



Surface characterization of polymers used in fabrication of interim prostheses after treatment with photopolymerized glaze



Daniela Micheline dos Santos^{a,*}, Betina Chiarelo Commar^a, Liliâne da Rocha Bonatto^a, Emily Vivianne Freitas da Silva^a, Mariana Vilela Sônego^a, Elidiane Cipriano Rangel^b, Aldieris Alves Pesqueira^a, Marcelo Coelho Goiato^a

^a Department of Dental Materials and Prosthodontics, Aracatuba Dental School, Univ Estadual Paulista (UNESP), José Bonifácio St., 1193, Aracatuba, São Paulo 16015-050, Brazil

^b Technological Plasma Laboratory (LaPTec), Experimental Campus of Sorocaba, Univ Estadual Paulista (UNESP), Tres de Março Av., 511, Sorocaba, Sao Paulo, 18087-180, Brazil

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ABSTRACT

The material used for interim prostheses fabrication must present excellent physical properties for greater longevity in the face of environmental conditions, which can occur in the oral cavity. The purpose of this study was to evaluate the effect of a photopolymerized glaze on the physical and mechanical properties of polymers used for the fabrication of interim prostheses, before and after thermocycling and immersion in staining solutions. One hundred samples of composite and acrylic resins were fabricated: Dencor chemically activated acrylic resin (CAAR) ($n = 20$) and heat-polymerized acrylic resin (HPAR) ($n = 20$), Charisma ($n = 20$), Structur ($n = 20$), and Protemp ($n = 20$). A mechanical polishing was performed on half of the samples, and a chemical polishing was performed on the remaining samples. Subsequently, all samples were submitted to thermocycling and immersion in coffee staining solution for 21 days. Analysis of color and microhardness, as well as atomic force microscopy (AFM), scanning electron microscopy (SEM), and energy dispersive x-ray spectrometry (EDS) were performed. The data were submitted to repeated-measures analysis of variance (ANOVA), followed by the Tukey test ($\alpha = 0.05$) and the Student t -test ($\alpha = 0.05$). It was verified that the glaze decreased the chromatic alteration values, and increased the microhardness values of the samples, with the exception of the Charisma resin. The samples that did not receive chemical polishing had the greatest number of surface irregularities. This study concluded that the groups with glaze presented less color alteration. In addition, Charisma and Structur resins exhibited the greatest chromatic stability. As to the microhardness, the values were greater when the samples were treated with the glaze, with the exception of the Charisma group.

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1. Introduction

Adequate interim restorations must furnish the esthetic, functional, and biologic needs of the patient [1–4]. In some cases such as periodontal surgeries, rehabilitations involving implant installations, or maxillo-facial rehabilitations [3], the use of interim restorations can be indicated for a greater period [3,5]. However, the physical and mechanical properties of the materials could suffer alterations over time, due to the action of the adverse environment of the oral cavity where the sorption of water and other liquids occur, decreasing the longevity of the restoration [6]. Therefore, the material used must present excellent physical and mechanical properties to achieve greater longevity [7,8].

Chromatic stability is the property a material possesses to retain its color for a period of time in a determined environment, which is an important physical property for dental materials [9,10]. The alteration of color in polymeric materials can be caused by intrinsic and extrinsic factors [10–14]. Modifications in the polymeric matrix with chromatic alteration of the restorative material are caused by intrinsic factors [10, 13]. Extrinsic factors such as solar radiation, thermal changes, humidity, absorption, and adsorption of substances can also cause discoloration [10–14]. Also, the surface and luminosity of the object must be considered as influencing factors in the determination of the color [15].

Another characteristic which can be influenced by the oral cavity is the surface microhardness, characterized by the resistance of the material to a permanent penetration. (artigo bruna) This characteristic is related to other properties of the material, such as the wear resistance [16–18]. Restorations suffer physical and mechanical alterations over time, causing surface degradation which allows the formation of microfractures that interfere in the maintenance of treatment [17–19].

* Corresponding author at: Department of Dental Materials and Prosthodontics, Aracatuba Dental School, Univ. Estadual Paulista, UNESP, Jose Bonifacio St., 1153, Vila Mendonca, Aracatuba, Sao Paulo, Brazil, 16015-050.

E-mail address: danielamicheline@foa.unesp.br (D.M. Santos).

Many materials and techniques exist for mechanical polishing of polymers to increase the surface smoothness, and therefore decrease porosity and bacterial adhesion. These include the use of abrasive bits with different granulations, and/or chemical polishing with the use of chemical substances on the material [20,21]. Currently, liquid polishing with photopolymerized sealants is being used to reduce the stages of polishing, providing a smooth and polished surface, and avoiding the accumulation of biofilm [22].

Therefore, the purpose of this study was to evaluate the influence of the application of photopolymerized glaze on the physical and mechanical properties of polymers used in the fabrication of interim prostheses, before and after thermocycling and immersion in coffee staining solution. The null hypothesis is that the application of photopolymerized glaze, and the processes of thermocycling and immersion of the samples, does not influence the physical and mechanical properties of the polymers used in the fabrication of interim prostheses.

2. Materials and methods

2.1. Sample preparation

Five different types of resins ($n = 20$) were analyzed: resin composed of Bis-acryl from Prottemp (3 M/ESPE, Seefeld, Germany) and Structur (VOCO, Vuxhaven, Germany); resin composed of Bis-GMA from Charisma (Heraeus Kulze South America Ltda, Sao Paulo, Sao Paulo, Brazil); and autopolymerized (CAAR) and heat-activated (HPAR) acrylic resins, from Dencor (Artigos Odontologicos Classico Ltda, Sao Paulo, Sao Paulo, Brazil).

A 3 mm thick molded metal matrix was used for standardization of the samples. The matrix contained 10 circular compartments in its interior, each with a 10 mm diameter [23].

The specimens were divided into 10 groups ($n = 10$): Prottemp without photopolymerized glaze (A), Prottemp with photopolymerized glaze (B), Charisma without photopolymerized glaze (C), Charisma with photopolymerized glaze (D), CAAR without photopolymerized glaze (E), CAAR with photopolymerized glaze (F), HPAR without photopolymerized glaze (G), HPAR with photopolymerized glaze (H), Structur without photopolymerized glaze (I), Structur with photopolymerized glaze (J).

During the fabrication of the samples, the inferior portion of the matrix was supported over a glass slide, a thin layer of solid petroleum jelly was applied, and the entire cavity was filled with the CAAR or composite resins (Charisma, Structur, or Prottemp). Another glass slide was positioned over the resin layer to drain the excess material, maintaining the surface smooth and homogeneous. Subsequently, the samples were polymerized according to the manufacturer's recommendations [24,25].

For the fabrication of the HPAR samples, the metal matrix and glass slide set was placed in a special muffle for microwave ovens (Artigos Odontologicos Classico Ltda, Sao Paulo, Sao Paulo, Brazil) with special plaster type IV (Durone; Dentsply Ind and Com Ltda, Petropolis, Rio de Janeiro, Brazil) and a proportion of 100 g of powder to 30 mL of water, spatulated for 1 min, and then poured under constant vibration. After the crystallization of the plaster, a second glass slide was positioned over the matrix already included in the plaster. A counter-muffle was positioned and special plaster type IV was poured over this last glass slide. Subsequently, the muffle was taken to the hydraulic bench press (VH Midas Dental Produtos Ltda, Araraquara, Sao Paulo, Brazil) and was put under a constant pressure of 1.25 tons for 3 min. After the crystallization of the plaster, the muffle was opened and the glass surface was cleaned with acetone (Labsynth Produtos Laboratorios Ltda, Diadema, Sao Paulo, Brazil). The HPAR was proportioned according to the manufacturer's instructions. The resin was polymerized by microwave energy (Brastemp, Sao Paulo, Sao Paulo, Brazil) for 3 min with 30% power, then 4 min with 0% power, and finally, 3 min with 60% power, according to the manufacturer's instructions [26,27]. After the

polymerization, the samples were submitted to finishing with Maxicut bits (Vicking, Sao Paulo, Sao Paulo, Brazil) for excess removal [26].

Subsequently, a polishing of all samples was performed in an automatic polisher (Ecomet 300PRO; Buehler, Illinois, USA) with 400- and 800-grain metallographic sandpaper (Buehler, Illinois, USA), under constant water irrigation at a velocity of 300 rpm [28]. Groups that did not receive the additional layer of photopolymerized glaze underwent additional polishing with 1000- and 1200-grain sandpaper, and the completion of the polishing with a diamond solution on a felt disc (Buehler, Illinois, USA). Each disc had its thickness measured with the assistance of a digital caliper (500–171-20B; Mitutoyo, Tokyo, Japan), to obtain the established dimensions [29].

The samples that received chemical polishing were submitted to a recoating of the photopolymerized glaze (Megadenta; Radeberg, Germany), with a fine, uniform layer being applied on the surface using a soft brush in one direction to avoid air bubbles. After a period of 20 s, photopolymerization was performed for 180 s (Strobolux; EDG Equipamentos, Sao Carlos, Sao Paulo, Brazil), according to the manufacturer's recommendations. Prior to the tests, the samples were stored in an artificial saliva solution in a digital bacterial incubator (CIENLAB Equipamentos Cientificos Ltda, Campinas, Sao Paulo, Brazil) at 37 ± 1 °C for 24 h [30].

Thereafter, tests for color alteration and microhardness were performed. For the surface characterization, atomic force microscopy (AFM), scanning electron microscopy (SEM), and energy dispersive x-ray spectrometry (EDS) were performed. The same tests were performed again after 2000 thermocycles (t1) and after 21 days of sample immersion in coffee staining solution (t2).

2.2. Color analysis

The color alterations were calculated by means of the CIE L*a*b* system, in accordance with the Comissim Internazionale de l'Eclairage – CIE (International Commission of Illumination) norms [31].

2.3. Microhardness analysis

The surface microhardness was analyzed by means of the HMV-2 T microdurometer (Shimadzu Corp, Kyoto, Japan), following ASTM (American Society for Testing Materials) specifications [32].

2.4. Atomic force microscopy

For the AFM analysis, an additional sample from each group was fabricated for each test period. The images produced were transferred to the Gwyddion 2.33 software program (Nanometrology Department, Czech Institute of Nanometrology, Prague, Czech Republic) to obtain 3-D images (5×5 μm).

2.5. SEM-EDS

The SEM analysis (JSM 610LA; JEOL, Tokyo, Japan) was performed on an additional sample of each group for each test period, with the registered images magnified $300\times$. The EDS analysis was performed simultaneously with the SEM on the order of $1\mu\text{m}^3$ [33].

2.6. Thermocycling

All samples were thermocycled (Convel, Sao Paulo, Sao Paulo, Brazil) by being immersed in distilled water and performing alternated baths of 30 s at a temperature of 5 ± 1 °C and 55 ± 1 °C, totaling 2000 cycles [34].

2.7. Immersion in coffee staining solution

During the immersion process in coffee staining solution (Cafe Pilao; Sara Lee, Jundiá, Sao Paulo, Brazil), each sample was placed in a

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