

EFFECTS OF THERMOCYCLING ON MECHANICAL PROPERTIES OF SOFT LINING MATERIALS

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ABSTRACT

Soft linings are materials used to reduce the tension and forces of mastication, forming all or part of the fitting surface of a denture. This study evaluated the effect of thermocycling on water absorption, solubility, Shore A hardness and color stability of permanent soft liner materials. **MATERIAL AND METHODS:** Two chemically activated soft liner materials (Sofreliner S; GC Reline Ultrasoft) were tested. Twenty cylindrical specimens (30.0 x 1.0 mm) were prepared for measuring water absorption and solubility and another twenty (30.0 x 3 mm) for analyzing Shore

A hardness and color stability. Color was measured by a spectrophotometer before and after 2000 thermocycles. A one-way ANOVA test and Tukey test at a 5% confidence level ($p < 0.05$) were performed. **RESULTS:** The results did not show statistical differences for water absorption, solubility or color stability. The post-thermocycling Shore A hardness values were significantly higher than those before the treatment. **CONCLUSION:** Thermocycling of soft liner materials increased Shore A hardness.

Key words: absorption, solubility, hardness, color, denture liners.

INFLUENCIA DEL TERMOCICLADO SOBRE LAS PROPIEDADES FÍSICAS DE LOS MATERIALES SOFTS PARA REBASADO

RESUMEN

Objetivo: Los materiales para rebasado tienen como propósito disminuir la tensión, la presión masticatoria en los tejidos de soporte y aumentar la retención de la dentadura, además de ser indicados para prótesis buco-maxilo-faciales. El objetivo de este estudio fue evaluar el efecto del termociclado en los materiales para rebasado blandos considerados definitivos y evaluar las alteraciones en relación a la absorción de agua, solubilidad, dureza Shore A y estabilidad de color. **Materiales y Métodos:** Dos materiales para rebasado blandos definitivos (Sofreliner S y GC Reline Ultrasoft) fueron examinados. Fueron confeccionados 20 muestras, midiendo 30 mm de diámetro x 1 mm de espesor; aquellos destinados al test de absorción y solubilidad y 20 muestras con 30 mm de diámetro x 3 mm de espesor para dureza y estabilidad de color. Fueron realizados los tests de absorción y solubilidad. La dureza fue medida en un durometro

y la estabilidad de color por medio de un espectrofotómetro en los periodos inicial y después de 2000 ciclos de termociclaje. Una vez obtenidos los datos, estos fueron submetidos al análisis de varianza (ANOVA) seguido por el test Tukey ($p < 0,05$). **Resultados:** En relación a la absorción, solubilidad y estabilidad de color no hubo diferencia estadísticamente significativa entre los materiales, en relación al test de dureza fue constatada una diferencia estadísticamente significativa entre los periodos y los materiales examinados. **Conclusión:** El termociclaje no interfirió en la absorción, solubilidad y estabilidad de color de los materiales evaluados, sin embargo interfirió de forma significativa en la dureza Shore A. Los materiales tuvieron sus valores promedios de dureza aumentados después del termociclaje.

Palabras clave: absorción; solubilidad; dureza; color; rebasado de dentadura.

INTRODUCTION

Soft liners enable missing facial components to be substituted without reducing the efficiency of the denture, by means of acrylic resins, polyurethanes, polyvinyl chlorides, polyethylenes and silicones, which are often used in the prosthetic reconstruction market^{1,2}. In addition to conventional prostheses, they are also used for oral and maxillofacial prostheses such as palatal obturators and ocular prosthesis linings, and for making nasal, auricular and ocular-palpebral prostheses³⁻⁵.

During the time that acrylic resin or silicone-based soft materials are used, they begin to show some undesirable features such as alterations in dimensions, color, solubility, fluid absorption and hardening, which affect their longevity⁶⁻⁸. Absorption and solubility are evaluated simultaneously through the processes of water gain and loss of soluble components, which occur by means of diffusion of molecules from the medium (water) into the material, altering the physical and mechanical properties⁹.

In addition to absorption and solubility, hardness is also responsible for the long-term failure of soft materials¹⁰. It may be related to the chemical composition of the material, manipulation, polymerization method and sample thickness, which according to some authors should be 3mm in order to be considered ideal^{11,12}.

There are several factors associated to color instability in resilient lining materials, such as accumulation of stains, dissolving of composites, degradation of intrinsic and extrinsic pigments, among others, which will affect long-term use¹³⁻¹⁵. Some studies have concluded that color stability is one of the most important clinical properties for all lining materials because it indicates the aging or damage that the lining materials may have undergone over time^{16,17}.

One way of evaluating the physical and mechanical properties of these materials is to subject them to an *in vitro* simulation of the aging that would occur in the medium in which they are used, by means of an accelerated aging test by thermocycling^{18,19}.

The aim of this study was to evaluate the effect of thermocycling on two soft liners considered permanent, regarding water absorption, solubility Shore A hardness and color stability.

MATERIALS AND METHODS

Two silicone-based lining materials were used for this study: Sofreliner S (Tokuyama Dental Corp., Tokyo, Japan) and GC Reliner Ultrasoft containing sealant (GC Corp., Tokyo, Japan). Forty samples were prepared – 20 for each material studied – using a mold 30 mm in diameter, with a thickness of 1mm for absorption and solubility tests, and 3 mm for hardness and color stability tests²⁰.

The materials studied (GC Reline, Sofreliner S) are sold as auto-mix products. They were placed in the metal mold and transferred to a hydraulic press (Midas Dental Products Ltda., Araraquara, Sao Paulo, Brazil) with a 1.25-ton load, and left for 20 minutes until the material was fully polymerized.

After polymerization, the samples were flaked and excess was trimmed with a scalpel blade # 5⁷.

For the GC Reline material, a sealant containing a modifier A + B was mixed and applied to the surface of the samples with 4 minutes polymerization time.

When the samples were ready, they were stored in distilled water in an oven at 37±1°C (Odontobras, Sao Paulo, Brazil), for 24 hours before starting the tests^{12,21}.

The specific samples for the absorption and solubility tests were initially subject to the desiccating

process according to ADA specification # 12, which consists of storing the samples in a glass desiccator containing silica gel in a suitable environment under vacuum. The samples remained in this environment in the oven at a temperature of 37±1°C (Odontobras, São Paulo, Brazil), and were weighed daily with an analytical precision balance (BEL Equipamentos Analítico, SP, Brazil) until they attained constant mass (W1).

The samples subsequently underwent the thermal cycling test and were weighed again (W2), and desiccated to the final weight (W3). The degree of absorption and solubility were calculated by means of the following formulae:²²

$$\% \text{ Absorption} = \frac{(W2-W3)}{W1} \cdot 100$$

$$\% \text{ Solubility} = \frac{(W1-W3)}{W1} \cdot 100$$

The Shore A hardness tests were conducted before and after thermocycling, using a Shore A durometer (model GSD 709 Teclock, Osaka, Japan) following specification D-2240 of the American Society for Testing and Materials (ASTM) at room temperature. This method is based on the penetration of a needle on the surface of the material with a constant 10N load. Hardness values are expressed in Shore A units on a scale of 0 to 100, and hardness is proportional to the penetration of the needle, i.e., the greater the penetration, the lower the value indicated on the scale. Five readings were taken for each sample in the direction of the diameter, to obtain an average value for Shore A hardness²³. Each reading was carried out after 1 second contact between the needle and the material.

All samples underwent an initial chromatic analysis using an Ultraviolet Visible Reflection Spectrophotometer*, Model UV-2450 (Shimadzu, Kyoto, Japan), with color alterations calculated by means of the CIE L*a*b* System established by the International Commission on Illumination (Comission International de L'Eclairage; CIE)²⁵. The axial coordinate "L" is known as luminosity and goes from 0 (black) to 100 (perfect white). Coordinate "a" represents the amount of red (positive values) and green (negative values), while coordinate "b" represents the amount of yellow (positive values) and blue (negative values). This system enables the

value of ΔE (color variation) to be calculated between two readings, by means of the formula:

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

After the initial readings for weight (W1), Shore A hardness and color stability, the samples underwent the thermocycling test in a thermal cycle simulation machine²⁰. Two thousand cycles were conducted, simulating 2 clinical years of use of these materials with the samples immersed in distilled water, undergoing alternate 60-second baths at temperatures of $5\pm 1^\circ\text{C}$ and $55\pm 1^\circ\text{C}$ ^{18,26}. At the end of the procedure, the samples underwent further readings of absorption and solubility, Shore A hardness and color stability, as described above.

RESULTS

Statistical analysis of the values was conducted by means of an analysis of variance test (ANOVA) followed by the Tukey test at a significance level of 5%.

There was no statistically significant difference ($p < 0.05$) in either material, whether for water absorption or for solubility (Tables 1, 2 and 3).

Considering both materials at similar times, there was a statistically significant difference in Shore A hardness values, both for the initial period ($p < 0.0001$) and for the period after thermocycling ($p = 0.018$). For both materials considered separately at different times, there was a statistically significant difference ($p < 0.0001$). These values are shown in Tables 4 and 5.

Table 1: ANOVA Solubility

Source	DF	Type III SS	Mean Square	F Value	Pr > F
Resin	1	0.1123501	0.1123501	0.2353	0.63813
Residue	18	8.5928888	0.4773827		
Total	19	8.7052388			

*Statistically significant at the 5% level.

Table 2: ANOVA Absorption

Source	DF	Type III SS	Mean Square	F Value	Pr > F
Resin	1	0.9825656	0.9825656	0.8166	0.61848
Residue	18	21.6592806	1.2032934		
Total	19	22.6418462			

*Statistically significant at the 5% level.

Table 3: Mean values (standard deviation) for water absorption, solubility.

Material	Water absorption (%)	Solubility(%)
Sofreliner S	1.78 (1.14) A	0.20 (0.96) A
GC Reline Ultrasoft	1.34 (1.04) A	0.05 (0.12) A

*Mean values followed by the same letter in columns do not differ statistically according to the Tukey test ($p < 0.05$).

Table 4: Shore A hardness ANOVA

Source	DF	Type III SS	Mean Square	F Value	Pr > F
Thermal cycling	1	970.2250000	970.2250000	403.7931	0.00001
Resin	1	275.6250000	275.6250000	114.7110	0.00001
Termoc x Resin	1	105.6250000	105.6250000	43.9595	0.00001
Residue	36	86.5000000	2.4027778		
Total	39	1437.9750000			

*Statistically significant at the 5% level.

Table 5: Mean values (standard deviation) for Shore A hardness.

Material	Before	After thermal cycling
Sofreliner S	13.1 (1.10) A,a	26.2 (1.13) A,b
GC Reline Ultrasoft	21.6 (1.57) B,a	28.2 (2.15) B,b

*Mean values followed by different capital letters in columns and lowercase in lines differ statistically according to the Tukey test ($p < 0,05$).

Table 6: Color ΔE ANOVA

Source	DF	Type III SS	Mean Square	F Value	Pr > F
Resin	1	0.0092507	0.0092507	0.00001	0.92662
Residue	18	20.5461249	1.1414514		
Total	19	20.5553756			

*Statistically significant at the 5% level.

There was no statistically significant difference between materials regarding the mean ΔE ($p < 0,05$) values, as shown in Tables 6 and 7.

DISCUSSION

The aim of this study was to evaluate the effect of thermocycling on absorption, solubility, Shore A hardness and color stability in two silicone-based soft lining materials (Sofreliner S and Reline Ultrasoft) considered permanent.

Table 3 shows that neither material had statistically significant differences either for absorption or solubility. This may be due to the fact that they are both based on silicone, which has a lot of crosslinking between the charge and the silicone, preventing the presence of micropores through which water molecules could diffuse^{22,27}. However, lengthy storage periods may promote water absorption in silicone-based materials, probably due to the type of charge in their composition, plus the low degree of adhesion among silicone polymers, leading to hardening²⁰.

Studies have shown that in order for a resilient lining material to be considered ideal, it must have values lower than 0.8 mg/cm² (2.45%) for absorption and 0.03 mg/cm² (0.08%) for solubility. Thus, we have found that the results obtained are in agreement with scientific literature^{7,22}.

Regarding Shore A hardness, Table 5 shows that the materials had statistically significant differences before and after thermocycling, and when compared to each other, there were also statistically significant differences before and after thermocycling.

Table 7: Mean values for color ΔE (standard deviation) between materials.

Material	ΔE (SD)
Sofreliner S	2.17 (1.15) A
GC Reline Ultrasoft	2.13 (0.97) A

*Mean values followed by different capital letters in columns and lowercase in lines differ statistically according to the Tukey test ($p < 0,05$).

According to the literature, the thermal cycling procedure can promote hardening of materials, consequently increasing hardness values. This is due to changes in the composition of silicone-based materials, and water absorption^{18,19,20}. In autopolymerizing materials, polymerization takes place at room temperature, and the process is continuous over time, which may be reflected by an increase in hardness values during its useful life^{10,12}.

Results in literature for the materials examined in this study also showed a statistically significant increase in mean values for Shore A hardness after thermocycling. The statistical difference between materials was probably due to their individual characteristics^{28,29}.

Table 7 shows that color altered in both materials (Sofreliner S y GC Reline Ultrasoft), as indicated by ΔE greater than zero. However, there was no statistically significant difference regarding the results of color stability between materials (Sofreliner S and GC Reline Ultrasoft). This color alteration may be attributed to the individual properties of each material, such as absorption and lack of solubility²².

According to the National Bureau of Standards (NBS), color variation ΔE lower than 1 is consid-

ered very low, 1 to 2 is clinically acceptable, and higher than 3.3, clinically perceptible^{30,31}. In this study, the materials evaluated had values very close to $\Delta E = 2$, (Table 7), indicating a clinically imperceptible color alteration. Color maintenance in the material is important to patients, who are more satisfied when the lining material remains stable and the color is undistinguishable from the basic prosthesis material³². These results are relevant in that they confirm the complexity of soft liner materials.

CORRESPONDENCE

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CONCLUSION

From the results obtained through the methodology we proposed, it may be concluded that:

- All samples underwent a color change considered clinically acceptable, and there was no statistical significant difference when compared.
- Thermocycling did not interfere with absorption, solubility or color stability of the soft liner materials.
- There was a statistically significant increase in the mean Shore A hardness values of the materials examined after thermocycling.

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