ max values ranged from ~ 7.0 to 25, which are extremely higher than the acceptable value of below 2.88.

At the top surface,  $\Delta E^*$ max ranged from ~ 3.0 to 12. Supreme was the only composite that kept the mean value of  $\Delta E^*$ max below 3.3 when light cured with HAL for double or for the recommended time by the manufacturer. The other composites showed mean values above 3.3. Therefore, the maximum color change of those materials would always be above from what is considered acceptable by the human vision. In a real clinical situation, the substitution of those composites restorations would have happen before they reach  $\Delta E^*$ max.

The improved behavior of Supreme regarding its top surface could not be observed at the bottom surface. At the bottom surface  $\Delta E^*$ max ranged from ~ 12 to 25, what was significantly higher than the values obtained for the other composites. This significant difference between the  $\Delta E^*$ max of the top and the bottom surface of Supreme may be explained by the fact that this composite was initially more opaque than the other composites used in this study, as its color was A2D, which means that it is recommended to restore the dentin layer of the restoration. The higher opacity of this composite may have prevented the light transmission through the composite body, affecting the polymerization of its bottom surface, turning it more prone to water sorption and consequently, to discoloration compared with the other composites (Christensen et al., 1999).

Taking into account all composite materials tested in this study, Venus was the one that has shown the lowest values for  $\Delta E^*$ max for both top and bottom surfaces. Its chemical composition may be responsible for a composite more resistant to water sorption, and therefore, to discoloration over time. However, for both surfaces,  $\Delta E^*$ max mean values were higher than 2.88.

The proper polymerization of composite restoration is still a concern in the dental practice. Although polymerization plays an important role on the final properties of the composite restorations; the longevity of the restorations may be also related to the chemical composition of the composite material when considering water sorption and consequently, discoloration. Therefore, based on the results found in the present study, the hypotheses were accepted; although many variables interfere with the color stability of the final restoration, and those should be taken into consideration when performing a composite filling.

# Conclusions

The effects of changes in curing time on resin discoloration by red wine vary significantly by the type (composition) of composite resin being stained and the surface being evaluated (top or bottom). The lightcuring for double of the recommended time seems to influence on the degree of color change of the bottom surface of some composite materials. Regarding the magnitude of the resulting color change, the LCU had a significant interaction with the type of composite resin, the surface to be evaluated and the days stored in red wine.

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# **CAPÍTULO 2**

# EFFECT OF ALCOHOLIC STAINING DRINK ON THE PROPERTIES OF COMPOSITES LIGHT CURED WITH TWO LIGHT CURING UNITS AND DIFFERENT EXPOSURE TIMES

**Objectives:** To evaluate the performance of two light curing units (LCUs) and three different exposure times on the degree of conversion of different types of composite resins, as well as the effect of red wine over the hardness and the surface roughness of those composite materials after a period of 30 days of immersion.

**Material and Methods:** Composite discs 2 mm thick x 5 mm diameter were fabricated from five composite resins: Filtek Supreme Plus A2D and Filtek Supreme XT A2E (3M ESPE), Durafill A2 (Heraeus Kulzer), Filtek Z250 A2 (3M ESPE) and Venus A2 (Heraeus Kulzer). The LCUs used were Halogen – Optilux 501 (Demetron) and Blue LED – Elipar FreeLight 2 (3M ESPE). The light curing times were half of, full of and double of the time recommended by the manufacturers. After 24 h in distilled water at 37°C, the specimens were polished and after desiccation, the degree of conversion (DC) was established with ATR-FTIR. Knoop microhardness (KHN) and surface roughness (Ra) were obtained before and after 30 days of red wine storage. The red wine was renewed every week and its pH was ~3.65. Statistical analysis was performed using Repeated Measures ANOVA/ stepdown Bonferroni for non related pairs and Bonferroni correction for related pairs (p < 0.05).

**Results:** For the DC, KHN and Ra, each of the factors studied (Resin, LCU, Cure Time, Wine Exposure and Surface) was a statistically significant factor (p < 0.05) either overall or in an interaction with one or more other factors. The roughness of composite surfaces was not significantly affected after the storage in the wine, except of the bottom surface of Durafill. The hardness of the top surface of composite specimens was significantly reduced after the wine storage, except

for Venus and Durafill. The hardness of the top surface was higher than the bottom surface, and significant difference was found between the DC of the surfaces.

**Conclusions:** The two types of LCUs, HAL and LED, did not significantly differ from one another in their general performance. Significant differences were found for specific composite materials and exposure times. In general, the light exposure for double of the recommended time did not improve the performance of the composite materials compared to the time recommended by the manufacturer.

**Key Words:** Composite resins; Light curing units; Microhardness; Roughness; Degree of conversion

# Introduction

The degree of cure of a light cured composite material may be affected by the type of light curing unit (LCU), by the exposure time, by the light intensity, by the type of composite material, by the size and volume of inorganic particles, by the nature of composite matrix, by the type of photo initiator system, by the color of the composite material, by the distance of the end of the light curing tip to the composite increment, by the thickness of the composite increment, and by the spectral wavelength emitted by the LCU (Rueggeberg & Jordan, 1993; Rueggeberg et al., 1994; Christensen et al., 1999; Price et al, 2000; Gagliani et al, 2002; Yoon et al, 2002). By negatively affecting the degree of cure, the performance and longevity of a composite restoration can be highly impaired.

An insufficient polymerized composite material may present unsuitable mechanical and physical properties (Yoon et al., 2002; Price et al., 2003; Vandewalle et al., 2004). Moreover, the composite restoration may become more prone to the action of oral fluids, such as water and organic solvents that can degrade the resin matrix and the silane bonding responsible for coupling the inorganic fillers to the resin matrix (Larsen & Munksgaard, 1991; Peutzfeldt, 1997; Vandewalle et al., 2004). An early debonding or fracture of the dental composite restoration, as well as, discoloration over the time are some of the factors

responsible for the early substitution of the composite restoration. In this manner, the establishment of an ideal LCU and exposure time is still a concern among the dental community.

High intensity LCUs are usually preferable to light cure dental composites, as they emit higher amount of photons per second per unit area. In this way, a greater amount of camphorquinone molecules can be reached and become ready for reaction (Cobb et al., 1996; Small, 2001). However, the light exposure time should be sufficient so that the reaction can properly occur. Therefore, the energy density, which corresponds to the product of light intensity and light exposure time, is somewhat more important than the light intensity by itself (Vandewalle et al., 2004).

The microhardness test is a very simple method to indirectly measure the degree of conversion of composite materials (Yearn, 1985). At the same time, it can predict an important mechanical property of those restorative materials. If the hardness of a composite material is rapidly affected after the immersion of the material in any type of solution, it can be inferred that this composite material is not properly cured. Also, the surface roughness is another method that shows the smoothness of a composite surface before and after the action of a liquid solution or finishing and polishing techniques (Yap et al., 2000). Changes in the surface roughness by a liquid solution may be due to effects over the resinous matrix and filler particles, what can be interpreted as a type of degradation of the composite material. A rougher surface may be responsible for the accumulation of plaque and pigments from the oral environment (Bollen et al., 1997), leading to discoloration of the composite restoration overtime (Dietschi et al., 1994; Sarac et al., 2006).

Subsequently, the aims of the present study were to evaluate the performance of two light curing units (LCUs) and three different exposure times (half of-; double of-; and the time recommended by the manufacturer) on the degree of conversion of different types of composite resins, as well as the effect of red wine over the hardness and the surface roughness of those composite materials after a period of 30 days of immersion. A correlation test between the

degree of conversion and microhardness tests was executed. The hypotheses were that both LCUs have similar performance and that a longer exposure time renders a composite more resistant to the water/ethanol degradation.

# Material and Methods

# Specimens Fabrication

Hundred and fifty disk specimens (n = 5) of composite resins were fabricated using a stainless steel metallic mould of 2 mm of height and 5 mm of diameter. A polyester matrix was placed on the bottom and top surfaces of the stainless steel mould, before and after the placement of the composite materials, respectively. A 500 g load was placed on top of the polyester matrix, composite material and stainless steel mould set for 30 seconds to guarantee the proper accommodation of the composite resin prior to polymerization. The type, color, composition, and light curing time recommended by the manufacturer for each composite material used in this study are shown on Table 1.

The composite disks were lightcured for different times using a conventional quartz-tungsten-halogen (HAL) light (Optilux 501, Demetron/Kerr Corp., Orange, CA, USA) and a second-generation blue light emitting diode (LED) (Elipar FreeLight 2, 3M ESPE, Seefeld, Germany). Light intensity measurements of both LCUs were made periodically throughout the specimens' fabrication (HAL: 890 mw/cm<sup>2</sup>; LED: 1250 mw/cm<sup>2</sup>), and the battery-operated LED unit was charged between each exposure. The cure times were based on half of-, double of- and the time recommended by the manufacturer's instructions of each composite type. During polymerization, the end of the light curing tip was placed over the polyester matrix on top of the stainless steel mould/composite resin set. Right after polymerization, the specimens were placed in plastic containers filled with distilled water and stored at 37°C.

Brand Name (Batch #)	Classification	Shade	Composition	Recommended time	Manufacturer
Filtek Supreme Plus (SD)	Nanofilled	A2D	Resin: Bis-GMA, UDMA, TEGDMA, bis-EMA	40 s	3M ESPE, St. Paul, MN, US
(5028A2D- 7BC)			Fillers: SiO <sub>2</sub> nanofiller (20 nm), $ZrO_2/SiO_2$ nanoclusters (0.6- 1.4 µm size), 78.5% wt, 59% v		
Filtek Supreme XT (SE)	Nanofilled	A2E	Resin: Bis-GMA, UDMA, TEGDMA, bis-EMA	20 s	3M ESPE, St. Paul, MN, US
(5028A2D- 7BC)			Fillers: SiO <sub>2</sub> nanofiller (20 nm), $ZrO_2/SiO_2$ nanoclusters (0.6- 1.4 µm size), 78.5% wt, 59% v		
Durafill VS	Microfilled	A2	Resin: UDMA	20 s	Heraeus Kulzer GmbH, Gruner Weg, Hanau, Germany
(010210)			Fillers: highly disperse SiO <sub>2</sub> (0.02-0.07 μm), splinter polymer (10-20 μm)		
Venus (010139)	Microhybrid	A2	Resin: Bis-GMA, TEGDMA	20 s	Heraeus Kulzer GmbH, Gruner Weg, Hanau, Germany
			Fillers: Barium Aluminium Fluoride Glass (0.7 – 2 µm), highly disperse SiO <sub>2</sub> (0.04 µm), 61% v		
Filtek 250 (6020A2-7BP)	Microhybrid	A2	Resin: Bis-GMA, UDMA, bis-EMA	20 s	3M ESPE, St. Paul, MN, US
			Fillers: ZrO <sub>2</sub> /SiO <sub>2</sub> (0.01-3.5 µm size), 60% v		

# Table 1. Composition of the materials tested

BisGMA: Bisphenol-A-diglycidyl methacrylate, UDMA: urethane dimethacrylate, TEGDMA: triethylene glycol dimethacrylate, bis-EMA: ethoxylated bisphenol-A. dimethacrylate

After 24 h of polymerization, the top surface of each specimen was ground with abrasive paper (LECO Corp., St. Joseph, MI, USA) with increasing grit size (400; 600 and 1,200) to remove the incompletely polymerized oxygen-inhibited layers. The top surfaces were polished using felt wheels (LECO Corp., St. Joseph, MI, USA) with diamond pastes of 3 and 1 micrometer. The specimens were ultrasonically cleaned between polishing felt wheels and further for 20 minutes.

#### Degree of Conversion

Before the establishment of the degree of conversion from the polymerized specimens, they were subjected to desiccation with silica pellets at a hot chamber at 37°C for 24 h to completely remove a ny remaining water from the composite body that could interfere with the reading from the spectrometer. The infrared spectra of the top and bottom surfaces of each of five specimens randomly selected from the experimental groups were obtained. The flat surface (top and bottom) of each specimen was pressed against the surface of a horizontal diamond in an attenuated total reflectance attachment (Universal ATR Sampling Acessory, Perkin Elmer do Brasil, São Paulo, SP, Brazil) placed with the optical bench of a Fourier transform infrared spectrometer (Spectrum 100, FTIR Spectrometer, Perkin Elmer do Brasil, São Paulo, SP, Brazil). A total of 32 spectra were obtained and averaged to derive a single specimen profile at a resolution of 4 cm<sup>-1</sup>. Spectra of the uncured composite were obtained by pressing a small amount of paste directly onto the diamond surface. The degree of conversion of each specimen was determined by changes in the ratio of aliphatic C=C (peak at 1638cm<sup>-1</sup>) to aromatic C-C (peak at 1608cm<sup>-1</sup>) in the cured and uncured states. The percent of monomeric aliphatic C=C bonds converted into C-C bonds was thus determined (degree of conversion) using the following formula:

 $DC = 1 - [(C=C/C-C \text{ cured})/(C=C/C-C \text{ uncured})] \times 100$ 

After determination of the degree of conversion, the composite specimens were stored in distilled water at 37°C for 24 h before the determination of microhardness and surface roughness.

# Knoop Microhardness

The hardness of individual composite specimens was determined with a Knoop indenter microhardness tester (Microhardness Tester, Future Tech FM-1E, Tokyo, Japan). A 25 g load was applied through the indenter with a dwell time of 20s to obtain the Knoop Hardness Number (KHN). Three readings were taken for each specimen on half of the top and of the bottom surfaces (Fig. 1); before and after specimens' storage in red wine ( $37^{\circ}$ C) for 30 days. The wine solution (Carbenet Sauvignon, Oak Leaf Vineyards, Ripon, CA, USA; Contains sulfites, Alcohol 12.5% by vol.) was changed every week and the pH was ~ 3.65. The larger diagonal length of indentation was measured with a monitor (9M 100A Teli, Tokyo, Japan), and an average value in µm was calculated to obtain a single value for top and bottom surface of each specimen. Subsequently, the KHN was calculated.

# Surface Roughness

The surface roughness (Ra) was determined with a profilometer (Surfcorder SE 1000, Kosaka Corp., Tokyo, Japan) at a 0,1 mm/s speed, 1.25 mm length and 0.25 mm cut-off. Three readings were taken for each specimen on the other half of the top and of the bottom surfaces (Fig. 1); before and after specimens' storage in the red wine for 30 days. An average value was calculated to obtain a single value for both top and bottom surfaces of each specimen.



**Figure 1.** A - Illustration of surface roughness test. B - Illustration of Knoop microhardness test. C – Localization of the microhardness test indentations on half of the top or bottom surface and illustration of the directions for the surface roughness test on the other half of the surface.

# Statistical Analysis

Statistical analysis was performed using Repeated Measures ANOVA and stepdown Bonferroni correction on t-test probability for non related pairs and Bonferroni correction for related pairs at significant level of 5%. Pearson's correlation test was performed between the degree of conversion and microhardness tests to verify the degree of the relationship between the linear related variables. The KHN values used for the correlation test were from before the wine storage.

#### Results

# Degree of Conversion

For the degree of conversion (DC), triple interactions (Light X Resin X Time; p = 0.05; Light X Resin X Surface; p = 0.02) were observed. Figure 2 shows the DC for all composite resins, light curing times and LCUs.

Statistically significant differences between the two LCUs could only be observed at the top surface of SE when lightcured for double of the recommended time. In this situation, the DC with HAL was significantly higher than with LED. The DC of the top surface compared to the bottom surface of SD and Venus was significantly higher, regardless of the exposure time and LCU used. Similar result was observed for SE, except when LED was used for double of the recommended time, where no significant difference could be observed between the DC of the top and of the bottom surfaces. When Z250 was lightcured for double of the time with either LCU, no significant difference was observed in the DC between the two surfaces. Nonetheless, when Z250 was lightcured for half of- and for the recommended time using HAL or LED, the top surface has showed a higher DC than the bottom surface (p < 0.05). Durafill was the only composite resin that no significant difference could be observed between the DC of the top and of the bottom surfaces of the amount of exposure time and LCU used.

Statistically significant differences in the DC among the different exposure times were only observed at the bottom surfaces of SD and Venus when lightcured with HAL; and of Durafill when lightcured with LED. For SE, the differences were observed at both the top and the bottom surfaces when HAL was used. The composite resin Z250 was the only one that did not show significant differences among the exposure times with either LCU. Significant differences were only observed between half of- and double of the recommended time. The DC of composites light cured for half of the recommended time was significantly less than that light cured for double of the time.

Differences in DC among the different types of composite resins were only observed between Durafill and the other composites at some specific surfaces, exposure times and LCUs. Significant differences were found at the top surfaces of Durafill and SD; Durafill and Venus; and Durafill and Z250 when lightcured with LED, regardless of the amount of exposure time. However, when HAL was used for the recommended time, significant differences were only observed at the top surfaces of Durafill and Venus; and of Durafill and Z250. Significant differences were also observed in the DC at the bottom surface of Durafill and SD when HAL was used for half of the time; and when LED was used for double of the time. Significant differences between Durafill and SE were observed at the top surface when LED was used for the recommended time and HAL was used for double of the time. At the bottom surface, significant differences were observed when LED was used for double of the recommended time. In all those situations, Durafill has always shown lower values of DC compared to the other types of composite resins.

Bot Top

LED

2

Bot Top

LED

2



**Figure 2.** Degree of conversion of the top and bottom surfaces for each type of composite resin, light curing time and LCU. Surface: Bot: Bottom; Cure Time: 0.5:Half; 1: Recommended; 2: Double.

2

1

0.5

1

2

Cure Time:

0.5

# Knoop Microhardness

For KHN, triple interactions (Light X Resin X Surface; p = 0.05; Light X Resin X Wine; p < 0.0001; Resin X Time X Surface; p = 0.008) were observed. Figure 3 shows the KHN for all composite resins, light curing times and LCUs before and after the storage in the wine.

No statistically significant differences in the KHN values could be observed between the two LCUs and among the exposure times for the different types of composite resins. Venus and SE have shown significant differences in the KHN between the two surfaces. As much as before or after the immersion in the wine, the top surface of Venus and SE had higher KHN than the bottom surface, regardless of the LCU used and of the amount of exposure time. For the other composites tested, the difference between the KHN from the top and the bottom surfaces varied according to the amount of exposure time and to the LCU used. Durafill was the only composite resin that did not show significant differences between the KHN from the top and the bottom surfaces after the wine storage for either LCU or exposure time.

Significant differences between the KHN before and after the red wine storage were only observed at the top surface, varying according to the LCU, to the type of composite resin and to the amount of exposure time. It was observed that after the wine storage for 30 days, the KHN of the top surfaces have diminished. For SE and Z250, this was observed for all exposure times when SE was lightcured with LED and Z250 with HAL. When Z250 was lightcured with LED, significant differences in the KHN between before and after the wine storage were only observed when double of the recommended time was used. For SD, significant differences were observed when lightcured for 80 s with HAL and for 20 s with LED. Venus and Durafill did not show any significant difference between the KHN before and after the storage in the wine, for either LCU, either surface or exposure time.

#### Capítulo 2



**Figure3.** Average of KHN of the top and bottom surfaces for each type of composite resin, light curing time and LCU before and after wine storage. Surface: Bot: Bottom; Cure Time: 0.5:Half; 1: Recommended; 2: Double.

The overall correlation test pointed out a medium correlation (r = 0.53) between the degree of conversion and microhardness test. When the DC and KNH values for each composite resin are individually correlated, a higher correlation was observed for SD (HAL: r = 0.85; LED: r = 0.89), SE (HAL: r = 0.81; LED: r = 0.86), Venus (HAL: r = 0.84; LED: r = 0.85) and Z250 (HAL: r = 0.80; LED: r = 0.74). Durafill was the only composite resin that has shown a low correlation between DC and KHN values (HAL: r = 0.44; LED: r = -0.24).

# Surface Roughness

For Ra, five triple interactions (Light X Resin X Surface; p = 0.045; Light X Time X Wine; p = 0.0008; Resin X Time X Surface; p < 0.0001; Resin X Time X Wine; p = 0.047; Resin X Wine X Surface; p = 0.022) were observed. Figure 4 shows the Ra for all composite resins, light curing times and LCUs before and after the storage in the wine.

No statistically significant difference in the Ra values could be observed between the two LCUs for the different exposure times and composite resins. Significant differences in Ra between the top and the bottom surfaces were only observed with Durafill after the wine storage when lightcured with either LCU, except when LED was used with the time recommended by the manufacturer. The surface roughness of the bottom surface of Durafill was significantly higher than the top surface after 30 days in the wine. Before the wine storage, Ra values for the bottom surface of Durafill were statistically higher than the top surface only when HAL and LED were used for half of the recommended time. The other types of composite resins did not show significant differences in Ra between the top and the bottom surfaces, either before or after the storage in the wine.

Significant differences in Ra for the different exposure times could only be observed at the bottom surface of Durafill, regardless of the type of LCU used. Before the wine storage, significant differences were observed between half of and the other exposures times when HAL was used. On the other hand, after the wine storage, significant differences were observed between half of and the other exposure times when LED was used.















**Figure 4.** Average of Ra of the top and bottom surfaces for each type of composite resin, light curing time and LCU before and after wine storage. Surface: Bot: Bottom; Cure Time: 0.5:Half; 1: Recommended; 2: Double.

In general, the composite resins did not show statistically significant differences in Ra before and after the storage in the wine. Durafill was the only composite that has shown increased values of Ra at the bottom surface after the storage in the wine when light cured with LED for half of the time and with HAL for the time recommended by the manufacturer.

### Discussion

In the present study, the effect of an alcoholic staining drink solution over different types of composite materials light cured with two types of LCUs and different exposure times was evaluated through the hardness and surface roughness of both the top and the bottom surfaces of composite specimens. It is expected that a less polymerized composite surface would be more susceptible to the water and organic solvents degradation (Larsen & Munksgaard, 1991; Ferracane, 1994; Vandewalle et al., 2004; Yap et al., 2004).

The red wine solution was chosen as it combines the presence of both water and an organic solvent, such as alcohol, in an acidic pH. Studies have shown that from all staining solutions, such as coffee, whisky, cola drinks, black tea and others; the wine is the most discoloring one (Patel et al., 2004; Kolbeck et al., 2006). If the wine is able to negatively affect the color of composite materials overtime, then it could negatively affect other aspects of the composite material, such as the hardness and the surface roughness.

Rougher composite surfaces are usually more prone to discoloration overtime, due to the accumulation of plaque and pigments from the diet (Dietschi et al., 1994; Sarac et al., 2006). The surface roughness of a composite material is related to the size and type of inorganic fillers; and to the finishing and polishing process after an adequate polymerization of the composite resin (Sarac et al., 2006). Nonetheless, in the present study, the Durafill composite resin, a microfilled resin, had shown higher values of surface roughness either before or after the storage in the wine compared to the other types of composite materials, especially at the bottom surface. This may be due to the low hardness and the high amount of organic matrix related to this composite. The other composite materials, nanofilled and microhybrid ones, did not statistically differ in their surface roughness. Usually, nanofilled composite materials show a smoother surface compared to microhybrid composites; as well as, the microfilled composites when compared to microhybrid ones (Sarac et al., 2006).

Also, the Durafill was the only composite material in which the surface roughness was affected by the wine solution. The other composite materials did not have too much influence of the wine solution over the surface roughness. It seems that the wine may act directly over the resin matrix changing the color of the composite material. The pigment from the wine may entrap in the resin matrix through the absorption of water from the environment; and not only because the wine can turn the composite surface rougher (Kolbeck et al., 2006). Also, the low pH from the wine may have contributed to turn the surface of Durafill rougher as this is a composite with very fine inorganic particles.

Hard surfaces with Ra values above 0.2 µm are more susceptible to bacterial colonization (Bollen et al., 1997). In the present study, the microhybrid and nanofilled composites kept the Ra values of both top and bottom surfaces below 0.2 µm even after the wine storage. Nonetheless, Durafill has shown Ra values above 0.2 µm for the bottom surface when lightcured for half of the time recommended with either HAL or LED. This may show the low hardness of the bottom surface of this type of composite resin when lightcured for half of the time recommended by the manufacturer. Although no significant difference has been found between the KHN values from the bottom and top surfaces of Durafill after the wine storage, the surface roughness of the bottom surface was hardly affected after the staining regimen. This result shows that the properties of composite materials cannot be independently analyzed.

The differences found in the hardness, degree of conversion and surface roughness among the different types of composite materials may be also related to their chemical composition, translucency/opacity and filler distribution (Imazato et al., 2001; Schulze et al, 2003; Silva et al., 2008 (a); Silva et al., 2008 (b)). Durafill was the only composite resin that did not show significant difference between the DC from the top and the bottom surfaces, this may be due to the translucency of this composite material that lets the transmission of the light through its whole body. However, from all composite materials, Durafill has shown the smallest values of hardness. As this is a microfilled composite, it is recommended for anterior restorations where less mechanical and physical properties are required compared to the posterior region (Raskin et al., 1999).

It is expected that as higher as the degree of conversion of a composite material, the higher would be its mechanical and physical properties, such as the hardness (Ferracane, 1985; Millar & Nicholson, 2001; Asmussen & Peutzfeldt, 2002). In the same way, it is expected that as longer as the exposure time, the higher would be the degree of conversion of a composite material and consequently, its final properties (Matsumoto et al., 1986; Bennett & Watts, 2004). Nonetheless, the longer exposure time used in the present study (double of the time recommended by the manufacturer) did not render a composite with higher degree of conversion neither with a higher hardness when compared to the time recommended by the composite materials (Rawls & Esquivel-Upshaw, 2005). Under regular pressure, temperature and atmosphere, composite resins are unable to reach higher degree of conversion values (up than 70%) even when light cured for double of the recommended time.

Moreover, significant differences were found in the hardness between the composites' top and bottom surfaces. Usually, the top surfaces had higher hardness when compared to the bottom surfaces. As the light is transmitted through the composite mass, part is reflected, part is absorbed and another part is scattered, and what really reaches the deepest portion of the composite material has reduced intensity when compared to the top surface where the light intensity is in its full state (Rueggeberg et al., 1994; Correr Sobrinho et al., 2000; Vandewalle et al., 2004). Although the top surface had higher hardness than the bottom surface, after the composite materials had been stored in the wine solution for 30 days, the hardness from the top surfaces was significantly reduced. Still, the hardness from the top surfaces was significantly higher than the bottom surfaces, even after the wine storage.

Significant differences were found for the degree of conversion between the top and the bottom surfaces of the composite specimens, additionally significant differences had been found between the hardness of both surfaces. As already mentioned in prior studies (Vandewalle et al., 2004; Price et al., 2003), the microhardness can be an indirect method for measuring the degree of conversion of a composite material; however, the hardness value cannot be used to predict the degree of conversion of a polymeric material (Ferracane, 1985). Still, the degree of conversion, measured by the percentage of monomers that can be converted to polymers does not mean that the polymer formed has enough amount of cross links between the polymer chains (Yap et al., 2004). The cross link density of a composite material can be indirectly measured through its hardness; therefore, it can be suspected that although the top surface had received a higher light intensity during polymerization compared to the bottom surface, the polymerization reaction may have occurred so fast that probably not enough cross links were formed (Soh & Yap, 2004). In this manner, the top surface was more susceptible to the action of water and alcohol from the wine solution and as a result, the hardness from the composites' top surfaces has diminished after the storage in the wine solution.

The only composites that showed a higher degree of conversion of the bottom surface after lightcured with double of the time recommended by the manufacturer were SE and Z250. No significant difference between the degree of conversion of the top and the bottom surfaces could be observed for these composites when lightcured for double of the time. However, the hardness from the top surfaces was still higher than the bottom surfaces. Again, although a positive correlation is found between the DC and KHN (Yap et al., 2004), they cannot be overestimated from one another. In the present study, a medium overall correlation was observed (r = 0.53); however, for all composite resins tested except for Durafill

a higher correlation was observed (above 0.70). The low correlation observed for Durafill (below 0.45) may be due to the low hardness observed for this type of composite resin compared to the other composite resins tested in this study.

The two types of LCU, HAL and LED, did not significantly differ from one another in their general performance. Significant differences were found for specific composite materials and exposure times. Moreover, in general, double of the exposure time did significantly differ from half of the time recommended by the manufacturer. However, it did not significantly differ from the time recommended by the manufacturer. The time recommended by the manufacturer was an intermediate exposure time between the double of- and the half of the recommended time, what sounds reasonable as it is what the manufacturers recommend. Therefore, based on the results found in the present study, the hypotheses were accepted; although many variables interfere with the performance of the final restoration, and those should be taken into consideration when performing a composite filling.

# Conclusions

The two types of LCU, HAL and LED, did not significantly differ from one another in their general performance. The alcohol staining drink most affected the hardness of the top surfaces of composite materials; however the roughness was not significantly affected for most of the composite resins, except for the bottom surface of Durafill. In general, the light exposure for double of the recommended time did not improve the performance of the composite material compared to the time recommended by the manufacturer. Light curing the composite material for half of the recommended time not always renders a composite restoration with the same properties as when light curing with the time recommended by the manufacturers.

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# CONSIDERAÇÕES GERAIS

Neste estudo, a influência do tipo de fonte de luz e tempo de fotoativação sobre a pigmentação de diferentes tipos de resinas compostas foi avaliada de diferentes formas. Primeiramente, uma análise espectroradiométrica da alteração de cor dos diferentes tipos de materiais resinosos após imersão em vinho tinto foi realizada. A fotopolimerização destes materiais foi executada com as fontes de luz LED e Halógena pela metade do tempo, pelo dobro do tempo e pelo tempo recomendado pelos fabricantes dos materiais resinosos. Nesta análise, foi possível prever o grau de pigmentação máxima (ΔE\*max) das superfícies de topo e base dos espécimes de resina composta a partir da elaboração de uma fórmula matemática. Foi observado que a superfície de base dos materiais resinosos absorveu maior quantidade de pigmentos que a superfície de topo. Ainda, no geral, quando os materiais foram fotopolimerizados pela metade do tempo houve maior pigmentação da superfície de base em relação a fotopolimerização pelo dobro do tempo. No entanto, ambas as superfícies e tempos de fotoativação apresentaram um  $\Delta E^*$ max acima do valor considerado clinicamente aceitável ( $\Delta E \le 3.3/2.88$ ) a visão humana (Ruyter *et al.*, 1987).

O principal objetivo em se comparar a alteração de cor entre as superfícies de topo e base dos materiais resinosos foi justamente poder visualizar a diferença entre o grau de polimerização das duas superfícies. Como a superfície de base do incremento de resina composta se encontra mais distante da ponta do aparelho fotoativador, uma menor intensidade de luz atingirá esta superfície. Da mesma forma, há também uma atenuação da intensidade DE luz à medida que esta atinge as porções mais profundas do incremento de resina composta pela própria absorção e dispersão da luz ao longo do material resinoso (Rueggeberg *et al.,* 1994; Correr Sobrinho *et al.,* 2000; Franco & Lopes, 2003; Vandewalle *et al.,* 2004). Desta forma, é de se esperar um menor grau de conversão dos monômeros em polímeros na porção basal do incremento de resina composta.

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A deficiência de polimerização do material resinoso pode levar a deficiências em suas propriedades físicas, mecânicas, como também a uma menor longevidade do material com o tempo (Larsen & Munksgaard, 1991; Peutzfeldt, 1997; Millar & Nicholson, 2001 ; Rawls, 2005). Estes materiais pobremente polimerizados são mais susceptíveis a ação hidrolítica e de solventes orgânicos que podem estar presentes no meio bucal (Ferracane & Condon, 1999; Yap et al., 2004; Rawls & Esquivel-Upshaw, 2005), assim como a uma maior tendência a sorção de água e pigmentos do meio e, consequentemente, solubilidade. Desta forma, o segundo capítulo deste estudo teve como objetivo avaliar a ação de uma solução pigmentadora alcoólica (vinho tinto) sobre a dureza e rugosidade de superfície tanto da superfície de topo e de base dos mesmos materiais restauradores resinosos utilizados no primeiro capítulo. Ainda o grau de conversão das superfícies de topo e base dos diferentes tipos de resina compostas fotopolimerizadas com as mesmas fontes de luz, Halógena e LED, e tempos de fotoativação, foi diretamente averiguado por meio de dispositivo de reflectância total atenuada (ATR) acoplado em um espectroscópio infravermelho de transformação de Fourier (FTIR); e indiretamente através da microdureza Knoop. Desta maneira, um teste de correlação entre grau de conversão e microdureza pôde ser realizado, como também a sensibilidade de cada metodologia de teste em se averiguar a ação do vinho sobre as superfícies dos materiais resinosos.

No geral, a dureza e porcentagem do grau de conversão da superfície de topo dos materiais resinosos foram significantemente maiores que da superfície de base. O que novamente mostra a atenuação da intensidade de luz pelo aumento da distância da ponta do aparelho fotoativador ao incremento de resina composta e pela absorção e dispersão da luz ao longo do incremento resinoso (Rueggeberg *et al.*, 1994; Correr Sobrinho *et al.*, 2000; Franco & Lopes, 2003; Vandewalle *et al.*, 2004), prejudicando assim, a polimerização da sua porção basal. Ainda, a utilização de uma resina composta mais opaca (Supreme A2D) também possibilitou mostrar maior pigmentação da sua superfície de base, como também

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menor grau de conversão e dureza, por impedir adequada transmissão da luz ao longo do incremento resinoso (Christensen *et al.,* 1999; Rueggeberg, 1999).

Embora alguns materiais não apresentassem diferença no grau de conversão entre a superfície de topo e de base, como no caso da Durafill, muito provavelmente por ser um material resinoso mais translúcido o que permite uma maior transmissão da luz até sua porção basal (Christensen et al., 1999; Rueggeberg, 1999); ainda sim, a superfície de base apresentou maior pigmentação que a superfície de topo após imersão por 30 dias na solução de vinho tinto. Este fato pode mostrar que o teste de pigmentação muitas vezes pode ser mais sensível que o teste de grau de conversão em si para se avaliar a qualidade da rede polimérica de um material resinoso. Da mesma forma, o teste de microdureza Knoop comparado com o teste de grau de conversão. Isto se deve ao fato de que o teste de grau de conversão avalia a porcentagem de ligações duplas de carbono presentes na cadeia alifática que são transformadas em ligações simples após a fotoativação do material resinoso. No entanto, o grau de conversão não é capaz de avaliar o número de ligações cruzadas que são formadas após fotopolimerização do material (Ferracane, 1985). Desta forma, o teste de pigmentação e microdureza podem ser considerados mais sensíveis que o teste de grau de conversão para se analisar a durabilidade de um material resinoso ao longo do tempo. Isto porque, embora um material apresente um alto grau de conversão isto não implica que ele tenha uma maior densidade de ligações cruzadas, que confere ao material maior estabilidade química e física ao longo do tempo. Materiais com baixa densidade de ligações cruzadas são mais susceptíveis à degradação pela lixiviação dos monômeros residuais e cadeias lineares pequenas que se encontram no meio da trama polimérica (Yap et al., 2004).

Vários fatores podem contribuir para a formação de um material resinoso pobremente polimerizado como um aparelho fotoativador com baixa intensidade de luz, pouco tempo de fotoativação, maior distância da ponta do aparelho fotoativador e incremento de resina composta, o comprimento de onda emitido pelo aparelho fotoativador, o tipo de resina composta com relação ao tamanho e volume das partículas de carga, tipo de matriz resinosa e fotoiniciador, cor, espessura, translucidez/opacidade do incremento de resina composta entre outros (Rueggeberg *et al.*, 1994; Christensen *et al.*, 1999; Rueggeberg, 1999; Correr Sobrinho *et al.*, 2000; Franco & Lopes, 2003; Vandewalle *et al.*, 2004).

No presente estudo, o tempo de fotoativação também foi avaliado. Observou-se que na maioria das vezes as diferenças se encontravam entre o tempo recomendado pelo fabricante e a metade do tempo; e entre o dobro do tempo e metade do tempo. Poucas diferenças foram observadas entre o tempo recomendado e o dobro do tempo em termos de grau de conversão, dureza e pigmentação. Quando essas diferenças eram observadas, ocorriam para alguns tipos de resina composta. Desta maneira, no geral, a fotopolimerização pelo dobro do tempo teve pouca influência sobre as propriedades finais de dureza e estabilidade de cor dos materiais resinosos testados; o que implica em dizer que o tempo recomendado pelo fabricante ainda é o mais adequado, não só pela economia de tempo, mas também pelo dano que a fotoexposição por um período de tempo prolongando e uma fonte de luz de alta intensidade podem acarretar sobre o aumento da temperatura intrapulpar (Duray et al., 2008). Assim, uma redução na espessura do incremento de resina composta (menor que 2,0 mm) é sugerida como uma forma de se melhorar a polimerização da porção mais basal do material resinoso.

Neste estudo, no geral, a rugosidade das superfícies de topo e de base dos materiais resinosos testados foram pouco alteradas pela ação do vinho tinto. A única exceção foi para a resina composta Durafill, que apresentou maior rugosidade da superfície de base após exposição ao vinho por 30 dias. Esta maior rugosidade apresentada pela resina Durafill pode ser devido ao fato de ser uma resina do tipo microparticulada, a qual apresenta uma menor dureza, e assim, maior rugosidade (Sarac *et al.,* 2006). No entanto, acreditava-se que a superfície de base dos outros materiais resinosos e mesmo a superfície de topo fossem apresentar maior rugosidade após 30 dias de imersão em vinho tinto, o que acarretaria assim, em maior pigmentação dessas superfícies após o tempo de

imersão. No entanto, como não se observou uma alteração significante na rugosidade de superfície destes materiais após ação do vinho, sugere-se que o vinho tenha uma ação direta sobre a matriz resinosa, por meio da água e álcool presentes na sua composição. Tanto a água como o álcool são considerados solventes orgânicos que implicam na degradação da matriz resinosa pobremente polimerizada e com pouca densidade de ligações cruzadas, o que acarreta na lixiviação de monômeros residuais e de cadeias lineares pequenas, como também no amolecimento do material (Ferracane & Condon, 1999; Yap *et al.*, 2004; Rawls & Esquivel-Upshaw, 2005), permitindo assim a penetração de seus pigmentos no interior do material resinoso e não simplesmente pelo fato de tornar a superfície resinosa mais rugosa. Talvez para que isso acontecesse, um maior tempo de imersão fosse necessário, assim como o teor de álcool presente na solução de vinho tinto.

No geral, as duas fontes de luz testadas, Halógena e LED, apresentaram performances semelhantes em relação a susceptibilidade à pigmentação, dureza, grau de conversão e rugosidade de superfície das resinas testadas, diferindo apenas em alguns casos específicos, sendo assim, altamente dependente do tipo de material resinoso utilizado, superfície de topo ou base analisada, tempo de fotoativação emitido, entre outros. Desta forma, com base nos resultados do presente estudo, a escolha da fonte de luz a ser utilizada pode ser baseada em fatores independentes, como custo-benefício de aquisição e manutenção do aparelho fotoativador, assim como ergonomia durante sua utilização.

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# CONCLUSÃO

Diante dos resultados obtidos neste estudo, pôde-se concluir que:

1) As fontes de luz LED e Halógena apresentaram performance semelhante no que diz respeito a alteração de cor dos diversos materiais resinosos; assim como em relação ao grau de conversão, rugosidade de superfície e dureza tanto das superfícies de topo quanto de base.

2) Ambas as fontes de luz tiveram interação significante com tipo de resina composta, superfície de avaliação e tempo no vinho no que diz respeito a alteração de cor.

3) A fotopolimerização pelo dobro do tempo foi mais eficiente do que a fotopolimerização pela metade do tempo recomendado pelo fabricante. Em relação ao tempo recomendado, a fotopolimerização pelo dobro do tempo só foi mais eficiente para superfície de base de dois tipos de resina composta, Supreme A2D e Venus, onde foi observada uma redução da pigmentação pela solução de vinho tinto.

4) De todas as resinas compostas, a Supreme A2D foi a que apresentou menor  $\Delta E^*Max$  da superfície de topo, porém maior  $\Delta E^*max$  da superfície de base.

5) Todas resinas compostas apresentaram  $\Delta E^*$ max inaceitável ( $\Delta E \ge$  2.88) tanto na superfície de topo quanto na superfície de base.

**6)** Todas resinas compostas apresentaram maior ΔE\*Max na base (maior pigmentação) que na superfície de topo, com exceção da Venus quando polimerizada pelo dobro do tempo.

7) O grau de conversão entre as superfícies de topo e de base diferiu conforme o tipo de resina composta, tempo de fotoativação e fonte de luz. No entanto, a resina composta Durafill foi a única que não apresentou diferença no

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grau de conversão entre as duas superfícies, embora estes valores tenham sido mais baixos.

8) A dureza da superfície de topo foi geralmente maior que da superfície de base de acordo com tipo de resina composta, tempo de fotoativação e fonte de luz.

**9)** Após estocagem em vinho, a dureza da superfície de topo foi reduzida, porém ainda permaneceu maior que a dureza da superfície de base. As resinas Durafill e Venus não apresentaram diferença na dureza das superfícies após estocagem em vinho. E a resina Durafill foi a única que não apresentou diferença na dureza entre as superfícies de topo e de base após estocagem em vinho.

**10)** A rugosidade de superfície não diferiu entre as duas superfícies de topo e de base, nem antes e nem depois da estocagem no vinho; com exceção da resina Durafill que apresentou maior rugosidade das superfícies, principalmente da base, após imersão no vinho.
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<sup>\*</sup> De acordo com a norma da UNICAMP/FOP, baseadas na norma do International Committee of Medical Journal Editors – Grupo Vancouver. Abreviatura dos periódicos em conformidade com o Medline.

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3

# ANEXO

# Resultados da Análise Estatística

# A) Alteração de Cor

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The Mixed Procedure

Model Information

WORK.DE12M
DE00
Compound Symmetry
Resi*Ligh*Cure*Sampl
REML
Profile
Model-Based
Satterthwaite

## Class Level Information

Class	Levels	Values
Resin Light CureTime Sample Surface Day	4 2 3 2 3	Durafill Supreme Venus Z250 HAL LED 0.5 1 2 1 2 3 Bottom Top Dayl Day2 Max

## Dimensions

Covariance Parameters	2
Columns in X	720
Columns in Z	Ō
Subjects	72
Max Obs Per Subject	6

## Number of Observations

Number	of	Observations	Read	419
Number	of	Observations	Used	413
Number	of	Observations	Not Used	6

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The Mixed Procedure

### Iteration History

Iteration	Evaluations	-2 Res Log Like	Criterion
0 1 2	1 2 1	1003.05488367 970.74068000 970.74067729	0.0000001 0.0000000

Convergence criteria met.

#### Covariance Parameter Estimates

Cov Parm	Subject	Estimate
CS Residual	Resi*Ligh*Cure*Sampl	0.3863 1.0115

### Fit Statistics

-2 Res Log Likelihood	970.7
AIC (smaller is better)	974.7
AICC (smaller is better)	974.8
BIC (smaller is better)	979.3

### Null Model Likelihood Ratio Test

Pr > ChiSc	Chi-Square	DF
<.0001	32.31	1

## Type 3 Tests of Fixed Effects

Effect	Num DF	Den DF	F Value	Pr > F
Resin	3	47.1	118.51	<.0001
Light	1	47.1	0.00	0.9884
CureTime	2	47.1	13.62	<.0001

5

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## The Mixed Procedure

Type 3 Tests of Fixed Effects

Effect	Num DF	Den DF	F Value	Pr > F
Ettect Day Surface Resin*Light Resin*CureTime Resin*Day Resin*Surface Light*CureTime Light*Day Light*Surface CureTime*Day CureTime*Surface Surface*Day Resin*Light*CureTime Resin*Light*Surface Resin*CureTime*Day Resin*CureTi*Surface Resin*Surface*Day Light*CureTime*Day Light*CureTi*Surface Light*Surface*Day	DF 2 1 3 6 6 3 2 2 1 4 2 2 6 6 3 12 6 6 4 2 2	DF 224 223 47.1 47.1 224 223 47.1 224 223 224 223 223 47.1 224 223 224 223 224 223 224 223 224 223 224 223 224 223 223	F Value 2454.31 2323.95 1.37 1.08 35.25 284.55 1.88 0.46 2.23 4.43 24.60 189.37 0.83 3.34 2.41 2.24 2.29 15.97 0.91 0.62 1.71	Pr > F <.0001 <.0001 0.2627 0.3864 <.0001 <.0001 0.1643 0.6337 0.1369 0.0018 <.0001 <.0001 <.0001 0.5510 0.00365 <.0001 0.0365 <.0001 0.4560 0.5385 0.1832
CureTime*Surface*Day	4	223	5.65	0.0002
Resi*Light*CureT*Day	12	224	1.38	
Resi*Ligh*Cure*Surfa	6	223	0.57	0.7559
Resi*Light*Surfa*Day	6	223	2.23	0.0417
Resi*CureT*Surfa*Day	12	223	0.75	0.6997
Ligh*CureT*Surfa*Day	4	223	0.24	0.9141
Res*Lig*Cur*Surf*Day	12	223	0.19	0.9989

Resin	Light	CureTime	Day	Surface	DE00_N	DE00_Mean	DE00_StdDev	DE00_UCLM	DE00_LCLM
Durafill	HAL	0.5	Day1	Bottom	3	4.117617	1.222965	7.155631	1.079602
Durafill	HAL	0.5	Day1	Тор	3	2.217472	0.446056	3.325536	1.109407
Durafill	HAL	0.5	Day2	Bottom	3	5.144816	0.721897	6.938109	3.351524
Durafill	HAL	0.5	Day2	Тор	3	2.665506	0.793789	4.637386	0.693625
Durafill	HAL	0.5	Max	Bottom	3	12.97544	2.75068	19.8085	6.142368
Durafill	HAL	0.5	Max	Тор	3	7.861517	0.684127	9.560981	6.162052
Durafill	HAL	1	Day1	Bottom	3	3.274984	0.515359	4.555206	1.994761
Durafill	HAL	1	Day1	Тор	3	2.796706	0.009777	2.820994	2.772418
Durafill	HAL	1	Day2	Bottom	3	4.790983	0.187181	5.255966	4.325999
Durafill	HAL	1	Day2	Тор	3	3.117742	0.761822	5.010212	1.225271
Durafill	HAL	1	Max	Bottom	3	13.4074	0.318786	14.1993	12.61549
Durafill	HAL	1	Max	Тор	3	8.318128	2.636635	14.86789	1.768363
Durafill	HAL	2	Day1	Bottom	3	4.715664	0.612109	6.236228	3.195101
Durafill	HAL	2	Day1	Тор	3	2.555101	0.179781	3.001703	2.108499
Durafill	HAL	2	Day2	Bottom	3	4.783044	0.668476	6.443631	3.122457
Durafill	HAL	2	Day2	Тор	3	3.13308	0.457795	4.270307	1.995853
Durafill	HAL	2	Max	Bottom	3	12.96125	0.904103	15.20717	10.71534
Durafill	HAL	2	Max	Тор	3	9.019569	1.210143	12.02573	6.013407
Durafill	LED	0.5	Day1	Bottom	3	4.356443	1.100977	7.091421	1.621464
Durafill	LED	0.5	Day1	Тор	3	2.480315	1.029561	5.037886	-0.07726
Durafill	LED	0.5	Day2	Bottom	3	5.68052	0.47294	6.855369	4.505671
Durafill	LED	0.5	Day2	Тор	3	2.996028	0.828239	5.053489	0.938567
Durafill	LED	0.5	Max	Bottom	3	14.38182	0.812677	16.40062	12.36302
Durafill	LED	0.5	Max	Тор	3	8.896234	1.302812	12.1326	5.659868
Durafill	LED	1	Day1	Bottom	3	5.185528	0.948144	7.54085	2.830207
Durafill	LED	1	Day1	Тор	3	3.006824	0.109862	3.279737	2.733911
Durafill	LED	1	Day2	Bottom	3	5.766571	0.666948	7.423362	4.109779
Durafill	LED	1	Day2	Тор	3	3.769494	0.231627	4.344887	3.194101
Durafill	LED	1	Max	Bottom	3	14.36443	0.566453	15.77158	12.95728
Durafill	LED	1	Max	Тор	3	9.400132	0.533355	10.72506	8.075205
Durafill	LED	2	Day1	Bottom	3	3.518037	0.904664	5.765347	1.270728
Durafill	LED	2	Day1	Тор	3	2.07878	0.930799	4.391013	-0.23345
Durafill	LED	2	Day2	Bottom	3	3.416896	1.220886	6.449745	0.384048
Durafill	LED	2	Day2	Тор	3	2.367654	0.8928	4.585491	0.149817
Durafill	LED	2	Max	Bottom	3	11.69741	2.553701	18.04116	5.353668
Durafill	LED	2	Max	Тор	3	8.349121	1.632229	12.4038	4.294439
Supreme	HAL	0.5	Day1	Bottom	2	7.312338	2.632706	30.96627	-16.3416
Supreme	HAL	0.5	Day1	Тор	2	0.850427	0.002004	0.868431	0.832424
Supreme	HAL	0.5	Day2	Bottom	3	11.08089	2.208764	16.56776	5.594013
Supreme	HAL	0.5	Day2	Тор	3	1.110299	0.487426	2.321133	-0.10053
Supreme	HAL	0.5	Max	Bottom	3	25.6368	4.520531	36.86642	14.40718
Supreme	HAL	0.5	Max	Тор	2	4.880747	3.543152	36.71471	-26.9532
Supreme	HAL	1	Day1	Bottom	3	7.632746	0.67434	9.3079	5.957591
Supreme	HAL	1	Day1	Тор	3	1.492369	0.133099	1.823005	1.161733
Supreme	HAL	1	Day2	Bottom	3	8.720855	0.524316	10.02333	7.418383
Supreme	HAL	1	Day2	Тор	3	1.000194	0.171859	1.427115	0.573274
Supreme	HAL	1	Max	Bottom	3	19.33248	1.045628	21.92996	16.73499
Supreme	HAL	1	Max	Тор	3	3.690894	0.568997	5.104362	2.277426
Supreme	HAL	2	Day1	Bottom	3	7.498351	0.662301	9.143598	5.853104
Supreme	HAL	2	Day1	Тор	3	1.584393	0.726454	3.389005	-0.22022
Supreme	HAL	2	Day2	Bottom	3	8.688076	0.689266	10.40031	6.975845
Supreme	HAL	2	Day2	Тор	3	0.977497	0.559849	2.368239	-0.41324

									Anexo
Supreme	HAL	2	Max	Bottom	3	18.93468	2.431862	24.97577	12.8936
Supreme	HAL	2	Max	Тор	1	3.302655			
Supreme	LED	0.5	Dav1	Bottom	3	8.352106	0.893719	10.57223	6.131984
Supreme	LED	0.5	Dav1	Top	3	0.963244	0.378569	1.903662	0.022826
Supreme	LED	0.5	Dav2	Bottom	3	10 52751	0 75263	12 39714	8 657869
Supreme	LED	0.5	Day2	Ton	3	1 162663	0.498509	2 401028	-0.0757
Supreme	LED	0.5	Max	Bottom	3	23 54634	4 171844	33 90977	13 1829
Supreme	LED	0.5	Max	Ton	2	6 321151	0.020619	6 506402	6 1359
Supreme	LED I FD	1	Dav1	Bottom	3	8 155963	0.90753	10 41039	5 901535
Supreme	LED I FD	1	Day1	Ton	3	1 453326	0.310154	2 223791	0.68286
Supreme	LED	1	Day?	Bottom	3	9.452745	1.09/726	12 17210	6 733295
Supreme	LED I FD	1	Day2 Day2	Ton	3	1 143144	0.295478	1 877152	0.409135
Supreme	LED	1	Day2 Max	Bottom	3	20 7/800	2 63/095	27 29245	14 20554
Supreme	LED	1	Max	Top	1	7 100655	2.05+075	21.27243	14.20554
Supreme	LED	1	Dav1	Bottom	1	6 513448	1 318627	0 780101	3 237706
Supreme	LED	2	Day1	Top	3	1 23083	0.556654	2 613636	0.15108
Supreme	LED	2	Day1	Bottom	3	8 140200	1 33808	2.015050	-0.15198
Supreme	LED	2	Day2	Top	3	0.047601	0.523801	2 2/0108	4.810323
Supreme		2	Day2 Mox	Pottom	3	18 75757	2 030706	2.249108	-0.33373
Supreme		2	Mox	Top	2	7 763508	2.939700	20.00021	27 492
Vorue		2 0.5	Dow1	Pottom	2	2 210202	0.019751	5 501606	-37.403
Venus		0.5	Day1	Tom	2	3.219392	0.916/31	2.201090	0.937089
Venus	HAL	0.5	Day1	Top	3	1.750262	0.392732	2.725862	0.774003
Venus	HAL	0.5	Day2	Bottom	3	4.183205	0.749672	0.045494	2.320915
Venus	HAL	0.5	Day2		3	1.975645	0.444794	3.080574	0.870715
Venus	HAL	0.5	Max	Bottom	3	10.49461	0.822567	12.53798	8.45124
Venus	HAL	0.5	Max	Тор	3	4.709348	0.169/08	5.130926	4.28777
Venus	HAL	1	Dayl	Bottom	3	2.600747	0.307693	3.365098	1.836397
Venus	HAL	1	Dayl	Тор	3	1.747846	0.055374	1.885403	1.610289
Venus	HAL	1	Day2	Bottom	3	3.277557	0.505672	4.533/1/	2.021397
Venus	HAL	1	Day2	Тор	3	1.253064	0.224373	1.810438	0.695691
Venus	HAL	1	Max	Bottom	3	10.80679	1.311675	14.06517	7.548406
Venus	HAL	1	Max	Тор	3	6.1208	0.896688	8.348296	3.893304
Venus	HAL	2	Day1	Bottom	3	2.087306	0.176405	2.525519	1.649093
Venus	HAL	2	Day1	Тор	3	1.032961	0.883886	3.228657	-1.16273
Venus	HAL	2	Day2	Bottom	3	2.39925	0.506239	3.656818	1.141682
Venus	HAL	2	Day2	Тор	3	1.273971	0.805595	3.275179	-0.72724
Venus	HAL	2	Max	Bottom	3	6.974616	1.786966	11.41368	2.535547
Venus	HAL	2	Max	Тор	2	5.004552	0.529176	9.759012	0.250091
Venus	LED	0.5	Day1	Bottom	3	2.485782	0.421948	3.53396	1.437605
Venus	LED	0.5	Day1	Тор	3	1.320951	0.295512	2.055044	0.586859
Venus	LED	0.5	Day2	Bottom	3	3.02647	0.58167	4.471417	1.581523
Venus	LED	0.5	Day2	Тор	3	1.509826	0.338523	2.350765	0.668888
Venus	LED	0.5	Max	Bottom	3	11.33219	1.943236	16.15945	6.50492
Venus	LED	0.5	Max	Тор	2	4.10581	0.664563	10.07667	-1.86505
Venus	LED	1	Day1	Bottom	3	2.481422	0.47727	3.667026	1.295818
Venus	LED	1	Day1	Тор	3	0.912727	0.247278	1.526998	0.298455
Venus	LED	1	Day2	Bottom	3	3.473748	0.444457	4.577842	2.369655
Venus	LED	1	Day2	Тор	3	0.884417	0.432864	1.959711	-0.19088
Venus	LED	1	Max	Bottom	3	11.01536	0.970085	13.42518	8.605533
Venus	LED	1	Max	Тор	2	5.668023	0.871988	13.50253	-2.16648
Venus	LED	2	Day1	Bottom	3	2.109829	0.468562	3.273803	0.945856
Venus	LED	2	Day1	Тор	3	1.308307	0.329396	2.126572	0.490041
Venus	LED	2	Day2	Bottom	3	2.089657	0.37617	3.024115	1.155199
Venus	LED	2	Day2	Тор	3	1.267967	0.576926	2.70113	-0.1652

									Anexo
Venus	LED	2	Max	Bottom	3	7.57569	0.188343	8.04356	7.107821
Venus	LED	2	Max	Тор	2	4.310897	0.008752	4.389532	4.232261
Z250	HAL	0.5	Day1	Bottom	3	6.563956	0.154326	6.947324	6.180589
Z250	HAL	0.5	Day1	Тор	3	3.056139	0.631389	4.624596	1.487682
Z250	HAL	0.5	Dav2	Bottom	3	8.454825	0.720707	10.24516	6.664489
Z250	HAL	0.5	Day2	Тор	3	4.698054	0.602503	6.194754	3.201355
Z250	HAL	0.5	Max	Bottom	3	19.757	0.887116	21.96072	17.55329
Z250	HAL	0.5	Max	Тор	3	12.0396	0.744831	13.88986	10.18934
Z250	HAL	1	Day1	Bottom	2	6.093539	0.188131	7.783825	4.403253
Z250	HAL	1	Day1	Тор	2	2.917157	0.956017	11.50663	-5.67232
Z250	HAL	1	Day2	Bottom	2	7.650137	0.344072	10.7415	4.558771
Z250	HAL	1	Day2	Тор	2	3.506445	1.125656	13.62007	-6.60718
Z250	HAL	1	Max	Bottom	2	18.19013	0.452722	22.25768	14.12258
Z250	HAL	1	Max	Тор	2	11.61081	1.036134	20.92011	2.301513
Z250	HAL	2	Day1	Bottom	3	5.06423	1.282366	8.249803	1.878657
Z250	HAL	2	Day1	Тор	3	2.401682	0.853568	4.522062	0.281301
Z250	HAL	2	Day2	Bottom	3	6.531785	0.702491	8.276869	4.786701
Z250	HAL	2	Day2	Тор	3	3.106166	0.450533	4.225352	1.986981
Z250	HAL	2	Max	Bottom	3	16.94113	1.575853	20.85577	13.02649
Z250	HAL	2	Max	Тор	3	11.99757	2.402158	17.96486	6.030278
Z250	LED	0.5	Day1	Bottom	3	6.532982	0.268555	7.200109	5.865855
Z250	LED	0.5	Day1	Тор	3	3.495733	0.80065	5.484657	1.506808
Z250	LED	0.5	Day2	Bottom	3	8.003413	0.802217	9.99623	6.010595
Z250	LED	0.5	Day2	Тор	3	4.334137	0.759798	6.221581	2.446693
Z250	LED	0.5	Max	Bottom	3	17.26004	0.364078	18.16446	16.35562
Z250	LED	0.5	Max	Тор	3	10.40277	0.274569	11.08484	9.720706
Z250	LED	1	Day1	Bottom	3	5.96908	0.81047	7.982399	3.95576
Z250	LED	1	Day1	Тор	3	3.042064	0.179619	3.488261	2.595867
Z250	LED	1	Day2	Bottom	3	7.182053	1.038611	9.762105	4.602001
Z250	LED	1	Day2	Тор	3	3.966673	0.404027	4.970332	2.963014
Z250	LED	1	Max	Bottom	3	17.63979	1.323934	20.92862	14.35096
Z250	LED	1	Max	Тор	3	12.53804	1.39839	16.01183	9.064247
Z250	LED	2	Day1	Bottom	3	5.178183	1.122804	7.967383	2.388983
Z250	LED	2	Day1	Тор	3	2.471655	0.281772	3.171616	1.771694
Z250	LED	2	Day2	Bottom	3	6.373524	1.176607	9.296377	3.45067
Z250	LED	2	Day2	Тор	3	3.321626	0.457418	4.457914	2.185337
Z250	LED	2	Max	Bottom	3	14.74182	1.161424	17.62695	11.85668
Z250	LED	2	Max	Тор	3	9.730765	0.402759	10.73127	8.730257

										Anexo
					<u>DE Bonf</u>					
Resin	Light	Surface	Day	CureTime	Resin	Light	Surface	Day	CureTime	
Durafill	HAL	Bottom	D1	0.5	– Durafill	HAL	Bottom	Max	0.5	
Durafill	HAL	Bottom	D2	0.5	Durafill	HAL	Bottom	Max	0.5	
Durafill	HAL	Bottom	D2	0.5	Supreme	HAL	Bottom	D2	0.5	
Durafill	HAL	Bottom	Max	0.5	Durafill	HAL	Тор	Max	0.5	
Durafill	HAL	Bottom	Max	0.5	Supreme	HAL	Bottom	Max	0.5	
Durafill	HAL	Bottom	Max	0.5	Z250	HAL	Bottom	Max	0.5	
Durafill	HAL	Тор	D1	0.5	Durafill	HAL	Тор	Max	0.5	
Durafill	HAL	Тор	D2	0.5	Durafill	HAL	Тор	Max	0.5	
Durafill	HAL	Тор	Max	0.5	Z250	HAL	Тор	Max	0.5	
Durafill	HAL	Bottom	D1	1	Durafill	HAL	Bottom	Max	1	
Durafill	HAL	Bottom	D1	1	Supreme	HAL	Bottom	D1	1	
Durafill	HAL	Bottom	D2	1	Durafill	HAL	Bottom	Max	1	
Durafill	HAL	Bottom	D2	1	Supreme	HAL	Bottom	D2	1	
Durafill	HAL	Bottom	Max	1	Durafill	HAL	Тор	Max	1	
Durafill	HAL	Bottom	Max	1	Supreme	HAL	Bottom	Max	1	
Durafill	HAL	Bottom	Max	1	Z250	HAL	Bottom	Max	1	
Durafill	HAL	Тор	D1	1	Durafill	HAL	Тор	Max	1	
Durafill	HAL	Top	D2	1	Durafill	HAL	Тор	Max	1	
Durafill	HAL	Top	Max	1	Supreme	HAL	Тор	Max	1	
Durafill	HAL	Bottom	D1	2	Durafill	HAL	Bottom	Max	2	
Durafill	HAL	Bottom	D2	2	Durafill	HAL	Bottom	Max	2	
Durafill	HAL	Bottom	D2	2	Supreme	HAL	Bottom	D2	2	
Durafill	HAL	Bottom	Max	2	Durafill	HAL	Тор	Max	2	
Durafill	HAL	Bottom	Max	2	Supreme	HAL	Bottom	Max	2	
Durafill	HAL	Bottom	Max	- 2	Venus	HAL	Bottom	Max	- 2	
Durafill	HAL	Bottom	Max	2	7250	HAL	Bottom	Max	2	
Durafill	HAL	Top	D1	- 2	Durafill	HAL	Top	Max	- 2	
Durafill	HAL	Top	D2	- 2	Durafill	HAL	Top	Max	- 2	
Durafill	HAL	Top	Max	- 2	Supreme	HAL	Top	Max	- 2	
Durafill	LED	Bottom	D1	0.5	Durafill	LED	Bottom	Max	0.5	
Durafill	LED	Bottom	D1	0.5	Supreme	LED	Bottom	D1	0.5	
Durafill	LED	Bottom	D2	0.5	Durafill	LED	Bottom	Max	0.5	
Durafill	LED	Bottom	D2	0.5	Supreme	LED	Bottom	D2	0.5	
Durafill	LED	Bottom	Max	0.5	Durafill	LED	Тор	Max	0.5	
Durafill	LED	Bottom	Max	0.5	Supreme	LED	Bottom	Max	0.5	
Durafill	LED	Тор	D1	0.5	Durafill	LED	Тор	Max	0.5	
Durafill	LED	Top	D2	0.5	Durafill	LED	Тор	Max	0.5	
Durafill	LED	Top	Max	0.5	Venus	LED	Top	Max	0.5	
Durafill	LED	Bottom	D1	1	Durafill	LED	Bottom	Max	1	
Durafill	LED	Bottom	D2	1	Durafill	LED	Bottom	Max	1	
Durafill	LED	Bottom	Max	1	Durafill	LED	Тор	Max	1	
Durafill	LED	Bottom	Max	1	Supreme	LED	Bottom	Max	1	
Durafill	LED	Top	D1	1	Durafill	LED	Top	Max	1	
Durafill	LED	Top	D2	1	Durafill	LED	Top	Max	- 1	
Durafill	LED	Bottom	D1	2	Durafill	LED	Bottom	Max	2	
Durafill	LED	Bottom	D2	2	Durafill	LED	Bottom	Max	2	
Durafill	LED	Bottom	D2	2	Supreme	LED	Bottom	D2	2	
Durafill	LED	Bottom	Max	2	Durafill	LED	Тор	Max	2	
Durafill	LED	Bottom	Max	2	Supreme	LED	Bottom	Max	2	
Durafill	LED	Bottom	Max	2	Venus	LED	Bottom	Max	2	
Durafill	LED	Top	D1	2	Durafill	LED	Top	Max	2	
2		- ~r	~ 1	2			- ~r		-	

D£'11	LED	Тоя	D2		Dungfill	LED	Ter	M	2	Anexo
Durafill		Тор	D2	2	Durafill		Тор	Max	2	
Supreme	HAL	Bottom	DI	0.5	Supreme	HAL	Bottom	D2	0.5	
Supreme	HAL	Bottom	DI	0.5	Supreme	HAL	Bottom	Max	0.5	
Supreme	HAL	Bottom	DI	0.5	Supreme	HAL	Top	DI	0.5	
Supreme	HAL	Bottom	D2	0.5	Supreme	HAL	Bottom	Max	0.5	
Supreme	HAL	Bottom	D2	0.5	Supreme	HAL	Тор	D2	0.5	
Supreme	HAL	Bottom	D2	0.5	Venus	HAL	Bottom	D2	0.5	
Supreme	HAL	Bottom	Max	0.5	Supreme	HAL	Тор	Max	0.5	
Supreme	HAL	Bottom	Max	0.5	Supreme	HAL	Bottom	Max	1	
Supreme	HAL	Bottom	Max	0.5	Supreme	HAL	Bottom	Max	2	
Supreme	HAL	Bottom	Max	0.5	Venus	HAL	Bottom	Max	0.5	
Supreme	HAL	Bottom	Max	0.5	Z250	HAL	Bottom	Max	0.5	
Supreme	HAL	Тор	D1	0.5	Supreme	HAL	Тор	Max	0.5	
Supreme	HAL	Тор	D2	0.5	Supreme	HAL	Тор	Max	0.5	
Supreme	HAL	Тор	Max	0.5	Z250	HAL	Тор	Max	0.5	
Supreme	HAL	Bottom	D1	1	Supreme	HAL	Bottom	Max	1	
Supreme	HAL	Bottom	D1	1	Supreme	HAL	Тор	D1	1	
Supreme	HAL	Bottom	D1	1	Venus	HAL	Bottom	D1	1	
Supreme	HAL	Bottom	D2	1	Supreme	HAL	Bottom	Max	1	
Supreme	HAL	Bottom	D2	1	Supreme	HAL	Тор	D2	1	
Supreme	HAL	Bottom	D2	1	Venus	HAL	Bottom	D2	1	
Supreme	HAL	Bottom	Max	1	Supreme	HAL	Тор	Max	1	
Supreme	HAL	Bottom	Max	1	Venus	HAL	Bottom	Max	1	
Supreme	HAL	Тор	Max	1	Z250	HAL	Тор	Max	1	
Supreme	HAL	Bottom	D1	2	Supreme	HAL	Bottom	Max	2	
Supreme	HAL	Bottom	D1	2	Supreme	HAL	Тор	D1	2	
Supreme	HAL	Bottom	D1	2	Venus	HAL	Bottom	D1	2	
Supreme	HAL	Bottom	D2	2	Supreme	HAL	Bottom	Max	2	
Supreme	HAL	Bottom	D2	2	Supreme	HAL	Тор	D2	2	
Supreme	HAL	Bottom	D2	2	Venus	HAL	Bottom	D2	2	
Supreme	HAL	Bottom	Max	2	Supreme	HAL	Тор	Max	2	
Supreme	HAL	Bottom	Max	2	Venus	HAL	Bottom	Max	2	
Supreme	HAL	Тор	Max	2	Z250	HAL	Тор	Max	2	
Supreme	LED	Bottom	D1	0.5	Supreme	LED	Bottom	Max	0.5	
Supreme	LED	Bottom	D1	0.5	Supreme	LED	Тор	D1	0.5	
Supreme	LED	Bottom	D1	0.5	Venus	LED	Bottom	D1	0.5	
Supreme	LED	Bottom	D2	0.5	Supreme	LED	Bottom	Max	0.5	
Supreme	LED	Bottom	D2	0.5	Supreme	LED	Тор	D2	0.5	
Supreme	LED	Bottom	D2	0.5	Venus	LED	Bottom	D2	0.5	
Supreme	LED	Bottom	Max	0.5	Supreme	LED	Тор	Max	0.5	
Supreme	LED	Bottom	Max	0.5	Supreme	LED	Bottom	Max	2	
Supreme	LED	Bottom	Max	0.5	Venus	LED	Bottom	Max	0.5	
Supreme	LED	Bottom	Max	0.5	Z250	LED	Bottom	Max	0.5	
Supreme	LED	Top	D1	0.5	Supreme	LED	Ton	Max	0.5	
Supreme	LED	Top	D2	0.5	Supreme	LED	Ton	Max	0.5	
Supreme	LED	Bottom	D1	1	Supreme	LED	Bottom	Max	1	
Supreme	LED	Bottom	DI	1	Supreme	LED	Ton	D1	1	
Supreme	LED	Bottom	D1	1	Venue	LED	Bottom	D1	1	
Supreme	LED	Bottom	21	1	Supreme	LED	Bottom	Mav	1	
Supreme	LED	Bottom	2ם נת	1	Supreme	LED	Top	D2	1	
Supreme		Bottom	D2	1	Vonus		Pottom	D2	1	
Supreme		Dottom	D2 Mar	1	venus		Dottom	D2 Ma=	1	
Supreme		Dottom	Max	1	Supreme		1 op Dotte	IVIAX	1	
Supreme		Bottom		1	venus		Bottom	Max	1	
Supreme	LED	rob	וע	1	Supreme	LED	тор	Max	1	

<b>S</b>	LED	Tor	D2	1	Cupromo	LED	Tor	Mar	Anexo	-
Supreme	LED	Top	D2	1	Supreme	LED	Top	Max	1	
Supreme	LED	Bottom	DI	2	Supreme	LED	Bottom	Max	2	
Supreme	LED	Bottom	DI	2	Supreme	LED	Top	DI	2	
Supreme	LED	Bottom	DI	2	Venus	LED	Bottom	DI	2	
Supreme	LED	Bottom	D2	2	Supreme	LED	Bottom	Max	2	
Supreme	LED	Bottom	D2	2	Supreme	LED	Тор	D2	2	
Supreme	LED	Bottom	D2	2	Venus	LED	Bottom	D2	2	
Supreme	LED	Bottom	Max	2	Supreme	LED	Тор	Max	2	
Supreme	LED	Bottom	Max	2	Venus	LED	Bottom	Max	2	
Supreme	LED	Bottom	Max	2	Z250	LED	Bottom	Max	2	
Supreme	LED	Тор	D1	2	Supreme	LED	Тор	Max	2	
Supreme	LED	Тор	D2	2	Supreme	LED	Тор	Max	2	
Venus	HAL	Bottom	D1	0.5	Venus	HAL	Bottom	Max	0.5	
Venus	HAL	Bottom	D2	0.5	Venus	HAL	Bottom	Max	0.5	
Venus	HAL	Bottom	D2	0.5	Z250	HAL	Bottom	D2	0.5	
Venus	HAL	Bottom	Max	0.5	Venus	HAL	Тор	Max	0.5	
Venus	HAL	Bottom	Max	0.5	Z250	HAL	Bottom	Max	0.5	
Venus	HAL	Тор	Max	0.5	Z250	HAL	Тор	Max	0.5	
Venus	HAL	Bottom	D1	1	Venus	HAL	Bottom	Max	1	
Venus	HAL	Bottom	D2	1	Venus	HAL	Bottom	Max	1	
Venus	HAL	Bottom	D2	1	Z250	HAL	Bottom	D2	1	
Venus	HAL	Bottom	Max	1	Venus	HAL	Тор	Max	1	
Venus	HAL	Bottom	Max	1	Z250	HAL	Bottom	Max	-	
Venus	HAL	Ton	D1	1	Venus	HAL	Ton	Max	1	
Venus	HAL	Top	D2	1	Venus	HAL	Top	Max	1	
Venus	HAL	Top	Max	1	7250	HAL	Top	Max	1	
Venus	HAI	Bottom	D1	2	Venus	HAI	Bottom	Max	2	
Venus	HAI	Bottom	D1 D2	2	Venus	HAI	Bottom	Max	2	
Venus	НАІ	Bottom	D2 D2	2	7250	нат	Bottom	D2	2	
Venus	HAI	Bottom	D2 May	2	Z250	HAI	Bottom	D2 Max	2	
Venus		Ton	D1	2	Vonus		Ton	Mox	2	
Venus		Top	D1 D2	2	Vonus		Top	Max	2	
Venus	LIAL	Top	D2 Mov	2	7250	HAL	Top	Мах	2	
Venus		Dottom	D1	0.5	Z230		Top	Max	0.5	
Venus		Dottom		0.5	venus 7250		Bottom	D1	0.5	
Venus		Dottom		0.5	Z230		Bottom	DI	0.5	
Venus		Dottom	D2	0.5	venus 7250		Bottom	Max D2	0.5	
Venus	LED	Bottom	D2	0.5	Z250	LED	Bottom	D2	0.5	
Venus	LED	Bottom	Max	0.5	venus	LED		Max	0.5	
venus	LED	Bottom	Max	0.5	Z250	LED	Bottom	Max	0.5	
Venus	LED	Тор	Max	0.5	Z250	LED	Top	Max	0.5	
Venus	LED	Bottom	DI	1	venus	LED	Bottom	Max	1	
Venus	LED	Bottom	D2	1	Venus	LED	Bottom	Max	1	
Venus	LED	Bottom	Max	1	Venus	LED	Тор	Max	1	
Venus	LED	Bottom	Max	1	Z250	LED	Bottom	Max	1	
Venus	LED	Тор	D1	1	Venus	LED	Тор	Max	1	
Venus	LED	Тор	D2	1	Venus	LED	Тор	Max	1	
Venus	LED	Тор	Max	1	Z250	LED	Тор	Max	1	
Venus	LED	Bottom	D1	2	Venus	LED	Bottom	Max	2	
Venus	LED	Bottom	D2	2	Venus	LED	Bottom	Max	2	
Venus	LED	Bottom	D2	2	Z250	LED	Bottom	D2	2	
Venus	LED	Bottom	Max	2	Z250	LED	Bottom	Max	2	
Venus	LED	Тор	Max	2	Z250	LED	Тор	Max	2	
Z250	HAL	Bottom	D1	0.5	Z250	HAL	Bottom	Max	0.5	
Z250	HAL	Bottom	D1	0.5	Z250	HAL	Тор	D1	0.5	

 									Anexo
 Z250	HAL	Bottom	D2	0.5	Z250	HAL	Bottom	Max	0.5
Z250	HAL	Bottom	D2	0.5	Z250	HAL	Тор	D2	0.5
Z250	HAL	Bottom	Max	0.5	Z250	HAL	Тор	Max	0.5
Z250	HAL	Тор	D1	0.5	Z250	HAL	Тор	Max	0.5
Z250	HAL	Тор	D2	0.5	Z250	HAL	Тор	Max	0.5
Z250	HAL	Bottom	D1	1	Z250	HAL	Bottom	Max	1
Z250	HAL	Bottom	D2	1	Z250	HAL	Bottom	Max	1
Z250	HAL	Bottom	D2	1	Z250	HAL	Тор	D2	1
Z250	HAL	Bottom	Max	1	Z250	HAL	Тор	Max	1
Z250	HAL	Тор	D1	1	Z250	HAL	Тор	Max	1
Z250	HAL	Тор	D2	1	Z250	HAL	Тор	Max	1
Z250	HAL	Bottom	D1	2	Z250	HAL	Bottom	Max	2
Z250	HAL	Bottom	D2	2	Z250	HAL	Bottom	Max	2
Z250	HAL	Bottom	D2	2	Z250	HAL	Тор	D2	2
Z250	HAL	Bottom	Max	2	Z250	HAL	Тор	Max	2
Z250	HAL	Тор	D1	2	Z250	HAL	Тор	Max	2
Z250	HAL	Тор	D2	2	Z250	HAL	Тор	Max	2
Z250	LED	Bottom	D1	0.5	Z250	LED	Bottom	Max	0.5
Z250	LED	Bottom	D2	0.5	Z250	LED	Bottom	Max	0.5
Z250	LED	Bottom	D2	0.5	Z250	LED	Тор	D2	0.5
Z250	LED	Bottom	Max	0.5	Z250	LED	Тор	Max	0.5
Z250	LED	Тор	D1	0.5	Z250	LED	Тор	Max	0.5
Z250	LED	Тор	D2	0.5	Z250	LED	Тор	Max	0.5
Z250	LED	Bottom	D1	1	Z250	LED	Bottom	Max	1
Z250	LED	Bottom	D2	1	Z250	LED	Bottom	Max	1
Z250	LED	Bottom	Max	1	Z250	LED	Тор	Max	1
Z250	LED	Тор	D1	1	Z250	LED	Тор	Max	1
Z250	LED	Тор	D2	1	Z250	LED	Тор	Max	1
Z250	LED	Bottom	D1	2	Z250	LED	Bottom	Max	2
Z250	LED	Bottom	D2	2	Z250	LED	Bottom	Max	2
Z250	LED	Bottom	Max	2	Z250	LED	Тор	Max	2
Z250	LED	Тор	D1	2	Z250	LED	Тор	Max	2
Z250	LED	Тор	D2	2	Z250	LED	Тор	Max	2
Supreme	HAL	Bottom	D1	0.5	Venus	HAL	Bottom	D1	0.5
Venus	HAL	Bottom	Max	1	Venus	HAL	Bottom	Max	2
Venus	LED	Bottom	Max	0.5	Venus	LED	Bottom	Max	2

# B) Valores correspondentes entre CIELAB 1976 e 2000

P:\ClaudiaBatitucci-Santos\RelateDE2000to1976.sas: 02NOV09:12:41:15 1 where de1976 <= 4

The REG Procedure Model: MODEL1 Dependent Variable: DE2000

Number	of	Observations	Read		241
Number	of	Observations	Jsed		219
Number	of	Observations	with Missing	Values	22

## Analysis of Variance

		Sum of	Mean		
Source	DF	Squares	Square	F Value	Pr > F
Model	1	151.08668	151.08668	1011.06	<.0001
Error	217	32.42701	0.14943		
Corrected Total	218	183.51369			
Root MSE	0.38657	R-Square	0.8233		
Dependent Mean Coeff Var	2.15767 17.91589	Adj R-Sq	0.8225		

#### Parameter Estimates

Variable	DF	Parameter Estimate	Standard Error	t Value	Pr >  t
Intercept	1	0.08456	0.07024	1.20	0.2299
DE1976	1	0.84886	0.02670	31.80	<.0001

where de1976<=4

	Predicted
	Value of
DE1976	DE2000
1.0	0.93342
3.3	2.88579

1

# C) Grau de Conversão

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The Mixed Procedure

Model Information

Data Set	WORK.DC
Dependent Variable	DC
Covariance Structure	Compound Symmetry
Subject Effect	Samp*Ligh*Resi*CureT
Estimation Method	REML
Residual Variance Method	Profile
Fixed Effects SE Method	Model-Based
Degrees of Freedom Method	Satterthwaite

### Class Level Information

Class	Levels	Values
Sample	5	1 2 3 4 5
Light	2	Halogen LED
Resin	5	Durafil SuprA2D SuprA2E Venus Z250
CureTime	3	0.5 1 2
Surface	2	Bottom Top

#### Dimensions

Covariance	Parameters	2
Columns in	Х	216
Columns in	Z	0
Subjects		150
Max Obs Per	Subject	2

### Number of Observations

Number	of	Observations	Read	300
Number	of	Observations	Used	300
Number	of	Observations	Not Used	0

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The Mixed Procedure

### Iteration History

Iteration	Evaluations	-2 Res Log Like	Criterion
0	1	1297.11402715	0.0000000
1	1	1297.09574755	

Convergence criteria met.

#### Covariance Parameter Estimates

Cov Parm	Subject	Estimate
CS	Samp*Ligh*Resi*CureT	-0.1075
Residual		8.8169

#### Fit Statistics

-2 Res Log Likelihood	1297.1
AIC (smaller is better)	1301.1
AICC (smaller is better)	1301.1
BIC (smaller is better)	1307.1

#### Null Model Likelihood Ratio Test

DF	Chi-Square	Pr > ChiSq
1	0.02	0.8925

### Type 3 Tests of Fixed Effects

	Num	Den		
Effect	DF	DF	F Value	Pr > F
CureTime	2	120	48.56	<.0001
Light	1	120	6.28	0.0136
Resin	4	120	11.16	<.0001

2

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#### The Mixed Procedure

## Type 3 Tests of Fixed Effects

Num DF	Den DF	F Value	Pr > F
1	120	582.36	<.0001
2	120	2.00	0.1397
8	120	1.66	0.1141
2	120	11.90	<.0001
4	120	3.29	0.0134
1	120	16.27	<.0001
4	120	41.55	<.0001
8	120	1.96	0.0572
2	120	0.78	0.4589
8	120	0.89	0.5308
4	120	2.96	0.0227
8	120	1.14	0.3407
	Num DF 1 2 8 2 4 1 4 8 2 8 4 8 4 8 4 8	Num DF Den DF   1 120   2 120   8 120   2 120   4 120   1 120   4 120   8 120   2 120   4 120   8 120   2 120   8 120   8 120   8 120   8 120   8 120	Num DFDen DFDFDFF Value1120582.3621202.0081201.66212011.9041203.29112016.27412041.5581201.9621200.7881200.8941202.9681201.14

3

Resin	Light	CureTime	Surface	DC_N	DC_Mean	DC_StdDev	DC_UCLM	DC_LCLM
Durafil	Halogen	0.5	Bottom	5	48.81753	7.283153	57.86076	39.7743
Durafil	Halogen	0.5	Тор	5	53.18365	3.010901	56.92218	49.44512
Durafil	Halogen	1	Bottom	5	49.95757	3.746473	54.60943	45.30571
Durafil	Halogen	1	Тор	5	51.99675	2.642758	55.27816	48.71533
Durafil	Halogen	2	Bottom	5	53.51662	4.224843	58.76245	48.27078
Durafil	Halogen	2	Тор	5	56.52718	3.211221	60.51444	52.53992
Durafil	LED	0.5	Bottom	5	48.53044	2.63587	51.8033	45.25757
Durafil	LED	0.5	Тор	5	48.50837	2.140432	51.16607	45.85068
Durafil	LED	1	Bottom	5	51.9042	2.031038	54.42606	49.38233
Durafil	LED	1	Тор	5	46.59821	7.836497	56.32851	36.86792
Durafil	LED	2	Bottom	5	56.39559	2.441081	59.42659	53.36459
Durafil	LED	2	Тор	5	50.0017	4.505661	55.59622	44.40718
SuprA2D	Halogen	0.5	Bottom	5	40.15124	2.673352	43.47064	36.83183
SuprA2D	Halogen	0.5	Тор	5	58.47863	2.424654	61.48924	55.46802
SuprA2D	Halogen	1	Bottom	5	46.15462	2.214591	48.9044	43.40484
SuprA2D	Halogen	1	Тор	5	58.49671	1.216075	60.00666	56.98675
SuprA2D	Halogen	2	Bottom	5	50.66598	1.549509	52.58995	48.74201
SuprA2D	Halogen	2	Тор	5	59.86676	1.615134	61.87221	57.8613
SuprA2D	LED	0.5	Bottom	5	45.77117	2.535064	48.91887	42.62348
SuprA2D	LED	0.5	Тор	5	57.12284	0.683296	57.97127	56.27442
SuprA2D	LED	1	Bottom	5	47.97132	2.872229	51.53766	44.40498
SuprA2D	LED	1	Тор	5	57.75251	1.7152	59.88222	55.62281
SuprA2D	LED	2	Bottom	5	48.34031	1.846492	50.63303	46.04759
SuprA2D	LED	2	Тор	5	58.18608	0.61438	58.94894	57.42323
SuprA2E	Halogen	0.5	Bottom	5	42.75	2.711613	46.11692	39.38309
SuprA2E	Halogen	0.5	Тор	5	56.20398	2.233016	58.97663	53.43132
SuprA2E	Halogen	1	Bottom	5	45.79246	1.109106	47.1696	44.41532
SuprA2E	Halogen	1	Тор	5	56.7645	1.797625	58.99655	54.53246
SuprA2E	Halogen	2	Bottom	5	51.51309	1.149273	52.9401	50.08608
SuprA2E	Halogen	2	Тор	5	63.56375	8.298372	73.86754	53.25996
SuprA2E	LED	0.5	Bottom	5	42.28542	3.19128	46.24791	38.32292
SuprA2E	LED	0.5	Top	5	53,72005	1.282308	55.31225	52,12786
SuprA2E	LED	1	Bottom	5	45.71087	4.066902	50.7606	40.66115
SuprA2E	LED	1	Тор	5	57.55977	4.676579	63.36651	51.75303
SuprA2E	LED	2	Bottom	5	48.86212	0.927585	50.01387	47.71037
SuprA2E	LED	- 2	Top	5	54.91041	0.95133	56.09165	53,72918
Venus	Halogen	0.5	Bottom	5	46.17967	1.656071	48.23596	44.12339
Venus	Halogen	0.5	Top	5	58 26827	0 544308	58 94411	57 59242
Venus	Halogen	1	Bottom	5	50 71647	2 104299	53 32931	48 10364
Venus	Halogen	1	Top	5	60.07321	1 985647	62 53872	57 6077
Venus	Halogen	2	Bottom	5	53 2599	3 745742	57 91085	48 60894
Venus	Halogen	2	Ton	5	60 62798	0.806616	61 62952	59 62643
Venus	I FD	05	Bottom	5	47 64815	0.829848	48 67854	46 61776
Vonus	LED	0.5	Ton	5	57 6376	1 587365	50 60857	55 66663
Venus	LED	0.5	Bottom	5 5	47 13035	5 40/0/7	53 8/1036	40 42034
Venus	LED	1	Top	5	58 76225	0.035120	59 10117	-10.42934 57 10001
Venus	LED	1	Bottom	5 5	57 1696	1 200/21	53.42447 53.70117	51.10224
Venus	LED	2	Top	5 5	50 7/120	1.309431	67 20855	50.54275 57 77777
7250	Helocon	2	Bottom	5 5	J7.14130 16 50152	1.700703	47 00222	JI.21422 15 76571
Z230 7250	Halogen	0.5	Ton	5 5	40.J04JJ 58 JJN51	0.602020	47.90332 58 06015	4J.20374 57 17106
Z230 7250	Halogen	0.3	Bottom	5 5	JO.22031 17 12006	0.002939	50.20213	J1.4/100 12 20557
Z250 7250	Halogen	1	Ton	5 5	+7.12090 50 12207	2.397034	50.54055 61.0102	40.09001 57 71071
L230	паюден	1	rop	3	JY.13321	1.31814/	01.0183	J1.24824

									Anexo
Z250	Halogen	2	Bottom	5	50.47657	4.056269	55.51309	45.44005	
Z250	Halogen	2	Тор	5	57.11253	1.678365	59.1965	55.02857	
Z250	LED	0.5	Bottom	5	47.96955	1.79132	50.19377	45.74534	
Z250	LED	0.5	Тор	5	57.04815	1.500933	58.9118	55.18449	
Z250	LED	1	Bottom	5	48.54077	2.2589	51.34557	45.73598	
Z250	LED	1	Тор	5	59.87749	1.769935	62.07516	57.67983	
Z250	LED	2	Bottom	5	52.43715	2.713603	55.80653	49.06776	
Z250	LED	2	Тор	5	58.11178	2.598945	61.3388	54.88477	

# DC Bonf

Light	Resin	Surface	CureTime	_Light	_Resin	_Surface	_CureTime
Halogen	Durafil	Bottom	0.5	Halogen	SuprA2D	Bottom	0.5
Halogen	Durafil	Тор	1	Halogen	Venus	Тор	1
Halogen	SuprA2D	Bottom	0.5	Halogen	SuprA2D	Тор	0.5
Halogen	SuprA2D	Bottom	0.5	Halogen	SuprA2D	Bottom	2
Halogen	SuprA2D	Bottom	1	Halogen	SuprA2D	Тор	1
Halogen	SuprA2D	Bottom	2	Halogen	SuprA2D	Тор	2
Halogen	SuprA2E	Bottom	0.5	Halogen	SuprA2E	Тор	0.5
Halogen	SuprA2E	Bottom	0.5	Halogen	SuprA2E	Bottom	2
Halogen	SuprA2E	Bottom	1	Halogen	SuprA2E	Тор	1
Halogen	SuprA2E	Bottom	2	Halogen	SuprA2E	Тор	2
Halogen	SuprA2E	Тор	2	LED	SuprA2E	Тор	2
Halogen	Venus	Bottom	0.5	Halogen	Venus	Тор	0.5
Halogen	Venus	Bottom	1	Halogen	Venus	Тор	1
Halogen	Z250	Bottom	0.5	Halogen	Z250	Тор	0.5
Halogen	Z250	Bottom	1	Halogen	Z250	Тор	1
LED	Durafil	Bottom	0.5	LED	Durafil	Bottom	2
LED	Durafil	Тор	0.5	LED	SuprA2D	Тор	0.5
LED	Durafil	Тор	0.5	LED	Venus	Тор	0.5
LED	Durafil	Тор	0.5	LED	Z250	Тор	0.5
LED	Durafil	Тор	1	LED	SuprA2D	Тор	1
LED	Durafil	Тор	1	LED	SuprA2E	Тор	1
LED	Durafil	Тор	1	LED	Venus	Тор	1
LED	Durafil	Тор	1	LED	Z250	Тор	1
LED	Durafil	Bottom	2	LED	SuprA2D	Bottom	2
LED	Durafil	Bottom	2	LED	SuprA2E	Bottom	2
LED	Durafil	Тор	2	LED	SuprA2D	Тор	2
LED	Durafil	Тор	2	LED	Venus	Тор	2
LED	Durafil	Тор	2	LED	Z250	Тор	2
LED	SuprA2D	Bottom	0.5	LED	SuprA2D	Тор	0.5
LED	SuprA2D	Bottom	1	LED	SuprA2D	Тор	1
LED	SuprA2D	Bottom	2	LED	SuprA2D	Тор	2
LED	SuprA2E	Bottom	0.5	LED	SuprA2E	Тор	0.5
LED	SuprA2E	Bottom	1	LED	SuprA2E	Тор	1
LED	Venus	Bottom	0.5	LED	Venus	Тор	0.5
LED	Venus	Bottom	1	LED	Venus	Тор	1
LED	Venus	Bottom	2	LED	Venus	Тор	2
LED	Z250	Bottom	0.5	LED	Z250	Тор	0.5
LED	Z250	Bottom	1	LED	Z250	Тор	1
Halogen	SuprA2E	Тор	0.5	Halogen	SuprA2E	Тор	2
Halogen	Venus	Bottom	2	Halogen	Venus	Тор	2
Halogen	Durafil	Тор	1	Halogen	Z250	Тор	1
Halogen	Durafil	Тор	2	Halogen	SuprA2E	Тор	2
Halogen	Venus	Bottom	0.5	Halogen	Venus	Bottom	2

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# D) Microdureza Knoop

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The Mixed Procedure

Model Information

Data Set	WORK.KHN
Dependent Variable	KHN
Covariance Structure	Compound Symmetry
Subject Effect	Samp*Ligh*Resi*CureT
Estimation Method	REML
Residual Variance Method	Profile
Fixed Effects SE Method	Model-Based
Degrees of Freedom Method	Satterthwaite

#### Class Level Information

Class	Levels	Values
Sample Light Resin	5 2 5	1 2 3 4 5 Halogen LED Durafill SuprA2D SuprA2E Venus Z250
CureTime Wine Surface	3 2 2	0.5 1 2 After Before Bottom Top

#### Dimensions

Covariance	Parameters	2
Columns in	Х	648
Columns in	Z	0
Subjects		150
Max Obs Per	Subject	4

#### Number of Observations

Number	of	Observations	Read	600
Number	of	Observations	Used	600
Number	of	Observations	Not Used	0

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The Mixed Procedure

### Iteration History

Iteration	Evaluations	-2 Res Log Like	Criterion
0	1	3422.11588486	0.0000000
1	1	3407.13500444	

Convergence criteria met.

#### Covariance Parameter Estimates

Cov Parm	Subject	Estimate
CS	Samp*Ligh*Resi*CureT	7.6216
Residual		41.2488

#### Fit Statistics

-2 Res Log Likelihood	3407.1
AIC (smaller is better)	3411.1
AICC (smaller is better)	3411.2
BIC (smaller is better)	3417.2

Null Model Likelihood Ratio Test

DF	Chi-Square	Pr > ChiSq
1	14.98	0.0001

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#### The Mixed Procedure

Type 3 Tests of Fixed Effects

	Num	Den		
Effect	DF	DF	F Value	Pr > F
Light	1	120	1.46	0.2291
Resin	4	120	594.91	<.0001
CureTime	2	120	19.37	<.0001
Surface	1	360	2049.37	<.0001
Wine	1	360	218.26	<.0001
Light*Resin	4	120	1.08	0.3688
Light*CureTime	2	120	0.40	0.6684
Light*Surface	1	360	2.38	0.1241
Light*Wine	1	360	1.37	0.2422
Resin*CureTime	8	120	1.20	0.3070
Resin*Surface	4	360	33.22	<.0001
Resin*Wine	4	360	9.35	<.0001
CureTime*Surface	2	360	10.97	<.0001
CureTime*Wine	2	360	0.83	0.4384
Wine*Surface	1	360	100.92	<.0001
Light*Resin*CureTime	8	120	0.66	0.7294
Light*Resin*Surface	4	360	2.29	0.0591
Light*Resin*Wine	4	360	6.52	<.0001
Light*CureTi*Surface	2	360	1.16	0.3157
Light*CureTime*Wine	2	360	1.15	0.3188
Light*Wine*Surface	1	360	1.24	0.2662
Resin*CureTi*Surface	8	360	2.64	0.0080
Resin*CureTime*Wine	8	360	0.54	0.8272
Resin*Wine*Surface	4	360	2.28	0.0602
CureTim*Wine*Surface	2	360	0.54	0.5858
Ligh*Resi*Cure*Surfa	8	360	0.95	0.4738
Ligh*Resi*CureT*Wine	8	360	1.14	0.3382
Ligh*Resi*Wine*Surfa	4	360	2.09	0.0820
Ligh*Cure*Wine*Surfa	2	360	0.89	0.4120
Resi*Cure*Wine*Surfa	8	360	0.79	0.6116
Lig*Res*Cur*Win*Surf	8	360	1.02	0.4238

Resin	Light	CureTime	Wine	Surface	KHN N	KHN Mean	KHN StdDev	KHN I CI M	KHN UCIM
Durafill	Halogen	0.5	After	Bottom	<b>KIII\_I</b> \ 5	11 10673245	1 410282444	9 355635515	12.85782939
Durafill	Halogen	0.5	After	Ton	5	25 87258577	3 524228949	21 49667757	30 24849398
Durafill	Halogen	0.5	Before	Bottom	5	10 97100451	0.661281185	10 14991547	11 79209355
Durafill	Halogen	0.5	Before	Ton	5	28 31060447	2 839911106	24 78438909	31 83681985
Durafill	Halogen	0.5	After	Bottom	5	13 2292616	1 609731486	11 23051596	15 22800723
Durafill	Halogen	1	After	Ton	5	24 68234068	3 408873009	20 44966579	28 91501557
Durafill	Halogen	1	Before	Bottom	5	13 87439187	3 241789166	9 849178969	17 89960476
Durafill	Halogen	1	Before	Ton	5	27 89124229	2 412979223	24 89513286	30 88735172
Durafill	Halogen	2	After	Bottom	5	18 6826019	13 42261615	2.016222658	35 34898113
Durafill	Halogen	2	After	Ton	5	32,70426476	18 24050146	10.05569079	55 35283874
Durafill	Halogen	2	Before	Bottom	5	13 63367041	2.086439574	11.04301351	16 22432731
Durafill	Halogen	- 2	Before	Ton	5	35 47134053	2 589609281	32 25591592	38 68676514
Durafill	LED	05	After	Bottom	5	11 44758037	2.555624142	8 274353882	14 62080686
Durafill	LED	0.5	After	Ton	5	21.08161955	1 842296217	18 79410666	23 36913243
Durafill	LED	0.5	Before	Bottom	5	10 77268803	1 830297245	8 500073836	13 04530223
Durafill	LED	0.5	Before	Ton	5	23 48425265	2 832616468	19 96709476	27 00141053
Durafill	LED	0.5	After	Bottom	5	12 64591309	1 744117602	10 48030506	14 81152113
Durafill	LED	1	After	Ton	5	22 02519979	2 162247164	19 34041533	24 70998425
Durafill	LED	1	Before	Bottom	5	12 50512295	1 094872402	11 14565931	13 86458659
Durafill	LED	1	Before	Ton	5	28 74653422	4 260441182	23 45649779	34 03657065
Durafill	LED	2	After	Bottom	5	11 63030071	1 970889364	9 183118344	14 07748308
Durafill	LED	2	After	Ton	5	20.07261742	4.030675162	15 06787319	25 07736166
Durafill	LED	2	Before	Bottom	5	15 6031	2 736477262	12 29531/71	10 0008853
Durafill	LED	2	Before	Ton	5	28 67558775	0.7/3805821	16 5769/311	10.77423230
SuprA2D	Halogen	0.5	After	Bottom	5	38 86174709	1 8/2957059	32 8/8/2167	40.77423237
SuprA2D	Halogen	0.5	After	Ton	5	64 06812084	3 027720076	60 30871883	67 82754086
SuprA2D	Halogen	0.5	Refore	Bottom	5	42 05585865	3 165116082	38 1258/1796	15 98586934
SuprA2D	Halogen	0.5	Before	Ton	5	76 90037276	8 107286243	66 8338/173	86 96689821
SuprA2D	Halogen	0.5	After	Bottom	5	10.90037270	3 005001610	40 11773741	40.81750575
SuprA2D	Halogen	1	After	Ton	5	70.00301835	5.116057346	40.11773741 63.65170713	76 35634257
SuprA2D	Halogen	1	Before	Bottom	5	45 50450312	2 505028663	42 30208172	10.55054257 48.61602452
SuprA2D	Ualogon	1	Before	Ton	5	78 75460050	10 21502070	42.39298172	48.01002452
SuprA2D	Halogen	1	After	Bottom	5	51 40200321	10.31393079	45 01275855	56 801/2788
SuprA2D	Halogen	2	After	Ton	5	68 03664667	5 836866753	43.91273833	75 28407308
SuprA2D	Ualogon	2	Pafara	Pottom	5	51.06851020	6 56354463	42 01880222	50 21822626
SuprA2D	Ualogon	2	Before	Ton	5	26 25698729	8 722452246	42.91880222	07 18848022
SuprA2D		0.5	After	Pottom	5	36.00820773	5 16271276	20 5867015	<i>97.10040022</i> <i>43.40080306</i>
SuprA2D		0.3	After	Ton	5	50.99829775	7 82001281	55 22862627	43.40989390
SuprA2D		0.3	Defere	Pottom	5	04.94040390	2.052270022	27 2151024	14.03629339
SuprA2D		0.5	Before	Ton	5	39.87079555 87 50012524	11 00824444	72 82150442	42.4204677
SuprA2D		0.5	After	Pottom	5	44 52927025	2 66700522	11 22662412	101.1080701
SuprA2D		1	After	Ton	5	44.33827033 64.44027565	2.00709332	41.22003412 53.60422804	47.84990039
SuprA2D		1	Before	Bottom	5	04.44 <i>9273</i> 03	1 580/681	<i>12</i> 78213006	15.29452521
SuprA2D		1	Defore	Ton	5	44.7443413	6 211467720	42.78213090	40.70095104
SuprA2D		1	After	Pottom	5	79.41017730 51.1427870	0.511407729	/1.3/943331	07.23209902 57.19054472
SuprA2D		2	After	Ton	5	51.1457679	4.809070552	43.09803109	74 20540408
SuprA2D		2	Defere	Pottom	5	55 74907527	0.223032033	50 909 4 10 4 7	74.20349496
SuprA2D		2	Defore	Ton	5	78 80050504	3.976960991	74 2026259	00.06934020
SuprA2D	LED Halogan	2	After	Bottom	5 5	10.00030304	J.UZ1UJJ492 7 702567700	14.3030238 30.82020479	03.27130420 50 19107960
SuprA2E	Halagan	0.5	After	Ton	5 5	40.3072807	0.0128201208	50.05029478 51 10011176	JU.1042/002
SuprA2E	Halagan	0.5	Before	Bottom	5 5	40.71907307	7.713020427 0 807560117	24.402441/0 28 501/06/2	17.020/0738
SuprA2E	Halogen	0.5	Defore	Бощот Тот	5	40./09100/4	9.80/30011/	28.39140043	32.940/9303
SuprA2E	Halogen	0.5	Before	тор	3	/9./3138/44	10.91620849	00.19/12436	95.50505052

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SuprA2E	Halogen	1	After	Bottom	5	46.41004169	9.091000048	35.12207423	57.69800916
SuprA2E	Halogen	1	After	Тор	5	72.20022889	6.610802121	63.9918339	80.40862388
SuprA2E	Halogen	1	Before	Bottom	5	49.90918358	3.74001022	45.26534753	54.55301962
SuprA2E	Halogen	1	Before	Тор	5	79.17633036	8.89048715	68.13733254	90.21532818
SuprA2E	Halogen	2	After	Bottom	5	47.65445253	7.036165721	38.91789887	56.39100619
SuprA2E	Halogen	2	After	Тор	5	70.09895107	6.515250232	62.00919942	78.18870272
SuprA2E	Halogen	2	Before	Bottom	5	52.04271172	7.404395533	42.84894036	61.23648308
SuprA2E	Halogen	2	Before	Тор	5	77.14693377	21.93254468	49.91408265	104.3797849
SuprA2E	LED	0.5	After	Bottom	5	39.72416514	4.258265115	34.43683065	45.01149963
SuprA2E	LED	0.5	After	Тор	5	69.63848942	4.604065585	63.92178694	75.35519191
SuprA2E	LED	0.5	Before	Bottom	5	42.61823974	6.778452307	34.20167954	51.03479993
SuprA2E	LED	0.5	Before	Тор	5	88.577311	15.72074319	69.05743016	108.0971918
SuprA2E	LED	1	After	Bottom	5	43.34681316	18.00119606	20.99537609	65.69825023
SuprA2E	LED	1	After	Тор	5	61.0115257	20.16730288	35.97051177	86.05253964
SuprA2E	LED	1	Before	Bottom	5	47.50454529	6.486859849	39.45004496	55.55904563
SuprA2E	LED	1	Before	Тор	5	93.64635554	15.85815429	73.95585629	113.3368548
SuprA2E	LED	2	After	Bottom	5	44.81652026	6.221210629	37.091867	52.54117352
SuprA2E	LED	2	After	Тор	5	62.80399834	3.535904635	58.41359285	67.19440382
SuprA2E	LED	2	Before	Bottom	5	53.40879335	3.11856314	49.53658577	57.28100093
SuprA2E	LED	2	Before	Тор	5	87.52113319	4.170868552	82.34231587	92.69995051
Venus	Halogen	0.5	After	Bottom	5	22.97615675	3.276007791	18.90845582	27.04385768
Venus	Halogen	0.5	After	Тор	5	50.29219817	3.660533891	45.74704503	54.83735132
Venus	Halogen	0.5	Before	Bottom	5	23.52638903	4.973261562	17.3512692	29.70150887
Venus	Halogen	0.5	Before	Тор	5	64.41749865	9.205359151	52.9875356	75.8474617
Venus	Halogen	1	After	Bottom	5	28.88288462	1.730975396	26.73359478	31.03217445
Venus	Halogen	1	After	Тор	5	52.04878641	3.775771174	47.36054728	56.73702554
Venus	Halogen	1	Before	Bottom	5	32.8351065	4.505000711	27.24140931	38.4288037
Venus	Halogen	1	Before	Тор	5	64.69685609	1.585085233	62.72871282	66.66499936
Venus	Halogen	2	After	Bottom	5	30.95831865	2.238982307	28.17825492	33.73838237
Venus	Halogen	2	After	Тор	5	47.34261733	3.201853155	43.36699154	51.31824312
Venus	Halogen	2	Before	Bottom	5	31.86593778	7.62268425	22.40112517	41.33075038
Venus	Halogen	2	Before	Тор	5	61.55223505	5.305033277	54.96516622	68.13930388
Venus	LED	0.5	After	Bottom	5	26.44588842	2.781938425	22.99165563	29.9001212
Venus	LED	0.5	After	Тор	5	47.33072245	6.164696729	39.67624047	54.98520444
Venus	LED	0.5	Before	Bottom	5	23.37998546	2.017570473	20.87484084	25.88513008
Venus	LED	0.5	Before	Тор	5	58.07028394	3.830002261	53.31470802	62.82585987
Venus	LED	1	After	Bottom	5	31.373518	2.766862402	27.93800457	34.80903143
Venus	LED	1	After	Тор	5	52.90362914	1.864573085	50.58845587	55.21880241
Venus	LED	1	Before	Bottom	5	30.36426112	1.628818906	28.34181533	32.38670692
Venus	LED	1	Before	Тор	5	63.31725519	4.466528239	57.77132788	68.8631825
Venus	LED	2	After	Bottom	5	31.41772116	3.748806392	26.76296323	36.07247909
Venus	LED	2	After	Тор	5	48.08572517	2.202844976	45.35053187	50.82091847
Venus	LED	2	Before	Bottom	5	36.99689694	5.277851855	30.44357831	43.55021558
Venus	LED	2	Before	Тор	5	60.63974908	5.869657506	53.35160667	67.92789149
Z250	Halogen	0.5	After	Bottom	5	49.42361236	5.484210101	42.61406612	56.2331586
Z250	Halogen	0.5	After	Тор	5	67.37564458	8.076250376	57.34765524	77.40363391
Z250	Halogen	0.5	Before	Bottom	5	54.33097759	3.739124801	49.68824094	58.97371424
Z250	Halogen	0.5	Before	Тор	5	85.12150192	8.18167194	74.96261443	95.28038941
Z250	Halogen	1	After	Bottom	5	47.92340805	3.434872063	43.65845107	52.18836503
Z250	Halogen	1	After	Тор	5	62.37331007	11.56760341	48.01023337	76.73638678
Z250	Halogen	1	Before	Bottom	5	61.35787082	10.17907209	48.71888347	73.99685817
Z250	Halogen	1	Before	Тор	5	84.74872678	7.125387664	75.90138944	93.59606411
Z250	Halogen	2	After	Bottom	5	56.29466714	3.114404375	52.42762335	60.16171093
Z250	Halogen	2	After	Тор	5	73.4150587	3.865709039	68.61514696	78.21497044

									Anexo
Z250	Halogen	2	Before	Bottom	5	62.26842574	4.387456829	56.82067855	67.71617293
Z250	Halogen	2	Before	Тор	5	94.19240721	10.5888281	81.04464058	107.3401738
Z250	LED	0.5	After	Bottom	5	51.54897959	8.247540767	41.30830515	61.78965404
Z250	LED	0.5	After	Тор	5	63.33811991	4.00751169	58.36213692	68.3141029
Z250	LED	0.5	Before	Bottom	5	52.16018298	9.098213187	40.86325922	63.45710674
Z250	LED	0.5	Before	Тор	5	77.21289013	9.153329108	65.84753091	88.57824935
Z250	LED	1	After	Bottom	5	54.65316029	2.605619386	51.41785651	57.88846408
Z250	LED	1	After	Тор	5	71.46458916	1.694258489	69.36088939	73.56828893
Z250	LED	1	Before	Bottom	5	59.86657952	5.87048814	52.57740575	67.1557533
Z250	LED	1	Before	Тор	5	74.28097129	9.627129996	62.32731057	86.23463201
Z250	LED	2	After	Bottom	5	56.67650813	6.350917508	48.7908025	64.56221375
Z250	LED	2	After	Тор	5	68.96383539	4.981493124	62.77849472	75.14917605
Z250	LED	2	Before	Bottom	5	60.41786532	4.550494244	54.76768044	66.06805019
Z250	LED	2	Before	Тор	5	87.91012624	5.804689214	80.70265263	95.11759986

# KHN Bonf

Light	Resin	Wine	Surface	CureTime	_Light	_Resin	_Wine	_Surface	_CureTime
Halogen	Durafill	After	Bottom	0.5	Halogen	SuprA2D	After	Bottom	0.5
Halogen	Durafill	After	Bottom	0.5	Halogen	SuprA2E	After	Bottom	0.5
Halogen	Durafill	After	Bottom	0.5	Halogen	Z250	After	Bottom	0.5
Halogen	Durafill	After	Тор	0.5	Halogen	SuprA2D	After	Тор	0.5
Halogen	Durafill	After	Тор	0.5	Halogen	SuprA2E	After	Тор	0.5
Halogen	Durafill	After	Тор	0.5	Halogen	Venus	After	Тор	0.5
Halogen	Durafill	After	Тор	0.5	Halogen	Z250	After	Тор	0.5
Halogen	Durafill	Before	Bottom	0.5	Halogen	Durafill	Before	Тор	0.5
Halogen	Durafill	Before	Bottom	0.5	Halogen	SuprA2D	Before	Bottom	0.5
Halogen	Durafill	Before	Bottom	0.5	Halogen	SuprA2E	Before	Bottom	0.5
Halogen	Durafill	Before	Bottom	0.5	Halogen	Z250	Before	Bottom	0.5
Halogen	Durafill	Before	Тор	0.5	Halogen	SuprA2D	Before	Тор	0.5
Halogen	Durafill	Before	Тор	0.5	Halogen	SuprA2E	Before	Тор	0.5
Halogen	Durafill	Before	Тор	0.5	Halogen	Venus	Before	Тор	0.5
Halogen	Durafill	Before	Тор	0.5	Halogen	Z250	Before	Тор	0.5
Halogen	Durafill	After	Bottom	1	Halogen	SuprA2D	After	Bottom	1
Halogen	Durafill	After	Bottom	1	Halogen	SuprA2E	After	Bottom	1
Halogen	Durafill	After	Bottom	1	Halogen	Z250	After	Bottom	1
Halogen	Durafill	After	Тор	1	Halogen	SuprA2D	After	Тор	1
Halogen	Durafill	After	Тор	1	Halogen	SuprA2E	After	Тор	1
Halogen	Durafill	After	Тор	1	Halogen	Venus	After	Тор	1
Halogen	Durafill	After	Тор	1	Halogen	Z250	After	Тор	1
Halogen	Durafill	Before	Bottom	1	Halogen	SuprA2D	Before	Bottom	1
Halogen	Durafill	Before	Bottom	1	Halogen	SuprA2E	Before	Bottom	1
Halogen	Durafill	Before	Bottom	1	Halogen	Venus	Before	Bottom	1
Halogen	Durafill	Before	Bottom	1	Halogen	Z250	Before	Bottom	1
Halogen	Durafill	Before	Тор	1	Halogen	SuprA2D	Before	Тор	1
Halogen	Durafill	Before	Тор	1	Halogen	SuprA2E	Before	Тор	1
Halogen	Durafill	Before	Тор	1	Halogen	Venus	Before	Тор	1
Halogen	Durafill	Before	Тор	1	Halogen	Z250	Before	Тор	1
Halogen	Durafill	After	Bottom	2	Halogen	SuprA2D	After	Bottom	2
Halogen	Durafill	After	Bottom	2	Halogen	SuprA2E	After	Bottom	2
Halogen	Durafill	After	Bottom	2	Halogen	Z250	After	Bottom	2
Halogen	Durafill	After	Тор	2	Halogen	SuprA2D	After	Тор	2
Halogen	Durafill	After	Тор	2	Halogen	SuprA2E	After	Тор	2
Halogen	Durafill	After	Тор	2	Halogen	Z250	After	Тор	2

										Anexo
Halogen	Durafill	Before	Bottom	2	Halogen	Durafill	Before	Тор	2	
Halogen	Durafill	Before	Bottom	2	Halogen	SuprA2D	Before	Bottom	2	
Halogen	Durafill	Before	Bottom	2	Halogen	SuprA2E	Before	Bottom	2	
Halogen	Durafill	Before	Bottom	2	Halogen	Venus	Before	Bottom	2	
Halogen	Durafill	Before	Bottom	2	Halogen	Z250	Before	Bottom	2	
Halogen	Durafill	Before	Тор	2	Halogen	SuprA2D	Before	Тор	2	
Halogen	Durafill	Before	Тор	2	Halogen	SuprA2E	Before	Тор	2	
Halogen	Durafill	Before	Тор	2	Halogen	Venus	Before	Тор	2	
Halogen	Durafill	Before	Тор	2	Halogen	Z250	Before	Тор	2	
Halogen	SuprA2D	After	Bottom	0.5	Halogen	SuprA2D	After	Тор	0.5	
Halogen	SuprA2D	Before	Bottom	0.5	Halogen	SuprA2D	Before	Тор	0.5	
Halogen	SuprA2D	Before	Bottom	0.5	Halogen	Venus	Before	Bottom	0.5	
Halogen	SuprA2D	After	Bottom	1	Halogen	SuprA2D	After	Тор	1	
Halogen	SuprA2D	After	Тор	1	Halogen	Venus	After	Тор	1	
Halogen	SuprA2D	Before	Bottom	1	Halogen	SuprA2D	Before	Тор	1	
Halogen	SuprA2D	After	Bottom	2	Halogen	SuprA2D	After	Тор	2	
Halogen	SuprA2D	After	Bottom	2	Halogen	Venus	After	Bottom	2	
Halogen	SuprA2D	After	Тор	2	Halogen	SuprA2D	Before	Тор	2	
Halogen	SuprA2D	After	Тор	2	Halogen	Venus	After	Тор	2	
Halogen	SuprA2D	Before	Bottom	2	Halogen	SuprA2D	Before	Тор	2	
Halogen	SuprA2D	Before	Bottom	2	Halogen	Venus	Before	Bottom	2	
Halogen	SuprA2D	Before	Тор	2	Halogen	Venus	Before	Тор	2	
Halogen	SuprA2E	After	Bottom	0.5	Halogen	SuprA2E	After	Тор	0.5	
Halogen	SuprA2E	After	Bottom	0.5	Halogen	Venus	After	Bottom	0.5	
Halogen	SuprA2E	Before	Bottom	0.5	Halogen	SuprA2E	Before	Тор	0.5	
Halogen	SuprA2E	After	Bottom	1	Halogen	SuprA2E	After	Тор	1	
Halogen	SuprA2E	After	Bottom	1	Halogen	Venus	After	Bottom	1	
Halogen	SuprA2E	After	Тор	1	Halogen	Venus	After	Тор	1	
Halogen	SuprA2E	Before	Bottom	1	Halogen	SuprA2E	Before	Тор	1	
Halogen	SuprA2E	After	Bottom	2	Halogen	SuprA2E	After	Тор	2	
Halogen	SuprA2E	After	Тор	2	Halogen	Venus	After	Тор	2	
Halogen	SuprA2E	Before	Bottom	2	Halogen	SuprA2E	Before	Тор	2	
Halogen	SuprA2E	Before	Bottom	2	Halogen	Venus	Before	Bottom	2	
Halogen	Venus	After	Bottom	0.5	Halogen	Venus	After	Тор	0.5	
Halogen	Venus	After	Bottom	0.5	Halogen	Z250	After	Bottom	0.5	
Halogen	Venus	Before	Bottom	0.5	Halogen	Venus	Before	Тор	0.5	
Halogen	Venus	Before	Bottom	0.5	Halogen	Z250	Before	Bottom	0.5	
Halogen	Venus	Before	Тор	0.5	Halogen	Z250	Before	Тор	0.5	
Halogen	Venus	After	Bottom	1	Halogen	Venus	After	Тор	1	
Halogen	Venus	After	Bottom	1	Halogen	Z250	After	Bottom	1	
Halogen	Venus	Before	Bottom	1	Halogen	Venus	Before	Тор	1	
Halogen	Venus	Before	Bottom	1	Halogen	Z250	Before	Bottom	1	
Halogen	Venus	Before	Тор	1	Halogen	Z250	Before	Тор	1	
Halogen	Venus	After	Bottom	2	Halogen	Venus	After	Тор	2	
Halogen	Venus	After	Bottom	2	Halogen	Z250	After	Bottom	2	
Halogen	Venus	After	Тор	2	Halogen	Z250	After	Тор	2	
Halogen	Venus	Before	Bottom	2	Halogen	Venus	Before	Тор	2	
Halogen	Venus	Before	Bottom	2	Halogen	Z250	Before	Bottom	2	
Halogen	Venus	Before	Тор	2	Halogen	Z250	Before	Тор	2	
Halogen	Z250	After	Bottom	0.5	Halogen	Z250	After	Тор	0.5	
Halogen	Z250	After	Тор	0.5	Halogen	Z250	Before	Тор	0.5	
Halogen	Z250	Before	Bottom	0.5	Halogen	Z250	Before	Тор	0.5	
Halogen	Z250	After	Тор	1	Halogen	Z250	Before	Тор	1	
Halogen	Z250	Before	Bottom	1	Halogen	Z250	Before	Тор	1	

										Anexo
Halogen	Z250	After	Bottom	2	Halogen	Z250	After	Тор	2	
Halogen	Z250	After	Тор	2	Halogen	Z250	Before	Тор	2	
Halogen	Z250	Before	Bottom	2	Halogen	Z250	Before	Тор	2	
LED	Durafill	After	Bottom	0.5	LED	SuprA2D	After	Bottom	0.5	
LED	Durafill	After	Bottom	0.5	LED	SuprA2E	After	Bottom	0.5	
LED	Durafill	After	Bottom	0.5	LED	Z250	After	Bottom	0.5	
LED	Durafill	After	Тор	0.5	LED	SuprA2D	After	Тор	0.5	
LED	Durafill	After	Тор	0.5	LED	SuprA2E	After	Тор	0.5	
LED	Durafill	After	Тор	0.5	LED	Venus	After	Тор	0.5	
LED	Durafill	After	Тор	0.5	LED	Z250	After	Тор	0.5	
LED	Durafill	Before	Bottom	0.5	LED	SuprA2D	Before	Bottom	0.5	
LED	Durafill	Before	Bottom	0.5	LED	SuprA2E	Before	Bottom	0.5	
LED	Durafill	Before	Bottom	0.5	LED	Z250	Before	Bottom	0.5	
LED	Durafill	Before	Тор	0.5	LED	SuprA2D	Before	Тор	0.5	
LED	Durafill	Before	Тор	0.5	LED	SuprA2E	Before	Тор	0.5	
LED	Durafill	Before	Тор	0.5	LED	Venus	Before	Тор	0.5	
LED	Durafill	Before	Тор	0.5	LED	Z250	Before	Тор	0.5	
LED	Durafill	After	Bottom	1	LED	SuprA2D	After	Bottom	1	
LED	Durafill	After	Bottom	1	LED	SuprA2E	After	Bottom	1	
LED	Durafill	After	Bottom	1	LED	Venus	After	Bottom	1	
LED	Durafill	After	Bottom	1	LED	Z250	After	Bottom	1	
LED	Durafill	After	Top	1	LED	SuprA2D	After	Top	1	
LED	Durafill	After	Top	- 1	LED	SuprA2E	After	Top	1	
LED	Durafill	After	Top	1	LED	Venus	After	Top	1	
LED	Durafill	After	Top	1	LED	7250	After	Top	1	
LED	Durafill	Before	Bottom	1	LED	Durafill	Before	Top	1	
LED	Durafill	Before	Bottom	1	LED	SuprA2D	Before	Bottom	1	
LED	Durafill	Before	Bottom	1	LED	SuprA2E	Before	Bottom	1	
LED I FD	Durafill	Before	Bottom	1	LED I FD	Venus	Before	Bottom	1	
LED I FD	Durafill	Before	Bottom	1	LED I FD	7250	Before	Bottom	1	
	Durafill	Before	Ton	1		SuprA2D	Before	Ton	1	
	Durafill	Before	Top	1		SuprA2D	Before	Top	1	
	Durafill	Before	Тор	1		SuprA2E Vopus	Before	Top	1	
	Durafill	Defore	Top	1		7250	Defore	Top	1	
	Durafill	After	Pottom	1		2230 Supr A 2D	After	Pottom	1	
	Durafill	After	Dottom	2		SuprA2D	After	Dottom	2	
	Durann	After	Dottom	2		SuprA2E	After	Dottom	2	
	Duranni Durafill	After	Bottom	2		venus	After	Bottom Dettern	2	
	Duranni Durafill	After	Bottom	2		Z250	After	Bottom	2	
	Duranii	Atter	тор	2	LED	SuprA2D	After	тор	2	
LED	Durafill	After	Тор	2	LED	SuprA2E	After	Тор	2	
LED	Durafill	Atter	Тор	2	LED	venus	Atter	Top	2	
LED	Durafill	After	Top	2	LED	Z250	After	Top	2	
LED	Durafill	Before	Bottom	2	LED	SuprA2D	Before	Bottom	2	
LED	Durafill	Before	Bottom	2	LED	SuprA2E	Before	Bottom	2	
LED	Durafill	Before	Bottom	2	LED	Venus	Before	Bottom	2	
LED	Durafill	Before	Bottom	2	LED	Z250	Before	Bottom	2	
LED	Durafill	Before	Тор	2	LED	SuprA2D	Before	Тор	2	
LED	Durafill	Before	Тор	2	LED	SuprA2E	Before	Тор	2	
LED	Durafill	Before	Тор	2	LED	Venus	Before	Тор	2	
LED	Durafill	Before	Тор	2	LED	Z250	Before	Тор	2	
LED	SuprA2D	After	Bottom	0.5	LED	SuprA2D	After	Тор	0.5	
LED	SuprA2D	After	Тор	0.5	LED	SuprA2D	Before	Тор	0.5	
LED	SuprA2D	After	Тор	0.5	LED	Venus	After	Тор	0.5	
LED	SuprA2D	Before	Bottom	0.5	LED	SuprA2D	Before	Тор	0.5	

\_

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LED	SuprA2D	Before	Тор	0.5	LED	Venus	Before	Тор	0.5	
LED	SuprA2D	After	Bottom	1	LED	SuprA2D	After	Тор	1	
LED	SuprA2D	Before	Bottom	1	LED	SuprA2D	Before	Тор	1	
LED	SuprA2D	After	Bottom	2	LED	Venus	After	Bottom	2	
LED	SuprA2D	After	Тор	2	LED	Venus	After	Тор	2	
LED	SuprA2D	Before	Bottom	2	LED	SuprA2D	Before	Тор	2	
LED	SuprA2D	Before	Bottom	2	LED	Venus	Before	Bottom	2	
LED	SuprA2D	Before	Тор	2	LED	Venus	Before	Тор	2	
LED	SuprA2E	After	Bottom	0.5	LED	SuprA2E	After	Тор	0.5	
LED	SuprA2E	After	Тор	0.5	LED	SuprA2E	Before	Тор	0.5	
LED	SuprA2E	After	Тор	0.5	LED	Venus	After	Тор	0.5	
LED	SuprA2E	Before	Bottom	0.5	LED	SuprA2E	Before	Тор	0.5	
LED	SuprA2E	Before	Bottom	0.5	LED	Venus	Before	Bottom	0.5	
LED	SuprA2E	Before	Тор	0.5	LED	Venus	Before	Тор	0.5	
LED	SuprA2E	After	Bottom	1	LED	SuprA2E	After	Тор	1	
LED	SuprA2E	After	Тор	1	LED	SuprA2E	Before	Тор	1	
LED	SuprA2E	Before	Bottom	1	LED	SuprA2E	Before	Тор	1	
LED	SuprA2E	Before	Тор	1	LED	Venus	Before	Тор	1	
LED	SuprA2E	Before	Тор	1	LED	Z250	Before	Тор	1	
LED	SuprA2E	After	Bottom	2	LED	SuprA2E	After	Тор	2	
LED	SuprA2E	After	Тор	2	LED	SuprA2E	Before	Тор	2	
LED	SuprA2E	Before	Bottom	2	LED	SuprA2E	Before	Тор	2	
LED	SuprA2E	Before	Тор	2	LED	Venus	Before	Тор	2	
LED	Venus	After	Bottom	0.5	LED	Venus	After	Тор	0.5	
LED	Venus	After	Bottom	0.5	LED	Z250	After	Bottom	0.5	
LED	Venus	Before	Bottom	0.5	LED	Venus	Before	Тор	0.5	
LED	Venus	Before	Bottom	0.5	LED	Z250	Before	Bottom	0.5	
LED	Venus	Before	Тор	0.5	LED	Z250	Before	Тор	0.5	
LED	Venus	After	Bottom	1	LED	Venus	After	Тор	1	
LED	Venus	After	Bottom	1	LED	Z250	After	Bottom	1	
LED	Venus	After	Тор	1	LED	Z250	After	Тор	1	
LED	Venus	Before	Bottom	1	LED	Venus	Before	Тор	1	
LED	Venus	Before	Bottom	1	LED	Z250	Before	Bottom	1	
LED	Venus	After	Bottom	2	LED	Venus	After	Тор	2	
LED	Venus	After	Bottom	2	LED	Z250	After	Bottom	2	
LED	Venus	After	Тор	2	LED	Z250	After	Тор	2	
LED	Venus	Before	Bottom	2	LED	Venus	Before	Тор	2	
LED	Venus	Before	Bottom	2	LED	Z250	Before	Bottom	2	
LED	Venus	Before	Тор	2	LED	Z250	Before	Тор	2	
LED	Z250	Before	Bottom	0.5	LED	Z250	Before	Тор	0.5	
LED	Z250	After	Bottom	1	LED	Z250	After	Тор	1	
LED	Z250	After	Тор	2	LED	Z250	Before	Тор	2	
LED	Z250	Before	Bottom	2	LED	Z250	Before	Тор	2	
Halogen	SuprA2E	Before	Bottom	0.5	Halogen	Venus	Before	Bottom	0.5	
Halogen	SuprA2E	Before	Bottom	1	Halogen	Venus	Before	Bottom	1	
Halogen	SuprA2E	Before	Тор	2	Halogen	Z250	Before	Тор	2	
Halogen	Venus	After	Тор	0.5	Halogen	Z250	After	Тор	0.5	
LED	SuprA2E	Before	Bottom	1	LED	Venus	Before	Bottom	1	

# E) Teste de Correlação de Pearson

P:\ClaudiaBatitucci-Santos\KHNvsDC.SAS: 090CT09:14:36:14 1								
The CORF	R Procedure							
2 Va	ariables:	DC KHN						
		51	mple statisti	CS				
Variable	e N	Mean	Std Dev	Sum	Minimum	Maximum		
DC KHN	300 300	52.64816 53.57258	6.05861 25.09148	15794 16072	34.30873 9.04385	76.57005 118.31931		
Simple S	Statistics							
Variable	e Label							
DC KHN	DC KHN							
Pearson Correlation Coefficients, N = 300 Prob >  r  under HO: Rho=0								
	DC	KHN						
DC DC	1.00000	0.53651 <.0001						
KHN KHN	0.53651 <.0001	1.00000						

2

Maximum

59.44388

39.69636

P:\ClaudiaBatitucci-Santos\KHNvsDC.SAS: 090CT09:14:36:14 Resin=Durafill Light=Halogen The CORR Procedure 2 Variables: DC KHN Simple Statistics Variable Std Dev Minimum Ν Mean Sum 52.33322 21.69204 DC 30 4.67685 1570 38.71724 30 650.76127 KHN 9.66520 10.22792 Simple Statistics Variable Label DC DC KHN KHN Pearson Correlation Coefficients, N = 30 Prob > |r| under H0: Rho=0 DC KHN DC 1.00000 0.44384 0.0140 DC KHN 0.44384 1.00000 0.0140 KHN

3

Maximum

59.39751

41.72803

P:\ClaudiaBatitucci-Santos\KHNvsDC.SAS: 090CT09:14:36:14 Resin=Durafill Light=LED The CORR Procedure 2 Variables: DC KHN Simple Statistics Variable Std Dev Minimum Ν Mean Sum 50.32309 19.97955 DC 30 4.95705 1510 34.30873 599.38643 KHN 30 8.61330 9.04385 Simple Statistics Variable Label DC DC KHN KHN Pearson Correlation Coefficients, N = 30 Prob > |r| under H0: Rho=0 DC KHN DC 1.00000 -0.24847 DC 0.1855 KHN -0.24847 1.00000 0.1855 KHN

4

P:\ClaudiaBatitucci-Santos\KHNvsDC.SAS: 090CT09:14:36:14

# Resin=SuprA2D Light=Halogen

# The CORR Procedure

2 Variables: DC KHN

Simple Statistics

Variable		Ν	Меа	an S	Std Dev	Sum	Minimu	m Maximum
DC KHN		30 30	52.3023 63.4401	32 7 L4 19	7.67209 0.11409	1569 1903	37.1500 37.4227	9 62.40071 5 101.05258
Simple Statistics								
Variable	Variable Label							
DC KHN	DC KHN							
Pearson Correlation Coefficients, N = 30 Prob >  r  under HO: Rho=0								
	DC	2	ŀ	(HN				

DC DC	1.00000	0.85770 <.0001
KHN KHN	0.85770 <.0001	1.00000

5

Maximum

P:\ClaudiaBatitucci-Santos\KHNvsDC.SAS: 090CT09:14:36:14 Resin=SuprA2D Light=LED The CORR Procedure 2 Variables: DC KHN Simple Statistics Variable Std Dev Minimum Ν Mean Sum DC 30 52.52404 5.59979 1576 42.37018 60.35068 30 64.34686 100.48725 KHN 19.41683 1930 38.20237 Simple Statistics Variable Label DC DC KHN KHN Pearson Correlation Coefficients, N = 30 Prob > |r| under H0: Rho=0 DC KHN DC 1.00000 0.89320 <.0001 DC KHN 0.89320 1.00000 <.0001 KHN

6

P:\ClaudiaBatitucci-Santos\KHNvsDC.SAS: 090CT09:14:36:14

## Resin=SuprA2E Light=Halogen

### The CORR Procedure

2 Variables: DC KHN

Simple Statistics

Variable		Ν	Mean	Std Dev	Sum	Minimum	Maximum
DC KHN		30 30	52.76463 63.13261	7.92755 19.49072	1583 1894	39.40530 30.40591	76.57005 99.37074
Simple St	atistics	5					
Variable	Label						
DC	DC						

KHN KHN

## Pearson Correlation Coefficients, N = 30 Prob > |r| under H0: Rho=0

	DC	KHN
DC DC	1.00000	0.81405 <.0001
KHN KHN	0.81405 <.0001	1.00000
7

Maximum

P:\ClaudiaBatitucci-Santos\KHNvsDC.SAS: 090CT09:14:36:14 Resin=SuprA2E Light=LED The CORR Procedure 2 Variables: DC KHN Simple Statistics Variable Minimum Std Dev Ν Mean Sum 63.18150 118.31931 DC 30 50.50811 6.07938 1515 40.41655 30 68.87940 KHN 23.58336 2066 30.87989 Simple Statistics Variable Label DC DC KHN KHN Pearson Correlation Coefficients, N = 30 Prob > |r| under H0: Rho=0 DC KHN DC 1.00000 0.86649 DC <.0001 KHN 0.86649 1.00000 KHN <.0001

8

P:\ClaudiaBatitucci-Santos\KHNvsDC.SAS: 090CT09:14:36:14

# Resin=Venus Light=Halogen

# The CORR Procedure

2 Variables: DC KHN

Simple Statistics

Variable	Ν	Mean	Std Dev	Sum	Minimum	Maximum
DC KHN	30 30	54.85425 46.48234	5.69351 18.48683	1646 1394	44.81293 16.20481	62.76853 74.72170

Simple Statistics

Variable Label

DC DC KHN KHN

# Pearson Correlation Coefficients, N = 30 Prob > |r| under H0: Rho=0

	DC	KHN
DC DC	1.00000	0.84844 <.0001
KHN KHN	0.84844 <.0001	1.00000

9

Maximum

61.83491

70.24116

P:\ClaudiaBatitucci-Santos\KHNvsDC.SAS: 090CT09:14:36:14 Resin=Venus Light=LED The CORR Procedure 2 Variables: DC KHN Simple Statistics Variable Std Dev Minimum Ν Mean Sum 5.66253 16.49597 DC 30 53.76641 1613 37.91454 30 45.46141 20.57280 KHN 1364 Simple Statistics Variable Label DC DC KHN KHN Pearson Correlation Coefficients, N = 30 Prob > |r| under H0: Rho=0 DC KHN DC 1.00000 0.85796 DC <.0001 KHN 0.85796 1.00000

<.0001

KHN

10

P:\ClaudiaBatitucci-Santos\KHNvsDC.SAS: 090CT09:14:36:14 Resin=Z250 Light=Halogen The CORR Procedure

2 Variables: DC KHN

Simple Statistics

Variable	1	N N	Mean	Std Dev	Sum	Minimum	Maximum
DC KHN	30 30	) 53.10 ) 73.60	0806 6999 1	5.68807 6.73304	1593 2210	43.68404 49.62722	60.59965 107.59924
Simple Sta	tistics						
Variable	Label						
DC KHN	DC KHN						
Pearson Co Pro	orrelatio b >  r	on Coeffic under HO:	cients, N : Rho=0	= 30			
	DC		KHN				

DC DC	1.00000	0.80032 <.0001
KHN KHN	0.80032 <.0001	1.00000

11

Maximum

P:\ClaudiaBatitucci-Santos\KHNvsDC.SAS: 090CT09:14:36:14 Resin=Z250 Light=LED The CORR Procedure 2 Variables: DC KHN Simple Statistics Variable Std Dev Minimum Ν Mean Sum 5.11264 14.21084 DC 30 53.99748 1620 45.13260 62.59542 68.64144 95.08572 KHN 30 2059 38.07075 Simple Statistics Variable Label DC DC KHN KHN Pearson Correlation Coefficients, N = 30 Prob > |r| under H0: Rho=0 DC KHN DC 1.00000 0.74813 <.0001 DC KHN 0.74813 1.00000

<.0001

KHN

Anexo	,
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12 P:\ClaudiaBatitucci-Santos\KHNvsDC.SAS: 090CT09:14:36:14 The GLM Procedure Class Level Information Levels Class Values Resin 5 Durafill SuprA2D SuprA2E Venus Z250 Light 2 Halogen LED Number of Observations Read 300 Number of Observations Used 300 The GLM Procedure Dependent Variable: KHN KHN Sum of Source DF Squares Mean Square F Value Pr > FMode1 19 160947.4615 86.89 <.0001 8470.9190 Error 280 27297.6279 97.4915 Corrected Total 299 188245.0894 R-Square Coeff Var Root MSE KHN Mean 0.854989 18.43066 9.873780 53.57258 Pr > F Source DF Type III SS Mean Square F Value 40427.34218 882.50162 1624.55046 1 40427.34218 414.68 <.0001 DC Resin 4 3530.00647 9.05 <.0001 DC\*Resin 6498.20184 16.66 4 <.0001 9.63415 Light 1 9.63415 0.10 0.7535 18.20589 18.20589 0.19 0.6660 DC\*Light 1 Resin<sup>\*</sup>Light 4 1825.65929 456.41482 0.0011 4.68 DC\*Resin<sup>\*</sup>Light 4 2211.06873 552.76718 5.67 0.0002

P:\ClaudiaBa	atitucci-Sar	ntos\KHNvs	DC.SAS:	090СТ0	9:14:36:14		14
The GLM Proc	cedure						
	Class	5 Level Ir	formati	on			
Class	Levels	Values					
Resin	5	Durafill	SuprA2	D SuprA	2E Venus Z250		
Light	2	Halogen	LED				
Number of Ol Number of Ol	oservations oservations	Read Used	300 300				
The GLM Prod	cedure						
Dependent Va	ariable: DC	DC					
Source		DF	Sq Sq	um of uares	Mean Square	F Value	Pr > F
Model		19	7100.	57660	373.71456	27.01	<.0001
Error		280	3874.	74311	13.83837		
Corrected To	otal	299	10975.	31971			
R-Square	Coeff Var	Root	: MSE	DC	Mean		
0.646959	7.065766	3.71	9996	52.6	4816		
Source		DF	Туре І	II SS	Mean Square	F Value	Pr > F
KHN Resin KHN*Resin Light KHN*Light Resin*Light KHN*Resin*L	ight	1 4 1 1 4 4	3191.2 1559.6 285.7 50.9 160.7 74.4 172.2	53983 23071 93021 27276 60576 15624 35747	3191.253983 389.905768 71.448255 50.927276 160.760576 18.603906 43.058937	$230.61 \\ 28.18 \\ 5.16 \\ 3.68 \\ 11.62 \\ 1.34 \\ 3.11$	<.0001 <.0001 0.0005 0.0561 0.0007 0.2537 0.0158

1

# F) Rugosidade Superficial

P:\ClaudiaBatitucci-Santos\RAdata.sas: 03SEP09:16:37:29

The Mixed Procedure

Model Information

Data Set	WORK.RA
Dependent Variable	Ra
Covariance Structure	Compound Symmetry
Subject Effect	Samp*Ligh*Resi*CureT
Estimation Method	REML
Residual Variance Method	Profile
Fixed Effects SE Method	Model-Based
Degrees of Freedom Method	Satterthwaite

# Class Level Information

Class	Levels	Values
Sample	5	12345 Halogen LED
Resin	5	Durafill SuprA2D SuprA2E Venus
CureTime	3	Z250 0.5 1 2
Wine	2	After Before Bottom Top
DULTACE	2	Doccom Tob

#### Dimensions

Covariance	Parameters	2
Columns in	Х	648
Columns in	Z	0
Subjects		150
Max Obs Per	Subject	4

#### Number of Observations

Number	of	Observations	Read	599
Number	of	Observations	Used	599
Number	of	Observations	Not Used	0

#### Iteration History

Iteration	Evaluations	-2 Res Log Like	Criterion
0	1	-2273.77529892	
1	2	-2308.95957094	0.0000000

# Convergence criteria met.

Covariance Parameter Estimates

Cov Parm	Subject	Estimate

CS Samp\*Ligh\*Resi\*CureT 0.000084 Residual 0.000256

#### Fit Statistics

-2 Res Log Likelihood	-2309.0
AIC (smaller is better)	-2305.0
AICC (smaller is better)	-2304.9
BIC (smaller is better)	-2298.9

Null Model Likelihood Ratio Test

DF	Chi-Square	Pr > ChiSq
1	35.18	<.0001

# Type 3 Tests of Fixed Effects

	Num	Den		
Effect	DF	DF	F Value	Pr > F
Light	1	119	2.03	0.1570
Resin	4	119	51.64	<.0001
CureTime	2	119	4.73	0.0106
Surface	1	359	34.23	<.0001
Wine	1	359	14.52	0.0002
Light*Resin	4	119	1.34	0.2583
Light*CureTime	2	119	0.13	0.8791
Light*Surface	1	359	2.37	0.1244
Light*Wine	1	359	0.01	0.9317
Resin*CureTime	8	119	2.84	0.0063
Resin*Surface	4	359	65.42	<.0001
Resin*Wine	4	359	15.67	<.0001
CureTime*Surface	2	359	5.04	0.0069
CureTime*Wine	2	359	2.34	0.0979
Wine*Surface	1	359	5.19	0.0234
Light*Resin*CureTime	8	119	0.61	0.7648
Light*Resin*Surface	4	359	2.46	0.0449
Light*Resin*Wine	4	359	1.63	0.1668
Light*CureTi*Surface	2	359	0.77	0.4655
Light*CureTime*Wine	2	359	7.29	0.0008
Light*Wine*Surface	1	359	0.11	0.7437
Resin*CureTi*Surface	8	359	4.41	<.0001
Resin*CureTime*Wine	8	359	1.99	0.0470
Resin*Wine*Surface	4	359	2.89	0.0225
CureTim*Wine*Surface	2	359	1.30	0.2730
Ligh*Resi*Cure*Surfa	8	359	1.00	0.4353
Ligh*Resi*CureT*Wine	8	359	1.40	0.1950
Ligh*Resi*Wine*Surfa	4	359	0.38	0.8232
Ligh*Cure*Wine*Surfa	2	359	0.95	0.3879
Resi*Cure*Wine*Surfa	8	359	0.44	0.8994
Lig*Res*Cur*Win*Surf	8	359	0.35	0.9440

2

P:\ClaudiaBatitucci-Santos\RAdata.sas: 03SEP09:16:37:29 These comparisons have been adjusted using the Bonferroni correction.

					С					
				S	u				S	u
				11	r				11	r
	L	R		r	e	L	 R		r	ē
	i	e	W	f	Т	i	e	W	f	Т
0	a	s	i	a	i	a	s	i	- a	i
b	9 h	i	n	C	m	9 h	i	n	C	m
ŝ	+	r	 A	0		+	r	 A	0	
U	C		C	0	C	C		C	6	C
1	Halogen	Durafill	After	Bottom	0.5	Halogen	Durafill	After	Тор	0.5
2	Halogen	Durafill	After	Bottom	0.5	Halogen	Durafill	After	Bottom	2.0
3	Halogen	Durafill	After	Bottom	0.5	Halogen	SuprA2D	After	Bottom	0.5
4	Halogen	Durafill	After	Bottom	0.5	Halogen	SuprA2E	After	Bottom	0.5
5	Halogen	Durafill	After	Bottom	0.5	Halogen	Venus	After	Bottom	0.5
6	Halogen	Durafill	After	Bottom	0.5	Halogen	Z250	After	Bottom	0.5
7	Halogen	Durafill	Before	Bottom	0.5	Halogen	Durafill	Before	τορ	0.5
8	Halogen	Durafill	Before	Bottom	0.5	Halogen	Durafill	Before	Bottom	1.0
9	Halogen	Durafill	Before	Bottom	0.5	Halogen	Durafill	Before	Bottom	2.0
10	Halogen	Durafill	Before	Bottom	0.5	Halogen	SuprA2D	Before	Bottom	0.5
11	Halogen	Durafill	Before	Bottom	0.5	Halogen	SuprA2E	Before	Bottom	0.5
12	Halogen	Durafill	Before	Bottom	0.5	Halogen	Venus	Before	Bottom	0.5
1.3	Halogen	Durafill	Before	Bottom	0.5	Halogen	7250	Before	Bottom	0.5
14	Halogen	Durafill	After	Bottom	1.0	Halogen	Durafill	After	Top	1.0
15	Halogen	Durafill	After	Bottom	1.0	Halogen	Durafill	Before	Bottom	1.0
16	Halogen	Durafill	After	Bottom	1.0	Halogen	SuprA2D	After	Bottom	1.0
17	Halogen	Durafill	After	Bottom	1.0	Halogen	SuprA2E	After	Bottom	1.0
18	Halogen	Durafill	After	Bottom	1.0	Halogen	Venus	After	Bottom	1.0
19	Halogen	Durafill	After	Bottom	1 0	Halogen	7250	After	Bottom	1 0
20	Halogen	Durafill	After	Bottom	2.0	Halogen	Durafill	After	Тор	2.0
21	Halogen	Durafill	After	Bottom	2 0	Halogen	SuprA2D	After	Bottom	2 0
22	Halogen	Durafill	After	Bottom	2 0	Halogen	SuprA2E	After	Bottom	2 0
23	Halogen	Durafill	After	Bottom	2 0	Halogen	Venus	After	Bottom	2 0
24	Halogen	Durafill	After	Bottom	2 0	Halogen	7250	After	Bottom	2 0
25	LED	Durafill	After	Bottom	0 5	LED	Durafill	After	Top	0 5
26	LED	Durafill	After	Bottom	0.5	LED	Durafill	Before	Bottom	0.5
27	LED	Durafill	Aftor	Bottom	0.5	LED	Durafill	Aftor	Bottom	1 0
28	LED	Durafill	Aftor	Bottom	0.5	LED	Durafill	After	Bottom	2 0
29	LED	Durafill	Aftor	Bottom	0.5	LED	Supra2D	Aftor	Bottom	0 5
30	LED	Durafill	Aftor	Bottom	0.5	LED	SuprA2F	After	Bottom	0.5
30	IED	Durafill	Alter	Bottom	0.5	IED	Venus	Alter	Bottom	0.5
32	IED	Durafill	Alter	Bottom	0.5	IED	7250	Alter	Bottom	0.5
32		Durafill	Roforo	Bottom	0.5		Durafill	Roforo	Top	0.5
31		Durafill	Poforo	Bottom	0.5		Suprach	Poforo	Pottom	0.5
24 25	רם ד חם ד	Durafili	Derore	Bottom	0.5	רם ד חם ד	SUPIAZD	Derore	Bottom	0.5
35	תקת תקת	Durafili	Berore	Bottom Rottom	0.5	תקת תקת	VODUC	Berore	Bottom Bottom	0.5
20	רם ז חיזים	Durafill	Before	Bottom	0.5	חם ז חיזים	venus 7250	Before	Bottom	0.5
ン1 20	U E D		Derore	Dottom	1 0	U E D		Derore	Dottom	1 0
Sб	лдп	Duralitt	ALLET	BULTOM	τ.Ο	п₽Л	SUPTAZD	ALLET	BOLTOM	τ.υ

3

P:\ClaudiaBatitucci-Santos\RAdata.sas: 03SEP09:16:37:29 These comparisons have been adjusted using the Bonferroni correction.

					С				_	C
				S	u				S	u
				u	r	_	_		u	r
	L	R		r	е	L	R	_	r	е
	i	е	W	f	Т	i	е	W	f	Т
0	g	S	i	a	i	g	S	i	a	i
b	h	i	n	С	m	h	i	n	С	m
S	t	n	е	е	е	t	n	е	е	е
39	LED	Durafill	After	Bottom	1.0	LED	SuprA2E	After	Bottom	1.0
40	LED	Durafill	After	Bottom	1.0	LED	Venus	After	Bottom	1.0
41	LED	Durafill	After	Bottom	1.0	LED	Z250	After	Bottom	1.0
42	LED	Durafill	After	Bottom	2.0	LED	Durafill	After	Тор	2.0
43	LED	Durafill	After	Bottom	2.0	LED	SuprA2D	After	Bottom	2.0
44	LED	Durafill	After	Bottom	2.0	LED	SuprA2E	After	Bottom	2.0
45	LED	Durafill	After	Bottom	2.0	LED	Venus	After	Bottom	2.0
46	LED	Durafill	After	Bottom	2.0	LED	Z250	After	Bottom	2.0
47	LED	SuprA2D	After	Bottom	0.5	LED	SuprA2E	After	Bottom	0.5
48	LED	Durafill	Before	Bottom	1.0	LED	Z250	Before	Bottom	1.0

Resin	Light	CureTime	Surface	Wine	Ra_N	Ra_Mean	Ra_StdDev	Ra_UCLM	Ra_LCLM
Durafill	Halogen	0.5	Bottom	After	5	0.174493	0.05218	0.239283	0.109704
Durafill	Halogen	0.5	Bottom	Before	5	0.14658	0.009468	0.158336	0.134824
Durafill	Halogen	0.5	Тор	After	5	0.076047	0.00458	0.081734	0.07036
Durafill	Halogen	0.5	Тор	Before	5	0.072127	0.014216	0.089778	0.054475
Durafill	Halogen	1	Bottom	After	5	0.14706	0.034343	0.189703	0.104417
Durafill	Halogen	1	Bottom	Before	5	0.099033	0.029103	0.135169	0.062897
Durafill	Halogen	1	Тор	After	5	0.08508	0.010916	0.098633	0.071527
Durafill	Halogen	1	Тор	Before	5	0.062093	0.005111	0.068439	0.055747
Durafill	Halogen	2	Bottom	After	5	0.1263	0.055534	0.195254	0.057346
Durafill	Halogen	2	Bottom	Before	5	0.09236	0.012258	0.10758	0.07714
Durafill	Halogen	2	Тор	After	4	0.063608	0.005572	0.072474	0.054742
Durafill	Halogen	2	Тор	Before	5	0.064293	0.006478	0.072337	0.05625
Durafill	LED	0.5	Bottom	After	5	0.18162	0.041018	0.232551	0.130689
Durafill	LED	0.5	Bottom	Before	5	0.127167	0.026439	0.159995	0.094338
Durafill	LED	0.5	Тор	After	5	0.097227	0.016762	0.118039	0.076414
Durafill	LED	0.5	Тор	Before	5	0.081007	0.009184	0.09241	0.069604
Durafill	LED	1	Bottom	After	5	0.1205	0.034809	0.163721	0.077279
Durafill	LED	1	Bottom	Before	5	0.108887	0.019788	0.133457	0.084316
Durafill	LED	1	Тор	After	5	0.102873	0.024454	0.133237	0.072509
Durafill	LED	1	Тор	Before	5	0.082	0.016995	0.103102	0.060898
Durafill	LED	2	Bottom	After	5	0.131807	0.026458	0.164659	0.098954
Durafill	LED	2	Bottom	Before	5	0.099087	0.017748	0.121123	0.07705
Durafill	LED	2	Тор	After	5	0.087007	0.012149	0.102092	0.071921
Durafill	LED	2	Тор	Before	5	0.082047	0.00885	0.093035	0.071058
SuprA2D	Halogen	0.5	Bottom	After	5	0.066553	0.012477	0.082046	0.051061
SuprA2D	Halogen	0.5	Bottom	Before	5	0.07304	0.005819	0.080265	0.065815
SuprA2D	Halogen	0.5	Тор	After	5	0.07236	0.00779	0.082033	0.062687
SuprA2D	Halogen	0.5	Тор	Before	5	0.076747	0.014157	0.094325	0.059169
SuprA2D	Halogen	1	Bottom	After	5	0.06402	0.006095	0.071588	0.056452
SuprA2D	Halogen	1	Bottom	Before	5	0.06514	0.010045	0.077612	0.052668

SuprAD	Halogen	1	Ton	After	5	0.062147	0.002521	0.065290	0.050005
SuprA2D	Halogen	1	Top	Atter	5	0.064652	0.002551	0.065289	0.059005
SuprA2D	Halogen	1	тор Важал	After	5	0.004033	0.003703	0.071809	0.037498
SuprA2D	Halogen	2	Dottom	Deferre	5	0.060413	0.010878	0.07992	0.032907
SuprA2D	Halogen	2	Dolloin Ton	After	5	0.069307	0.018348	0.092288	0.040723
SuprA2D	Halogen	2	тор Тор	Deferre	5	0.00742	0.013204	0.083813	0.051025
SuprA2D	Halogen	2	Top	Belore	5	0.074575	0.009396	0.08624	0.062906
SuprA2D	LED	0.5	Bottom	Atter	5	0.10594	0.042511	0.158724	0.053156
SuprA2D	LED	0.5	Bottom	Before	5	0.070867	0.018413	0.093729	0.048004
SuprA2D	LED	0.5	Тор	After	5	0.097767	0.031915	0.137394	0.058139
SuprA2D	LED	0.5	Тор	Before	5	0.0604	0.006653	0.068661	0.052139
SuprA2D	LED	1	Bottom	After	5	0.06284	0.006657	0.071106	0.054574
SuprA2D	LED	1	Bottom	Before	5	0.074533	0.022037	0.101896	0.047171
SuprA2D	LED	1	Тор	After	5	0.072633	0.020399	0.097961	0.047305
SuprA2D	LED	1	Тор	Before	5	0.081887	0.024163	0.111889	0.051884
SuprA2D	LED	2	Bottom	After	5	0.063633	0.012648	0.079337	0.047929
SuprA2D	LED	2	Bottom	Before	5	0.062447	0.015699	0.081939	0.042954
SuprA2D	LED	2	Тор	After	5	0.06464	0.007334	0.073746	0.055534
SuprA2D	LED	2	Тор	Before	5	0.076147	0.016871	0.097095	0.055199
SuprA2E	Halogen	0.5	Bottom	After	5	0.061053	0.013753	0.07813	0.043976
SuprA2E	Halogen	0.5	Bottom	Before	5	0.07018	0.012109	0.085215	0.055145
SuprA2E	Halogen	0.5	Тор	After	5	0.06372	0.006206	0.071425	0.056015
SuprA2E	Halogen	0.5	Тор	Before	5	0.07898	0.012164	0.094083	0.063877
SuprA2E	Halogen	1	Bottom	After	5	0.060727	0.006178	0.068398	0.053055
SuprA2E	Halogen	1	Bottom	Before	5	0.064847	0.00842	0.075301	0.054392
SuprA2E	Halogen	1	Тор	After	5	0.06734	0.007534	0.076695	0.057985
SuprA2E	Halogen	1	Тор	Before	5	0.0675	0.005859	0.074774	0.060226
SuprA2E	Halogen	2	Bottom	After	5	0.06126	0.005625	0.068244	0.054276
SuprA2E	Halogen	2	Bottom	Before	5	0.054913	0.006971	0.063569	0.046257
SuprA2E	Halogen	2	Тор	After	5	0.0709	0.012309	0.086184	0.055616
SuprA2E	Halogen	2	Тор	Before	5	0.072427	0.010343	0.085269	0.059584
SuprA2E	LED	0.5	Bottom	After	5	0.058247	0.00388	0.063064	0.053429
SuprA2E	LED	0.5	Bottom	Before	5	0.072273	0.019558	0.096558	0.047989
SuprA2E	LED	0.5	Тор	After	5	0.060793	0.005211	0.067264	0.054323
SuprA2E	LED	0.5	Тор	Before	5	0.08032	0.015907	0.100071	0.060569
SuprA2E	LED	1	Bottom	After	5	0.06846	0.013317	0.084996	0.051924
SuprA2E	LED	1	Bottom	Before	5	0.077253	0.013189	0.09363	0.060877
SuprA2E	LED	1	Top	After	5	0.067733	0.007939	0.077591	0.057876
SuprA2E	LED	1	Top	Before	5	0.079127	0.020211	0.104222	0.054031
SuprA2E	LED	2	Bottom	After	5	0.06448	0.023974	0.094247	0.034713
SuprA2E	LED	2	Bottom	Before	5	0.07158	0.024832	0 102414	0.040746
SuprA2E	LED	2	Ton	After	5	0 074447	0.018651	0.097605	0.051288
SuprA2E	LED	2	Top	Refore	5	0.09446	0.031498	0.133569	0.051200
Venus	Halogen	05	Bottom	After	5	0.05440	0.012831	0.082605	0.050741
Venus	Halogen	0.5	Bottom	Before	5	0.067933	0.015421	0.087081	0.048786
Vonus	Halogon	0.5	Ton	After	5	0.007955	0.008260	0.076068	0.048780
Venus	Halogen	0.5	Top	Before	5	0.0058	0.011338	0.078084	0.050820
Vonus	Halogen	0.5	Pottom	After	5	0.004907	0.026503	0.078984	0.050829
Venus	Halogen	1	Bottom	Refora	5	0.00324	0.020303	0.110140	0.052552
Vonus	Halogen	1	DOLIOIII Ton	After	ג ב	0.000000	0.004803	0.072309	0.000042
Vonus	Halogen	1	Top	Aner Refere	ג ב	0.073227	0.010008	0.000390	0.002030
Venus	Halagen	1	TOP Dottom	After	5	0.004/	0.00/155	0.0/3384	0.059762
v enus	Halogen	2	Bottom	After	5	0.070147	0.013126	0.091358	0.058/62
venus	Halogen	2	Bottom	Before	5	0.072147	0.011/42	0.086/27	0.022716
venus	Halogen	2	тор	After	5	0.077253	0.035063	0.12079	0.033/16
venus	Halogen	2	тор	Belore	5	0.077687	0.012226	0.092867	0.062507

									Anexo
Venus	LED	0.5	Bottom	After	5	0.069653	0.02086	0.095555	0.043752
Venus	LED	0.5	Bottom	Before	5	0.059647	0.007701	0.069209	0.050085
Venus	LED	0.5	Тор	After	5	0.078347	0.006213	0.086061	0.070632
Venus	LED	0.5	Тор	Before	5	0.060707	0.003629	0.065212	0.056201
Venus	LED	1	Bottom	After	5	0.066293	0.019685	0.090736	0.041851
Venus	LED	1	Bottom	Before	5	0.067527	0.013878	0.084759	0.050295
Venus	LED	1	Тор	After	5	0.072587	0.015584	0.091937	0.053237
Venus	LED	1	Тор	Before	5	0.068053	0.013268	0.084527	0.05158
Venus	LED	2	Bottom	After	5	0.08044	0.018842	0.103835	0.057045
Venus	LED	2	Bottom	Before	5	0.068473	0.01117	0.082342	0.054604
Venus	LED	2	Тор	After	5	0.058833	0.005299	0.065413	0.052254
Venus	LED	2	Тор	Before	5	0.068147	0.011207	0.082062	0.054231
Z250	Halogen	0.5	Bottom	After	5	0.07322	0.03307	0.114282	0.032158
Z250	Halogen	0.5	Bottom	Before	5	0.06458	0.008187	0.074745	0.054415
Z250	Halogen	0.5	Тор	After	5	0.086213	0.062996	0.164433	0.007994
Z250	Halogen	0.5	Тор	Before	5	0.069087	0.015281	0.088061	0.050112
Z250	Halogen	1	Bottom	After	5	0.054853	0.004131	0.059983	0.049724
Z250	Halogen	1	Bottom	Before	5	0.061233	0.00481	0.067206	0.055261
Z250	Halogen	1	Тор	After	5	0.061953	0.005799	0.069153	0.054753
Z250	Halogen	1	Тор	Before	5	0.062773	0.003551	0.067183	0.058364
Z250	Halogen	2	Bottom	After	5	0.070387	0.010882	0.083898	0.056875
Z250	Halogen	2	Bottom	Before	5	0.070527	0.016786	0.091369	0.049685
Z250	Halogen	2	Тор	After	5	0.07232	0.013751	0.089394	0.055246
Z250	Halogen	2	Тор	Before	5	0.05936	0.009549	0.071217	0.047503
Z250	LED	0.5	Bottom	After	5	0.07514	0.022606	0.103209	0.047071
Z250	LED	0.5	Bottom	Before	5	0.0626	0.012227	0.077782	0.047418
Z250	LED	0.5	Тор	After	5	0.071167	0.023766	0.100677	0.041657
Z250	LED	0.5	Тор	Before	5	0.064127	0.012682	0.079873	0.04838
Z250	LED	1	Bottom	After	5	0.058927	0.003437	0.063194	0.054659
Z250	LED	1	Bottom	Before	5	0.064	0.012353	0.079338	0.048662
Z250	LED	1	Тор	After	5	0.063453	0.007115	0.072288	0.054619
Z250	LED	1	Тор	Before	5	0.067687	0.014667	0.085898	0.049476
Z250	LED	2	Bottom	After	5	0.062313	0.008572	0.072957	0.05167
Z250	LED	2	Bottom	Before	5	0.059793	0.006439	0.067788	0.051799
Z250	LED	2	Тор	After	5	0.065147	0.013637	0.08208	0.048214
Z250	LED	2	Тор	Before	5	0.06656	0.012858	0.082526	0.050594