

RESEARCH AND EDUCATION

Optical parameters and hardness of two maxillofacial elastomers after immersion in different solutions of Brazilian green propolis extract



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Rehabilitation with maxillofacial prostheses restores esthetics and function, protects remaining tissues, and improves the quality of life of wearers.¹⁻³ Maxillofacial prostheses are fabricated from room temperature or heat-polymerized elastomers. These materials are translucent, easy to manipulate and pigment,⁴⁻⁶ promote excellent esthetics,⁷ and have a lifetime of 6 months to 3 years⁸⁻¹⁰

The optical properties of maxillofacial elastomers determine the quantity and quality of transmitted, reflected, and absorbed light and are therefore essential for color perception.^{11,12} Transmittance is a measure of the fraction of incident light at a specified wavelength, and spectral reflectance is a fraction of incident light that is reflected at an interface

ABSTRACT

Statement of problem. Maxillofacial elastomers undergo physical and mechanical degradation with disinfecting solutions. Solutions of Brazilian green propolis extract may be suitable alternatives for infection control of maxillofacial prostheses. However, their effects on the properties of the material are unknown.

Purpose. The purpose of this in vitro study was to evaluate the effect of disinfection with solutions of Brazilian green propolis extract on the transmittance, translucency parameter, contrast ratio, and hardness of 2 maxillofacial elastomers (MDX4-4210 and MED-4014).

Material and methods. Fifty disk-shaped specimens (3×10 mm) of each elastomer were randomly and equally divided into 4 groups of disinfectant agents and 1 control group: 3 separate groups of 11% green propolis extracts including aqueous (PAQ), glycolic (PGL), and alcoholic (PAL), a 2% chlorhexidine gluconate (CHX) group, and the control group of distilled water. Specimens were subjected to disinfection by immersion 3 times a week for 60 days. Color differences (ΔE values) were calculated with CIE Lab and CIE DE2000 formulas. Optical parameters and Shore A hardness were determined at 2 time points: at baseline and after the period of specimen disinfection. Data were analyzed by parametric and nonparametric analysis of variance and by multiple-comparison tests ($\alpha=.05$).

Results. The ΔE values of specimens immersed in 11% PAL were not clinically acceptable for either elastomer. Regarding translucency parameter and contrast ratio, the immersion in 11% PAL and 11% PGL resulted in greater opacity and lower translucency of the material. Mean Shore A hardness values were not statistically significantly different at baseline or after 60 days of immersion in the solutions.

Conclusions. The solution of Brazilian green propolis extract tested showed changes in optical parameters. Elastomers immersed in 11% alcoholic green propolis extract showed clinically unacceptable color and translucency changes. All hardness values of the tested elastomers were clinically acceptable after immersion in all tested disinfectant groups. (*J Prosthet Dent* 2019;122:168-75)

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Clinical Implications

Based on the results of this initial study, the use of the Brazilian green propolis extract as a disinfectant for maxillofacial prostheses appears to be promising based on color and hardness measurements. Clinicians may consider the solution for disinfection by immersion for 15 minutes if 11% aqueous or glycolic green propolis extracts are used, depending on the elastomer of the maxillofacial prostheses.

such as porosity. Translucency parameter (TP) values are defined by the difference in color values obtained between the light reflected by a material positioned on black and white backgrounds.¹² Contrast ratio (CR) parameter values are related to the translucency of the material ranging from complete opacity to complete translucency and can be measured by spectral reflectance.¹²⁻¹⁴ Visual color perception by means of measuring devices permits the identification of these changes without the subjectivity of the human eye by using the CIELab and CIEDE2000 systems,^{4,14-19} expressed as numerical data, where L* (lightness) is value axis, a* is red-green axis, b* is yellow-blue axis, C* (chroma), and h (hue angle).

Preservation of the color, translucency, brightness, and resiliency of the prosthesis is important, but color changes may occur, for example, from prosthesis disinfection.^{9,10} Regular prosthesis disinfection is essential, and the prosthesis is constantly exposed to the environment.²⁰ Previous investigations^{9,21,22} have reported that the esthetic properties of a prosthesis are directly involved in its durability and in wearer satisfaction.

Chlorhexidine gluconate (CHX) solution has been used as a disinfectant,^{9,10} causing possible changes in the structure of the elastomer.²³ Phytotherapeutic agents have been investigated for the disinfection of prosthetic materials with reported antimicrobial efficacy, although with color changes.^{9,24} A solution of Brazilian green propolis extract may be an economical and available alternative disinfectant for maxillofacial prostheses.²⁵⁻²⁹ Propolis is a substance of varied color and consistency, consisting of resins and balsams (55%), wax (30%), volatile oils (10%), and pollen (5%), which constitute a complex and heterogeneous mixture.^{28,30,31} The main plant species pollinated by bees and native to Brazil is field rosemary, *Baccharis dracunculifolia*. However, studies on the effects of solutions of Brazilian green propolis extract on the physical and mechanical properties of maxillofacial elastomers are lacking.

The purpose of this in vitro study was to evaluate the transmittance color change (ΔE) values, L* and C* transmittance coordinates, spectral reflectance and color parameters, TP and CR values, and the hardness of

Table 1. Manufacturer and chemical composition of disinfectants and elastomers evaluated

| Source | Material | Manufacturer | Chemical Composition |
|---------------|---|----------------------|---|
| Elastomers | MDX4-4210, room temperature vulcanization (RTV) | Dow Corning Corp | Poly(dimethylsiloxane) |
| | MED-4014, high-temperature vulcanization (HTV) | NuSil Technology LLC | Not available |
| Disinfectants | 11% Propolis aqueous extract | Bee Propolis Brasil | Deionized water and Brazilian green propolis |
| | 11% Propolis glycolic extract | Bee Propolis Brasil | Propylene glycol and Brazilian green propolis |
| | 11% Propolis alcoholic extract | Bee Propolis Brasil | Neutral grain alcohol and Brazilian green propolis |
| | 2% Chlorhexidine solution | Maquira | 2% Chlorhexidine gluconate, methylparaben, purified water |

2 elastomers immersed in solutions of Brazilian green propolis extract as an alternative to periodic disinfection. The null hypothesis was that the 60-day period of immersion in the proposed solutions would not alter the optical parameters or hardness of the maxillofacial elastomers.

MATERIAL AND METHODS

Fifty disk-shaped specimens were fabricated from 2 maxillofacial silicone elastomers (Table 1) in a metal matrix (3 mm in thickness, 10 mm in diameter).¹⁹ The specimens were fabricated in a dental flask (VIPI STG; VIPI Ind Com Ltd) filled with Type IV stone (Durone IV; Dentsply Sirona). A smooth glass slide was embedded in the stone with its upper surface parallel to the margins of the flask. The metal matrix was positioned on the glass slide covered with a similar glass slide, and extra-hard laboratory silicone was used to hold the assembly (Zetalabor; Zhermack SpA). The stone of the flask was isolated by using a separating medium (Vaseline; Rioquímica), and a counter flask was positioned and filled with Type IV stone (Durone IV; Dentsply Sirona). The room temperature vulcanization (RTV) elastomer was manually mixed at a ratio of 10:1 (base:catalyst) by using a stainless-steel spatula (no. 36; SS White Duflex) and inserted into the internal surfaces of the matrix at room temperature (23 ± 2 °C) and at $50 \pm 10\%$ relative humidity (Fig. 1). The flask was left to stand at room temperature and humidity for 72 hours for complete polymerization of the material.^{9,10} The high-temperature vulcanization (HTV) elastomer was mixed mechanically, and a polyurethane sheet (AI-513; Factor II, Inc) was positioned on the glass slides. Silicone was then inserted into the inner surfaces of the matrix, and the flask was placed in an oven (CE-210/100; CIENLAB Equipamentos Científicos Ltda-EPP) at a temperature of approximately 116 ± 3 °C for 10 minutes. The flask was left to stand at room temperature and humidity for 3 hours for complete polymerization of the material.

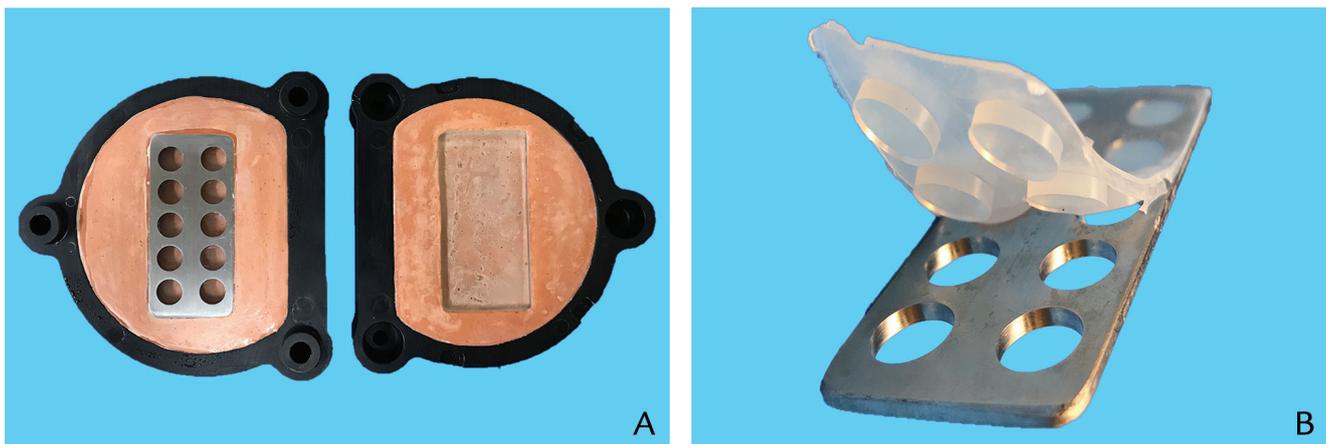


Figure 1. Specimen fabrication. A, Metal matrix embedded in silicone and dental stone for manipulation and insertion of maxillofacial elastomer into their circular internal surfaces. B, Detachment of specimens from metal matrix after complete polymerization.

The transmittance and reflectance of light (in percentage) of all specimens were measured by using a spectrophotometer (CM-3700D; Konica Minolta) with a perimeter of approximately 24 mm in the 360- to 740-nm wavelength range, with a D65 illuminant at a 2-degree angle.¹⁸ For each test, the specimens were measured at 2 time points, baseline and after 60 days of immersion in the solutions, with the aid of a device and by the same calibrated operator (A.M.).¹⁰

ΔE values for transmittance were calculated with CIELab (ΔE_{ab}^*) and CIEDE2000 (ΔE_{00}) formulas as established by the Commission Internationale de l'Éclairage³² and by previous studies.^{9,10,12,14,15,19,33} The CIELab (ΔE_{ab}^*) system calculated the color variation between 2 points in a 3D color space according to the following formula: $\Delta E_{ab}^* = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2}$, where ΔL^* , Δa^* , and Δb^* are the differences in the respective coordinates for a pair of readings. Therefore, L^* , a^* , b^* coordinates are shown through ΔE values, and C^* and h are polar coordinates and can be defined from a^* and b^* : $C^* = \sqrt{(a^*)^2 + (b^*)^2}$; $\tan h = \left(\frac{b^*}{a^*}\right)$. The CIEDE2000 (ΔE_{00}) color differences were calculated according to the following formula: $\Delta E_{00} = \sqrt{\left(\frac{\Delta L}{k_L S_L}\right)^2 + \left(\frac{\Delta C}{k_C S_C}\right)^2 + \left(\frac{\Delta H}{k_H S_H}\right)^2 + R_T \left(\frac{\Delta C}{k_C S_C}\right)^2 \left(\frac{\Delta H}{k_H S_H}\right)^2}$, where R_T is a rotation function; S_L , S_C , and S_H are weighting functions that adjust the total color difference for variation in a perceived magnitude with variation in the location of color difference pair in L , a , b coordinates; and the parametric terms k_L , k_C , and k_H , are correction terms for experimental conditions (under reference conditions, they are all set at 1).

Color coordinates for reflectance of light were recorded from specimens placed on black and white backgrounds to simulate the illumination of the facial

substrates. A coupling agent,¹⁴ glycerin solution (Glicerina bi-destilada; Farmax), was applied between the specimens and backgrounds only for reflectance measuring. TP and CR were calculated from the reflectance data after 60 days of immersion in the solutions based on the following formula according to previous studies: $TP = \sqrt{(L_B^* - L_W^*)^2 + (a_B^* - a_W^*)^2 + (b_B^* - b_W^*)^2}$ and $CR = \frac{Y_B}{Y_W}$, where $Y = \left(\frac{L+16}{116}\right)^3 \times Y_n$.^{12,34,35} The spectral reflectance Y (luminance from tristimulus color space/XYZ) was calculated from L^* values, and the specified white stimulus normally chosen was one with the appearance of a perfect reflecting diffuser, normalized by a common factor so that Y_n is equal to 100. ΔE values lower than 3.0/4.4 (light/dark specimens) for CIELab and values lower than 2.1/3.1 for CIEDE2000 were considered within the threshold of acceptability.¹⁹

The specimens were submitted to baseline hardness evaluation. A digital Shore A durometer (HH-336; Mitutoyo) was used to test the hardness of the specimens according to American Society for Testing and Materials specifications D2240.³⁶ Three readings were obtained for each specimen by the same calibrated operator (N.B.M.), and the average value was expressed as Shore units (range: 0 to 100).^{6,9} Hardness was considered to be clinically acceptable when specimens had a Shore A of 12 to 35.

Specimens of each elastomer were equally divided into 4 groups of disinfectant agents and 1 control group—3 groups of 11% green propolis extracts, namely aqueous (PAQ), glycolic (PGL), and alcoholic (PAL); a 2% CHX group; and distilled water as a control group (Table 1)—according to the randomization sequence created using computer-generated random numbers (Excel 2007; Microsoft Corp).

The specimens were disinfected 3 times a week for 60 days by immersion for 15 minutes in each solution. Immediately after treatment, all specimens were rinsed in

Table 2. ΔE, TP, and CR values of all specimens after 60 days of immersion in solutions

| Elastomers | RTV | | | | HTV | | | |
|------------------|--------------------------|--------------------------|--------------------------|-------------------------|-------------------------|-------------------------|--------------------------|--------------------------|
| | ΔE* _{ab} | ΔE ₀₀ | TP | CR | ΔE* _{ab} | ΔE ₀₀ | TP | CR |
| 11% PAQ | 2.90 ±0.49 ^c | 2.25 ±0.29 ^c | 50.62 ±1.87 ^b | 0.11 ±0.01 ^c | 1.59 ±0.59 ^c | 1.05 ±0.41 ^c | 46.01 ±2.1 ^a | 0.18 ±0.02 ^{ab} |
| 11% PGL | 12.80 ±0.82 ^b | 8.29 ±0.43 ^b | 48.57 ±0.75 ^c | 0.14 ±0.01 ^b | 3.34 ±0.81 ^b | 2.40 ±0.50 ^b | 45.95 ±3.01 ^a | 0.19 ±0.03 ^a |
| 11% PAL | 19.47 ±0.89 ^a | 11.56 ±0.45 ^a | 48.48 ±0.84 ^c | 0.16 ±0.01 ^a | 6.80 ±1.53 ^a | 4.44 ±0.77 ^a | 47.10 ±2.86 ^a | 0.19 ±0.03 ^a |
| H ₂ O | 1.40 ±0.65 ^d | 0.98 ±0.37 ^d | 52.83 ±1.01 ^a | 0.10 ±0.01 ^c | 1.47 ±0.84 ^c | 1.01 ±0.51 ^c | 47.45 ±1.46 ^a | 0.17 ±0.01 ^{bc} |
| 2% CHX | 1.83 ±0.67 ^d | 1.24 ±0.42 ^d | 52.23 ±1.11 ^a | 0.11 ±0.01 ^c | 1.76 ±0.63 ^c | 1.10 ±0.37 ^c | 47.88 ±1.46 ^a | 0.15 ±0.01 ^c |

ΔE, color change; CHX, chlorhexidine gluconate; CR, contrast ratio; HTV, high-temperature vulcanization; PAL, alcoholic propolis extract; PAQ, aqueous propolis extract; PGL, glycolic propolis extract; RTV, room temperature vulcanization; TP, translucency parameter. Values are given as mean ±standard deviation. Different superscript letters indicate significant differences among solution groups for each optical parameter for both elastomers (*P*<.001, multiple testing procedure).

Table 3. Global test of ΔE values and TP and CR values of all elastomer specimens after 60 days of immersion in solutions

| Elastomers | ΔE* _{ab} | | | | ΔE ₀₀ | | | | TP | | | | CR | | | |
|---|-------------------|------|-----|----------|------------------|------|-----|----------|------------------|------|-----|----------|------------------|------|-----|----------|
| | Statistical Test | df1 | df2 | <i>P</i> | Statistical Test | df1 | df2 | <i>P</i> | Statistical Test | df1 | df2 | <i>P</i> | Statistical Test | df1 | df2 | <i>P</i> |
| RTV | | | | | | | | | | | | | | | | |
| ANOVA type test <i>P</i> value | 95.58 | 4.00 | 45 | <.001 | 123.49 | 4.00 | 45 | <.001 | 27.55 | 4.00 | 45 | <.001 | 51.42 | 4.00 | 45 | <.001 |
| McKeon approximation for Lawley-Hotelling test | 95.58 | 4.00 | 45 | <.001 | 123.49 | 4.00 | 45 | <.001 | 27.55 | 4.00 | 45 | <.001 | 51.42 | 4.00 | 45 | <.001 |
| Muller approximation for Bartlett-Nanda-Pillai test | 93.66 | 4.08 | 45 | <.001 | 121.02 | 4.08 | 45 | <.001 | 27.00 | 4.08 | 45 | <.001 | 50.39 | 4.08 | 45 | <.001 |
| Wilks Lambda | 95.58 | 4.00 | 45 | <.001 | 123.49 | 4.00 | 45 | <.001 | 27.55 | 4.00 | 45 | <.001 | 51.42 | 4.00 | 45 | <.001 |
| HTV | | | | | | | | | | | | | | | | |
| ANOVA type test <i>P</i> value | 33.12 | 4.00 | 45 | <.001 | 33.93 | 4.00 | 45 | <.001 | 2.57 | 4.00 | 45 | .050 | 8.78 | 4.00 | 45 | <.001 |
| McKeon approximation for Lawley-Hotelling test | 33.12 | 4.00 | 45 | <.001 | 33.93 | 4.00 | 45 | <.001 | 2.57 | 4.00 | 45 | .050 | 8.78 | 4.00 | 45 | <.001 |
| Muller approximation for Bartlett-Nanda-Pillai test | 33.49 | 4.08 | 45 | <.001 | 33.25 | 4.08 | 45 | <.001 | 2.52 | 4.08 | 45 | .053 | 8.60 | 4.08 | 45 | <.001 |
| Wilks Lambda | 33.12 | 4.00 | 45 | <.001 | 33.93 | 4.00 | 45 | <.001 | 2.57 | 4.00 | 45 | .050 | 8.78 | 4.00 | 45 | <.001 |

ΔE, color change; CR, contrast ratio; HTV, high-temperature vulcanization; RTV, room temperature vulcanization; TP, translucency parameter. *P*<.05 denotes statistically significant difference.

running water for 30 seconds and stored in the dark under constant temperature (23 ±2 °C) and relative humidity (50 ±10%). After the disinfection period of 60 days, new readings of optical parameters and hardness were obtained.

All color data were subjected to the Levene tests of homogeneity of variance ($\alpha=.05$). The ΔE, TP, and CR values after immersion in the solutions and the color coordinates; L* and C* of mean transmittance values of the elastomers after immersion in the solutions; and the spectral reflectance curves (mean value for each wavelength) and their corresponding standard errors were submitted to nonparametric multivariate analysis of variance. Nonparametric analysis of variance was based on the permutation test, a multivariate analog of the Fisher F-ratio calculated directly from any symmetric distance or dissimilarity matrix.³⁷ The analysis was performed for each elastomer, considering the spectral ranges of 39 bands (variables).

The statistical analyses were performed by using a statistical software program (R v3.5.1; Free Software Foundation’s GNU project and the R Foundation).³⁸ The *npmv*³⁹ package provides the R functions “nonpartest” and “ssnonpartest.” The first is used for the global test (Wilks Lambda) and the second for a multiple-comparison procedure. Four types of nonparametric statistical

tests were considered: ANOVA, Wilks Lambda test, Lawley-Hotelling test, and Bartlett-Nanda-Pillai test present in the *npmv* package.^{40,41} For spectral reflectance, the statistical significance of nonparametric multivariate analysis of variance was computed by permutation of the group memberships and conducted with the “adonis” function from the *vegan*⁴² package because of restrictions of the *npmv* package when the number of variables is too large.³⁷

Hardness data were submitted to 2-way repeated-measure analysis of variance, appropriate as the measurement of the dependent variable was made at 2 time points and assuming that the condition of sphericity had been met.

RESULTS

All specimens showed significant ΔE values and TP and CR values after 60 days of immersion in the solutions (Table 2). Table 3 shows the global test and the multiple-comparison procedure for ΔE values, TP, and CR. The differences in mean values were statistically significant among the solutions for both elastomers, except the mean TP values for the HTV elastomer. In general, the ΔE values of 11% PAQ and 2% CHX for both elastomers and those of 11% PGL for the HTV elastomer may be

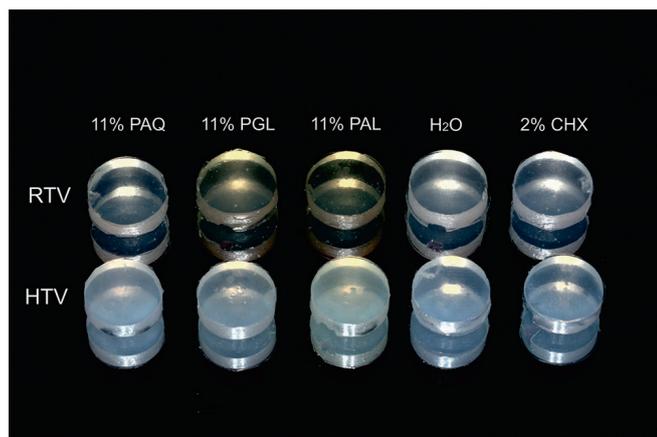


Figure 2. Specimens after 60 days of immersion in solutions. CHX, chlorhexidine gluconate; HTV, high-temperature vulcanization; PAL, alcoholic propolis extract; PAQ, aqueous propolis extract; PGL, glycolic propolis extract; RTV, room temperature vulcanization.

Table 4. Transmittance values of polar coordinates (C* and L*) of all elastomer specimens after 60 days of immersion in solutions

| Elastomers | RTV | | HTV | |
|------------------|--------------------------|--------------------------|----------------------------|--------------------------|
| | L* | C* | L* | C* |
| 11% PAQ | 91.04 ±0.40 ^b | 10.32 ±0.27 ^c | 83.60 ±1.36 ^a | 14.78 ±0.67 ^c |
| 11% PGL | 88.22 ±1.09 ^c | 19.01 ±0.99 ^b | 80.92 ±1.60 ^b | 17.96 ±2.54 ^b |
| 11% PAL | 85.85 ±0.66 ^d | 25.67 ±0.64 ^a | 81.07 ±2.93 ^{a,b} | 20.44 ±0.93 ^a |
| H ₂ O | 92.55 ±0.56 ^a | 7.20 ±0.30 ^d | 84.20 ±0.59 ^a | 14.60 ±0.85 ^c |
| 2% CHX | 92.01 ±0.69 ^a | 7.36 ±0.40 ^d | 84.18 ±1.13 ^a | 13.48 ±0.20 ^d |

CHX, chlorhexidine gluconate; HTV, high-temperature vulcanization; PAL, alcoholic propolis extract; PAQ, aqueous propolis extract; PGL, glycolic propolis extract; RTV, room temperature vulcanization. Values are given as means ±standard deviation. Different superscript letters indicate significant differences among solution groups for each optical parameter for both elastomers ($P < .001$, multiple testing procedure).

clinically imperceptible or clinically acceptable (Fig. 2). TP values were significantly lower for the RTV elastomer immersed in 11% PAQ (50.62). However, TP values for RTV were significantly higher ($P < .001$) than that for H₂O (52.83) and 2% CHX (52.23). Regarding opacity, 11% PAL (0.14 for RTV and 0.16 for HTV) and 11% PGL (0.19 for RTV/HTV) affected the CR values compared with H₂O and 2% CHX (Tables 2 and 3).

The mean transmittance values for polar coordinates (C* and L*) after 60 days of immersion in the solutions are shown in Table 4. The transmittance L* and C* coordinate values of the elastomers were affected by solutions and the type of elastomer ($P < .001$). For the RTV elastomer, the propolis extract solutions had the lowest L* coordinate values and the highest C* coordinate values, with statistically significant differences, compared with H₂O and 2% CHX. The transmittance L* (83.60) and C* (14.78) coordinate values of 11% PAQ were similar to those of H₂O (84.20 for L* and 14.60 for C*) for the HTV elastomer compared with other solutions (Fig. 3; Tables 4 and 5).

The spectral reflectance curves (mean values for each wavelength) and their standard error for all specimens

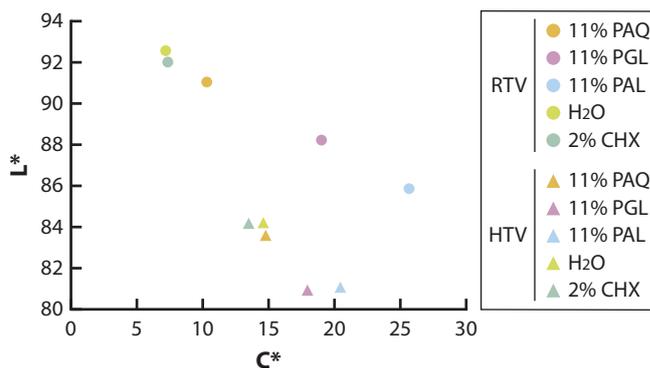


Figure 3. Color distribution of mean transmittance values in polar coordinates (C* and L*) of elastomer specimens after 60 days of immersion in solutions. CHX, chlorhexidine gluconate; HTV, high-temperature vulcanization; PAL, alcoholic propolis extract; PAQ, aqueous propolis extract; PGL, glycolic propolis extract; RTV, room temperature vulcanization.

Table 5. Global test of transmittance values of polar coordinates (C* and L*) of all elastomer specimens after 60 days of immersion in solutions

| Elastomers | Statistical Test | L* | | | C* | | | |
|---|------------------|------|-----|-------|------------------|------|-----|-------|
| | | df1 | df2 | P | Statistical Test | df1 | df2 | P |
| RTV | | | | | | | | |
| ANOVA type test | 91.39 | 4.00 | 45 | <.001 | 119.33 | 4.00 | 45 | <.001 |
| P value | | | | | | | | |
| McKeon approx. for Lawley-Hotelling test | 91.39 | 4.00 | 45 | <.001 | 119.33 | 4.00 | 45 | <.001 |
| Muller approx. for Bartlett-Nanda-Pillai test | 89.56 | 4.08 | 45 | <.001 | 116.94 | 4.08 | 45 | <.001 |
| Wilks Lambda | 91.39 | 4.00 | 45 | <.001 | 119.33 | 4.00 | 45 | <.001 |
| HTV | | | | | | | | |
| ANOVA type test | 7.42 | 4.00 | 45 | <.001 | 33.49 | 4.00 | 45 | <.001 |
| P value | | | | | | | | |
| McKeon approx. for Lawley-Hotelling test | 7.42 | 4.00 | 45 | <.001 | 33.49 | 4.00 | 45 | <.001 |
| Muller approx. for Bartlett-Nanda-Pillai test | 7.27 | 4.08 | 45 | <.001 | 32.82 | 4.08 | 45 | <.001 |
| Wilks Lambda | 7.42 | 4.00 | 45 | <.001 | 33.49 | 4.00 | 45 | <.001 |

HTV, high-temperature vulcanization; RTV, room temperature vulcanization. $P < .05$ denotes statistically significant difference.

after 60 days of immersion in the solutions are shown in Figure 4, with significant differences being detected ($P < .001$) (Table 6). For the HTV elastomer, H₂O, 2% CHX, and 11% PAQ showed similar spectral reflectance curves. Tables 7 and 8 show the mean values and the results of ANOVA for Shore A hardness for the maxillofacial elastomers during the testing period. Solution and period variables did not affect the hardness of the elastomers. In general, the mean Shore A hardness was 30.18 to 30.39 at baseline and 30.15 to 30.78 after 60 days of immersion in the solutions for RTV elastomers and 15.28 to 15.55 at baseline and 15.35 to 15.74 after 60 days of immersion in the solutions for HTV elastomers.

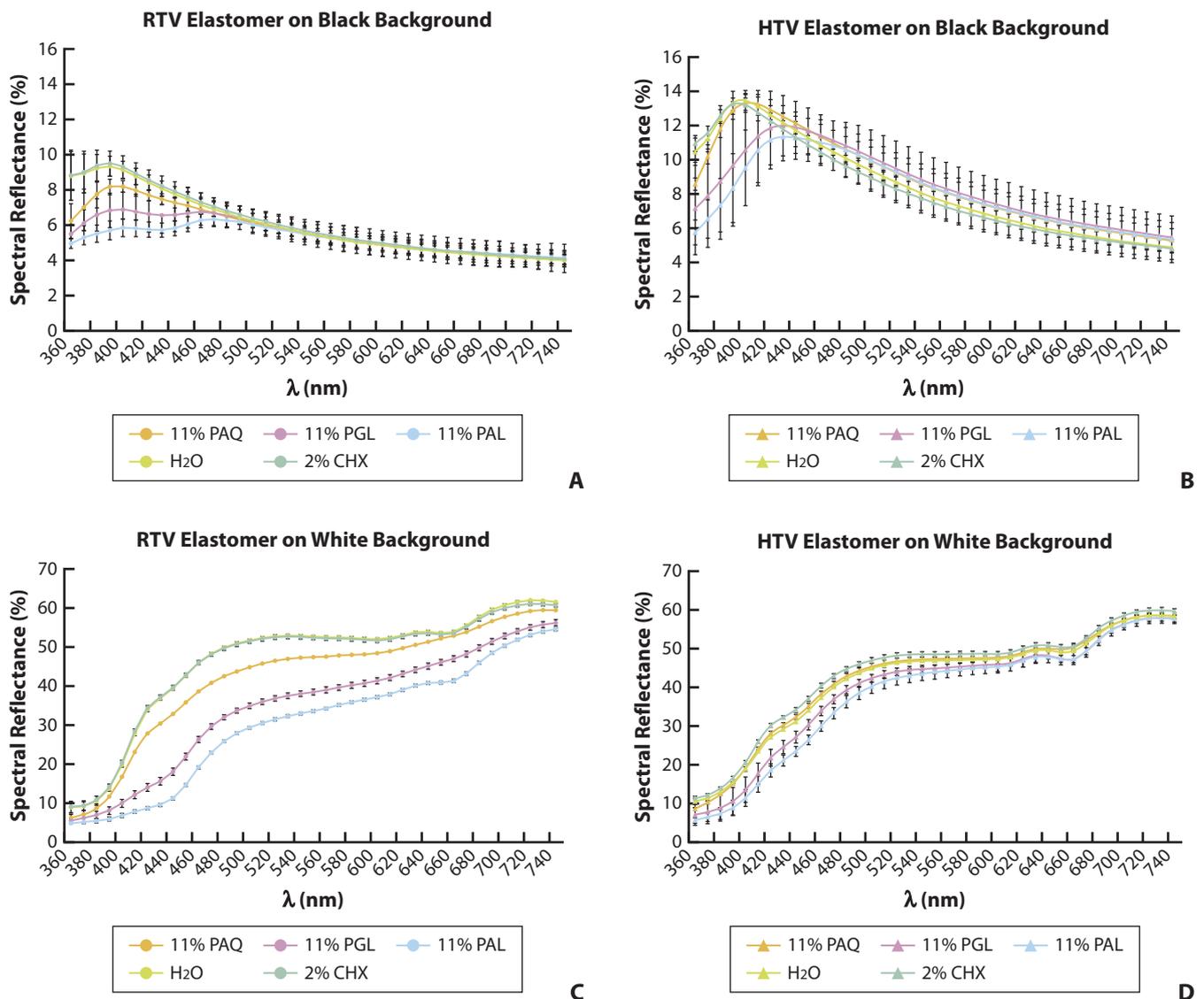


Figure 4. A-D, Mean (horizontal lines) and standard error (vertical lines) values of spectral reflectance of specimens after 60 days of immersion in solutions. CHX, chlorhexidine gluconate; HTV, high-temperature vulcanization; PAL, alcoholic propolis extract; PAQ, aqueous propolis extract; PGL, glycolic propolis extract; RTV, room temperature vulcanization.

DISCUSSION

Changes in color coordinate values, reflectance optical parameters, and spectral reflectance behavior according to wavelength and in the hardness of the 2 maxillofacial elastomers submitted to a period of immersion in different solutions were found in the present study. The null hypothesis that 60 days of immersion in solutions of Brazilian 11% green propolis extract, the control H₂O solution, and 2% CHX would not affect the optical parameters and hardness of maxillofacial elastomers was partially rejected.

Propolis is known to contain oil- or water-soluble substances or substances soluble in both solvents.³¹ Most of the propolis components are soluble in oil, and

therefore, the method most commonly used for propolis extraction uses hydrated ethyl alcohol as a solvent.^{28,29,31} In the present study, the pigmentation and change in the optical parameters of the elastomers in 11% PAL and 11% PGL may have been related to the polarity of the extract and the maxillofacial elastomer. As a solvent, ethanol permits the selective extraction of some components directly related to biological activity,²⁸ although the alcohol extract may be contraindicated for some individuals.³¹ The present results are consistent with those of the study by Heidrich et al²⁴ who reported that the color of acrylic resin complete denture bases changed when the polymer was exposed to PGL over 1 year.

Among the residual components of the propolis extract solutions evaluated, wax and chlorophyll contain dyes that

Table 6. Global test of spectral reflectance values of all elastomer specimens measured against black and white background after 60 days of immersion in solutions

| Elastomers | Black Background | | | | White Background | | | |
|---------------------|------------------|----------------|-------|-------|------------------|----------------|--------|-------|
| | df | Sum of Squares | F | P | df | Sum of Squares | F | P |
| RTV | | | | | | | | |
| Immersion solutions | 4 | 0.06 | 46.12 | <.001 | 4 | 0.35 | 302.54 | <.001 |
| Residual | 45 | 0.01 | — | <.001 | 45 | 0.01 | — | <.001 |
| HTV | | | | | | | | |
| Immersion solutions | 4 | 0.06 | 6.05 | <.001 | 4 | 0.03 | 13.25 | <.001 |
| Residual | 45 | 0.12 | — | <.001 | 45 | 0.03 | — | <.001 |

HTV, high-temperature vulcanization; RTV, room temperature vulcanization. $P < .05$ denotes statistically significant difference.

may have caused pigmentation on the surface of the elastomer, altering its chromatic pattern, translucency, and opacity. The results of the present study suggest that the propolis solution could be used for maxillofacial prosthesis disinfection when the formulation conditions are controlled. However, further studies are needed to test the reliability of various propolis sources.

The results showed that ΔE values obtained with 11% PAQ and 11% PGL for the HTV elastomer may be considered imperceptible and clinically acceptable. The increase in temperature during propolis extraction reduces the viscosity of a solution and increases its diffusibility through the gel layer and the membrane itself.^{26,31} The effect of temperature on the processing of an aqueous propolis extract leads to an increased permeate flow and reduction of the viscosity of the solution. Thus, the 11% PAQ solution is less viscous than the 11% PAL and 11% PGL solutions. The adherence of substances from 11% PAL to the elastomers is probably due to the surface energy relationship¹¹ between them. This adherence may interfere with the disinfection procedures because it may affect the esthetic properties of the elastomer.²⁴

Transmittance L^* and C^* coordinate values for 11% PAQ were similar to those of H_2O for the HTV elastomer. In addition, the TP values of the HTV elastomers were similar for the propolis extract solutions and H_2O . However, among the propolis extract solutions used, TP values showed a difference for the RTV elastomer. The difference in results between the elastomers for the same solutions may have been related to the contraction of continuous polymerization, which starts during polymerization and continues even after clinical polymerization is complete.⁶ It is believed that the additional or residual polymerization causes not only the dimensional alteration of the silicone, but also changes in the chromatic pattern.^{6,22} The amount of this factor may have effects on the difference between the materials to influence the absorption and solubility of the residue in the propolis solutions, altering the optical behavior of its polymer matrix.²² Also, the differences in the crystalline

Table 7. Hardness (Shore A) values of all specimens at baseline and after 60 days of immersion in solutions

| Elastomers | RTV | | HTV | | |
|------------|-----------|------------------|------------------|------------------|------------------|
| | Solutions | Baseline | After 60 d | Baseline | After 60 d |
| 11% PAQ | | 30.18 \pm 0.39 | 30.15 \pm 0.23 | 15.47 \pm 0.37 | 15.74 \pm 0.40 |
| 11% PGL | | 30.22 \pm 0.52 | 30.48 \pm 0.55 | 15.55 \pm 0.27 | 15.60 \pm 0.33 |
| 11% PAL | | 30.15 \pm 0.31 | 30.78 \pm 1.01 | 15.28 \pm 0.38 | 15.35 \pm 0.90 |
| H_2O | | 30.10 \pm 0.51 | 30.23 \pm 0.20 | 15.52 \pm 0.20 | 15.44 \pm 0.37 |
| 2% CHX | | 30.39 \pm 0.35 | 30.31 \pm 0.25 | 15.54 \pm 0.21 | 15.64 \pm 0.36 |

CHX, chlorhexidine gluconate; HTV, high-temperature vulcanization; PAL, alcoholic propolis extract; PAQ, aqueous propolis extract; PGL, glycolic propolis extract; RTV, room temperature vulcanization. Values are given as means \pm standard deviation.

Table 8. Two-way repeated-measures ANOVA of hardness (Shore A) for all specimens at baseline and after 60 days of immersion in solutions

| Variation Factor | df | Sum of Squares | Mean Square | F | P |
|--------------------------|----|----------------|-------------|------|-----|
| Solution | 4 | 1.37 | 0.34 | 1.18 | .33 |
| Between subjects | 45 | 13.08 | 0.29 | — | — |
| Period | 1 | 0.76 | 0.76 | 4.06 | .05 |
| Solution \times period | 4 | 1.59 | 0.40 | 2.11 | .09 |
| Within subjects | 45 | 8.47 | 0.19 | — | — |

$P < .05$ denotes statistically significant difference.

and amorphous structure between elastomers may also be related to their differences in opacity compromising the esthetics of the prosthesis.^{8,12}

In general, against a white background, the reflectance of both elastomers in the present study was lower for short wavelengths and increased with increasing wavelengths. Color reading against a black and white background causes a difference in the observation of the reflectance behavior of the elastomers.³⁴ The spectral reflectance of the specimens after immersion differed between the solutions, demonstrating that not only the perception of the color of the prosthesis but also its brightness and opacity can change for both elastomers.

No significant effect was found on the Shore A hardness for either elastomer, suggesting that the color change was only superficial. However, previous in vitro studies^{5,6,9,19} have reported that the MDX4-4210 elastomer submitted to disinfection and associated with the simulation of artificial aging showed reduction of hardness by sorption of substances and/or degradation of the material.

The present study had some limitations. The experimental disinfection period may be different from the period chosen by patients wearing a maxillofacial prosthesis. Further analyses of different propolis concentrations and extraction processes would be interesting for the understanding of potential color pigments.

CONCLUSIONS

Based on the findings of this in vitro study, the following conclusions were drawn:

1. HTV elastomers immersed in 11% aqueous and glycolic green propolis extracts showed clinically

acceptable and imperceptible color changes, whereas the room temperature vulcanized elastomers immersed in 11% alcoholic and glycolic green propolis extracts showed clinically unacceptable and perceptible changes.

- Both elastomers immersed in 11% aqueous green propolis extract and 2% CHX showed translucency and opacity parameters similar to those of the control group immersed in distilled water.
- The Shore A hardness of both elastomers showed clinically acceptable values following a 60-day immersion protocol in all tested disinfectant groups.

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