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Effect of different pigmentations, hygiene protocols, and time on maxillofacial silicone properties

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Objective: To evaluate the effects of pigmentation, hygiene protocols, and time on the physical, mechanical, and chemical properties, as well as the thermal behavior of MDX4-4210 and Silpuran 2420 silicones. **Methods:** Samples without pigmentation (WP;n=45), with intrinsic pigmentation (IP;n=45), and with intrinsic and extrinsic pigmentation (IEP;n=45) were tested. Samples were washed daily with neutral soap and immersed in water (control - C), washed daily with neutral soap and immersed in 2% hydrogen peroxide (HP2%), or washed daily with neutral soap and immersed in 0.15% triclosan (T0.15%) once a week for six months (T1). Subsequently, they were exposed to ultraviolet radiation and humidity, simulating 12 months (T2). Color, Shore A hardness, surface roughness, chemical properties, and thermal behavior were evaluated at T0, T1, and T2. Data were analyzed using a general linear model employing the Wald test and repeated measures for color and hardness changes, and ANOVA with Tukey's post hoc test for surface roughness (α=0.05). **Results:** In MDX4-4210, WP and IEP had the greatest color change at T2 with T0.15% ($p=0.020$). IEP showed higher roughness at T1 and T2 but lower hardness variation ($p<0.001$). Hygiene protocols influenced roughness and hardness ($p<0.05$). In Silpuran, IEP had the highest color variation at T2, with reduced roughness and hardness, regardless of hygiene ($p<0.001$). **Conclusion:** IEP impacted color stability over time in both silicones when exposed to HP2% and T0.15%. Hardness changes did not compromise material quality. IEP groups showed lower roughness variation. Chemical and thermal behavior were unaffected by pigmentation or hygiene.

Keywords: Maxillofacial prosthesis. Silicones. Disinfection. Surface properties.

Introduction

Prosthetic rehabilitation is a viable option when facial deformities cannot be surgically corrected. Medical silicones are commonly used in the fabrication of maxillofacial prostheses due to their comfort and favorable properties, such as hardness, roughness, tensile strength, and tear resistance[1-6]. However, alterations in these properties can shorten the prosthesis lifespan and increase treatment costs, especially in countries where medical silicone is not readily available. Therefore, further studies are needed to evaluate alternative, cost-effective materials with desirable properties.

Several factors can alter the properties of silicone, including processing techniques, variations in light and ambient humidity, atmospheric pollution, body fluids, hygiene methods[1,3,7-21], and the addition of pigments[5,7,22]. Among these, hygiene methods and pigmentation can be controlled. Regarding pigmentation, adding pigments to the silicone base paste before mixing it with the catalyst is necessary to achieve an aesthetic skin color match, as silicone is naturally colorless. This process is known as intrinsic pigmentation. Additionally, to obtain realistic features with different tones and shades, extrinsic pigmentation is applied to the prosthesis surface after the polymerization process[2,3,7,11,13]. This can be done using ceramic pigments, silicone-based pigments, makeup powder, and oxides. Therefore, selecting a pigment compatible with silicone and using the appropriate dosage is essential[2,7,9,13].

Regarding hygiene methods for cleaning all surfaces of silicone facial prostheses, the literature describes various approaches, including brushing, microwaves, ultraviolet C light, and chemical agents such as chlorhexidine, natural extracts, peroxides, sodium hypochlorite, soap, and probiotics with anti-biofilm properties[1,3,10,13-16,18-21]. However, there is no consensus on a precise and safe hygiene protocol for cleaning and disinfecting silicone prostheses. Hydrogen peroxide[23,24] and triclosan[25-27] solutions have demonstrated good efficacy in biofilm control. Diluted hydrogen peroxide can be an option for cleaning prostheses and tissues[23], as it promotes oxygen release, loosening debris and removing light stains[26]. Additionally, it produces free hydroxyl radicals that cause oxidation of DNA, proteins, and lipids *in vivo*[24].

Triclosan at 0.15% reduced the microbial load and promoted the remission of denture stomatitis[25,26]. It disrupts the lipid bilayer membrane, affecting RNA and protein synthesis, ultimately leading to cell death[27]. Triclosan is a broad-spectrum antibacterial agent effective against Gram-positive bacteria (*Bacillus subtilis*, *Mycobacterium smegmatis*, *Staphylococcus aureus*), Gram-negative bacteria (*Escherichia coli*, *Salmonella typhimurium*, *Shigella flexneri*), as well as fungi and yeasts[28]. However, concerns have been raised regarding its potential toxicity. According to Dann and Hontela[29] (2011), triclosan may exhibit toxic effects at concentrations above 0.3%, potentially affecting endocrine function and environmental safety. In Brazil, its use is regulated by the National Health Surveillance Agency (Anvisa) through Resolution RDC No. 29, of June 1, 2012, which establishes a maximum allowed concentration of 0.3% in formulations[30], aligning with international safety standards. Both hydrogen peroxide and triclosan are widely available and cost-effective. If proven safe for use with silicone, they could be recommended as hygiene methods for maxillofacial prostheses.

This *in vitro* study evaluated the color change, Shore A hardness, surface roughness, and microstructural properties of two medical silicones, considering the effects of pigmentation, hygiene protocols, and time. The evaluation was conducted after six months of natural aging and 12 months of accelerated aging. The tested hypothesis was that these factors would not significantly affect the materials' properties. This study addresses this gap, providing valuable insights to enhance the longevity and aesthetics of maxillofacial prostheses.

Materials and Methods

The materials used in this study are listed in Table 1. A total of 135 disk-shaped specimens (3 mm thick × 12 mm in diameter) were distributed into three groups: WP (colorless), IP (intrinsic pigmentation), and IEP (intrinsic + extrinsic pigmentation). The sample size was determined based on previous studies[2-6,8,9,13,18,22]. MDX4-4210 silicone was prepared at a 10:1 ratio (base:catalyst), while Silpuran 2420 was mixed in a 1:1 ratio (base:catalyst), following the manufacturers' recommendations.

Table 1. Materials used in the study.

Material	Trademark
Silastic® MDX4-4210	BioMedical Grade Elastomer, Dow-Corning, USA
Silpuran 2420	Wacker Chemie AG, München
Silc Pig® (Beige – PMS 488C)	Smooth-On, Inc., Macungie, Pennsylvania, USA
Silc Pig® (Blood – PMS 7421C)	Smooth-On, Inc., Macungie, Pennsylvania, USA
Pre-painting fluid	Smooth-On, Inc., Macungie, Pennsylvania, USA
Psycho Paint	Smooth-On, Inc., Macungie, Pennsylvania, USA
Solvent for psycho paint	D-Limonene, Dim Clay, São Paulo, São Paulo, Brasil
Neutral soap	Pleasant, Perol Commercial and Industrial Ltd., São Paulo, São Paulo, Brasil.
Hydrogen peroxide at 2%	Daterra Manipulation Pharmacy, Ribeirão Preto, São Paulo, Brasil
Triclosan solution at 0,15%	Mix das essências, Belo Horizonte, MG, Brasil. Solution prepared in Laboratory of Research in Oral Rehabilitation of School of Dentistry of Ribeirão Preto (100 mL of the sodium hydroxide, 0,056 M + 0.15 g Triclosan + water q.s.p = final concentration of the 0.15%

For the WP group, the base and catalyst pastes were weighed on a digital balance and mixed under vacuum (Turbomix, EDG, São Bernardo do Campo, São Paulo, Brazil) for 120 seconds[2,3]. The mixture was then inserted into rectangular Teflon molds (12 mm long × 4 mm wide × 3 mm deep) and placed in a pneumatic press at 20 psi for 15 minutes to eliminate bubbles. Next, the set was placed in a polymerization oven (Odontobras Ind. and Trade Equip. Med. Odont. Ltda, Ribeirão Preto, São Paulo, Brazil) at 100°C for 60 minutes. After polymerization, the silicone sheets were cut into 12 mm diameter disks using circular sharp cutters[2,3]. The final dimensions were verified with a digital caliper (CD-6 CSX-B, Mitutoyo Ltda, Suzano, São Paulo, Brazil).

For the IP group, the beige pigment was weighed and incorporated into the silicone base paste at a 2% ratio[2-4,6]. The catalyst was then added, and the mixture was homogenized under vacuum for 120 seconds. The subsequent steps followed the same protocol as described for the WP group. After polymerization, half of the samples underwent extrinsic pigmentation, forming the IEP group. The specimen surfaces were pre-treated with a pre-painting fluid. A total of 0.02 g of blood-colored pigment was mixed with 2 g of Psycho-Paint A, followed by the addition of 2 g of Psycho-Paint B. To obtain a thin and uniform pigment layer, the mixture was diluted in an equal volume of Psycho-Paint solvent (2 parts), and 2 g of the final solution was applied to the specimen surfaces using a dry brush. For polymerization of the extrinsic pigment, the specimens were kept at room temperature for 24 hours.

All specimens were individually numbered and stored in a closed container, protected from light[10,22], until the initial measurements (T0) of color, hardness, surface roughness, and thermal analysis were performed. The specimens were randomly assigned to subgroups based on hygiene protocols, with randomization conducted using computer-generated numbers (Excel 2013, Microsoft Windows). All specimens underwent daily cleaning by being washed with one drop of neutral soap, using the index finger and thumb for 20 seconds. Additionally, once a week, they were immersed for 20 minutes in 5 mL of either water (control, C), 2% hydrogen peroxide (HP2%), or 0.15% triclosan (T0.15%), followed by rinsing under running water for 20 seconds. This protocol was maintained for six months (T1). The hygiene protocols were adapted for this study based on literature regarding denture hygiene[25,26] and maxillofacial prosthesis care[2,23]. All procedures were performed by the same operator (C.M.A.P.M.) to ensure consistency and standardization.

During the interval, specimens were kept near to an open glass window, exposed to environment light, temperature, and humidity until the next cleaning procedure[2,22]. The experiment took place from February to July 2023, thus encompassing the summer, autumn and winter seasons. This model has aim to simulate the use of the prosthesis during 6 months (T1). Subsequently, specimens were exposed to ultraviolet-B (UV-lamp TL 40 W/12RS B medical, Phillips, The Netherlands) for 240 hours and subjected to condensation at 50°C for an additional 240 hours (UV Cond, Comexim and Raw Materials Industry Commerce Ltda, São Paulo, Brazil), simulating 12 months of prosthesis use (T2)[2].

Color change[1,2,5,8,10] was measured using a spectrophotometer (Color Eye 7000; Macbeth, Newburgh, NY) following the Color System Standard Commission Internationale de L'Eclairage (CIE LAB; American Dental Association). The color change (ΔEab) was calculated using the formula:

$$\Delta E_{ab} = \sqrt{(\Delta L)^2 + (\Delta a)^2 + (\Delta b)^2}$$

Where ΔL represents the variation in luminance (brightness), Δa indicates the variation in the red (+) or green (-) intensity, and Δb represents the variation in yellow (+) or blue (-) intensity. To classify the color change, the National Bureau of Standards (NBS) was applied using the formula:

$$NBS = \Delta E_{ab} \times 0.92$$

The color change was categorized as: very light (0.0-0.5), light (0.5-1.5), remarkable (1.5-3.0), appreciable (3.0-6.0), high (6.0-12.0), and very high (>12.0).

Shore A hardness analysis was conducted in accordance with ASTM D 2240-64 standards and previous studies[2,4]. A Shore A Durometer (Nishi Tokyo Seimitsu Co Ltd, Tokyo, Japan) was used with a 1 kg load applied for 5 seconds. The test specimen was visually divided into four quadrants, with measurements taken at the center of each quadrant and an additional measurement at the center of the specimen to obtain the mean value. To comply with ASTM D 2240-64 standards and perform indentations on 6 mm specimens, two specimens were stacked.

In the sequence, surface roughness[2,4,5] was measured with a rugosimeter (Rug 0.3; Prazis, Buenos Aires, Argentina) with 0.01 µm resolution, 0.8 mm cut-off length, 4.8 mm transverse length, and 0.5 mm/sec speed. Three measurements (in the center, at 5 mm to the right, and at 5 mm to the left of the center) were performed for each specimen and the mean value was used as the roughness value (Ra; µm).

Chemical property was analyzed using Fourier Transform Infrared Spectroscopy (FTIR) and the thermal comportment was analyzed by Differential Scanning Calorimetry (DSC), and Thermogravimetric Analysis (TGA)[2]. FTIR is a spectroscopic technique used to identify the functional groups and chemical bonds present in the material, revealing its molecular composition[31]. DSC analyzes thermal transitions (such as melting, crystallization, and glass transition), allowing the energetic behavior of the material during heating or cooling to be understood. TGA monitors the variation in material mass as a function of temperature, being essential for evaluating thermal stability and degradation processes[32]. FTIR was performed with the Nexus 4700 FTIR spectrophotometer (Thermo Nicolet, Berkeley, California, USA) and the measurements occurred in the region between 4000 to 400 cm-1, using 20 sweeps of the attenuated total reflection (ATR) kit. The DSC and TGA tests were performed simultaneously (SDT 600, TA Instruments, Newcastle, England), and the thermogravimetric curves were obtained in a DTA/TGA/DSC thermal analysis system (TA Instruments Q-600 - Simultaneous). The specimens of 4 mg were heated from room temperature (23°C) to 550°C in a synthetic air atmosphere. After reaching 550ººC, an oxygen atmosphere was used, with an average flow of 50 mL / minute^{-1} up to 900ººC.

Measurements of the color, Shore A hardness, surface roughness, chemical properties, and thermal comportment were obtained before hygiene protocols exposition (T0), after 6 months of hygiene protocols (T1), and after the accelerated aging (T2).

Normality (Shapiro-Wilk test) and homoscedasticity (Levene) tests were performed. Color change and Shore A hardness data met the assumptions of normal distribution. These dependent variables were analyzed by the General Linear Model (GLM) with repeated measures and Wald post-hoc test. Roughness data did not present a normal distribution, so data were analyzed by ANOVA with repeated measures and Tukey post-hoc test. A 95% confidence interval was considered. For each material, the independent variables included pigmentation (WP, IP, and IEP), hygiene protocols (control, HP2%, and T0.15%), and time (T0, T1, and T2). The time influence was analyzed by variation (D) between T1-T0 (DT1) and T2-T0 (DT2) for all dependent variables. The analyses were conducted using SPSS software (version 21, IBM Corp) by a blinded researcher (C.H.L.S). The results of the chemical and thermal analyses are presented in tables and figures and were interpreted and discussed.

Results

The environmental conditions for time T1 are in Figure 1. Color change (MDX4-4210: p=0.020; Silpuran 2420: p<0.001), shore A hardness (MDX4-4210: p<0.001; Silpuran 2420: p<0.001), and roughness (MDX4-4210: p<0.001; Silpuran 2420: p<0.001) were influenced by interaction of factors (Table 2). For MDX4-4210/IEP/T0.15% exhibited the highest color change, while the IP/T0.15% group showed small variations. Silpuran 2420/IEP/T0.15% exhibited the greatest color change at ΔT2. The NBS values are showed in Figure 2.

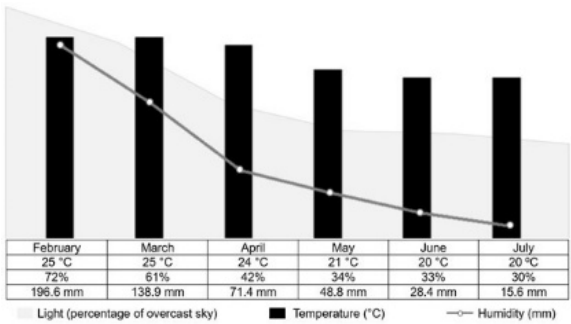


Figure 1. Environment conditions during first experimental period.

Table 2. Comparison [means (standard deviation)] of the interaction Pigmentation × Hygiene protocols × time on color change, hardness and surface roughness (μm).

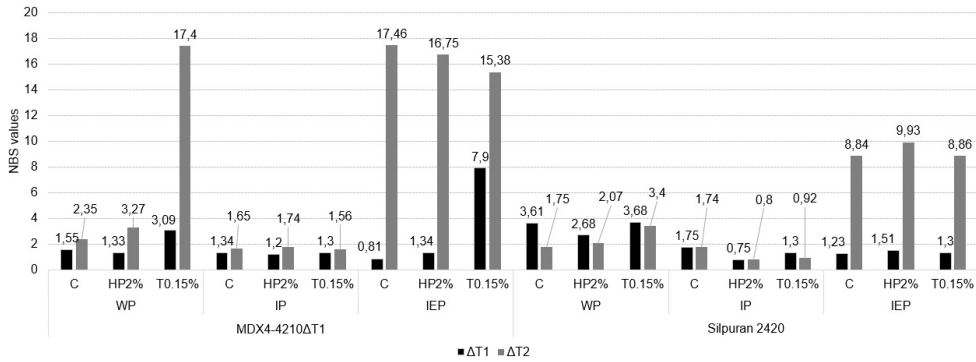
			MDX4-2410		P*	Silpuran2420		P*
			ΔT1	ΔT2		ΔT1	ΔT2	
Color change	WP	C	1.69 (0.56)	2.56 (0.87)	0.02	3.93 (0.89)	1.90 (0.75)	<0,00
			aBβ	bBα		aAα	bBβ	
		HP2%	1.45 (0.49)	3.55 (1.00)		2.91 (0.70)	2.25 (0.52)	
			aBβ	bBα		aBα	bBβ	
		T0.15%	3.36 (1.33)	18.98 (2.78)		4.01 (1.19)	3.67 (1.01)	
			aAβ	aAα		aAα	bAα	
	IP	C	1.46 (0.34)	1.80 (0.36)		1.91 (0.56)	1.89 (0.59)	
			abAα	bAα		bAα	bAα	
		HP2%	1.30 (0.29)	1.89 (0.68)		0.82 (0.48)	0.87 (0.50)	
			aAα	cAα		bBα	bAα	
		T0.15%	1.40 (0.45)	1.70 (0.64)		1.40 (0.48)	1.00 (0.55)	
			cAα	cAα		bABα	cAα	
	IEP	C	0.88 (0.62)	17.82 (1.76)		1.34 (0.84)	9.61 (1.12)	
			bBβ	aABα		bAβ	aAα	
		HP2%	1.46 (1.73)	18.21 (2.36)		1.64 (2.06)	10.8 (2.86)	
			aBβ	aAα		bAβ	aAα	
		T0.15%	8.59 (0.34)	16.72 (1.19)		1.41 (0.66)	9.64 (2.08)	
			bAβ	bBα		bAβ	aAα	

Continue

Continuation

Shore A hardness	WP	C	2.32 (0.74)	2.01 (0.86)	<0,00	2.48 (0.43)	1.56 (0.46)	<0,00
			cBα	cCβ		aAα	aAβ	
		HP2%	3.01 (0.51)	3.06 (0.63)		2.03 (0.44)	1.28 (0.48)	
			aAα	aBα		aAα	aAβ	
		T0.15%	3.36 (0.60)	3.78 (0.57)		2.08 (0.55)	1.45 (0.59)	
			aAβ	aAα		aAα	aAβ	
	IP	C	3.61 (0.33)	3.71 (0.28)		1.28 (0.65)	0.78 (0.68)	
			aAα	aAα		bBα	bBβ	
		HP2%	3.05 (0.60)	3.08 (0.62)		2.00 (0.60)	1.25 (0.45)	
			aBα	aBα		aAα	aAβ	
		T0.15%	2.73 (0.50)	2.05 (0.49)		1.81 (0.43)	1.28 (0.33)	
			bBα	bCβ		abAα	aAβ	
	IEP	C	2.91 (0.57)	2.58 (0.56)		1.73 (0.47)	0.38 (0.36)	
			bAα	bAβ		bAα	bAβ	
		HP2%	2.30 (0.38)	1.91 (0.44)		1.25 (0.46)	0.68 (0.41)	
			bBα	bBβ		bBα	bAβ	
		T0.15%	2.11 (0.33)	0.66 (0.26)		1.36 (0.55)	0.73 (0.47)	
			cBα	cCβ		bABα	bAβ	
Surface roughness	WP	C	0.43 (0.14)	0.51 (0.12)	<0.001	0.56 (0.11)	0.82 (0.12)	<0,001
			cAβ	bAα		cAβ	aAα	
		HP2%	0.38 (0.09)	0.46 (0.08)		0.49 (0.15)	0.74 (0.08)	
			cAβ	bAα		bABβ	bAα	
		T0.15%	0.37 (0.10)	0.44 (0.08)		0.42 (0.09)	0.61 (0.09)	
			cAβ	bAα		bBβ	bBα	
	IP	C	0.58 (0.08)	1.05 (0.20)		0.97 (0.12)	0.89 (0.08)	
			bAβ	aAα		aAa	aBβ	
		HP2%	0.52 (0.09)	0.50 (0.11)		0.86 (0.12)	1.00 (0.07)	
			bAα	bBα		aABβ	aAα	
		T0.15%	0.53 (0.19)	0.41 (0.06)		0.70 (0.07)	0.89 (0.08)	
			bAα	bBβ		aBβ	aBα	
	IEP	C	1.23 (0.15)	1.06 (0.07)		0.85 (0.14)	0.65 (0.07)	
			aAα	aAβ		bAα	bAβ	
		HP2%	1.24 (0.14)	0.97 (0.09)		0.85 (0.08)	0.59 (0.07)	
			aAα	aABβ		aAα	cAβ	
		T0.15%	1.17 (0.11)	0.91 (0.08)		0.77 (0.13)	0.57 (0.10)	
			aAα	aBβ		aAα	bAβ	

* Surface roughness: ANOVA with Tukey's post-hoc (Bonferroni-adjusted); Color change and Shore A hardness: Wald test (generalized linear model, Bonferroni-adjusted); Small letters: compare pigmentations for the same time and hygiene protocol; Capital letters: compare protocols for the same time and pigmentation; Greek letters compare time for the same protocol and pigmentation; Equal letters: indicate statistical similarity. WP: silicone colorless; IP: silicone with intrinsic pigment; IEP: silicone with intrinsic +extrinsic pigment; C: control - washing and immersion in water; HP2%: washing and immersion in hydrogen peroxide at 2%; T0.15%: washing and immersion in triclosan at 0.15%; ΔT1: variation between T1-T0; ΔT2: variation between T2-T0.



0.0-0.5: very light; 0.5-1.5: light; 1.5-3.0: remarkable; 6.0-12.0: high; >12.0: very high
Figure 2. NBS values and color change classification.

MDX4-4210 is a material with higher average hardness values than Silpuran (Table 3). Analyzing the variations of each material (Table 2), MDX4-4210/C/WP and IP were more stability over time; IEP/T0.15% had pronounced changes, with hardness increasing over time. For Silpuran 2420, hardness variation was smallest with WP/HP2%. The C protocol caused greater variation. Surface roughness was most affected in the IEP/T0.15%, while WP/C presents the least surface roughness for both materials (Table 2).

Table 3. Means [standard deviation (SD)] of the Shore A hardness.

			MDX4-4210			Silpuran		
			T0	T1	T2	T0	T1	T2
WP	C	Mean	35.25	37.58	37.27	10.98	13.47	12.55
		SD	0.58	0.43	0.46	0.38	0.21	0.29
	HP2%	Mean	34.72	37.73	37.78	11.20	13.23	12.48
		SD	0.56	0.38	0.36	0.41	0.37	0.27
	T0.15%	Mean	34.72	38.08	38.50	11.37	13.45	12.82
		SD	0.38	0.44	0.46	0.48	0.25	0.32
IP	C	Mean	34.92	38.53	38.63	9.87	11.15	10.65
		SD	0.35	0.16	0.41	0.65	0.40	0.23
	HP2%	Mean	35.22	38.28	38.30	9.80	11.80	11.05
		SD	0.57	0.27	0.45	0.58	0.39	0.29
	T0.15%	Mean	35.35	38.08	37.40	9.93	11.75	11.22
		SD	0.52	0.26	0.70	0.35	0.46	0.19
IEP	C	Mean	32.92	35.83	35.50	10.62	12.35	11.00
		SD	0.60	0.39	0.35	0.33	0.48	0.23
	HP2%	Mean	33.47	35.77	35.38	10.57	11.82	11.25
		SD	0.31	0.26	0.36	0.35	0.35	0.30
	T0.15%	Mean	33.70	35.82	34.37	10.63	12.00	11.37
		SD	0.34	0.29	0.39	0.36	0.38	0.19

WP: silicone colorless; IP: silicone with intrinsic pigment; IEP: silicone with intrinsic +extrinsic pigment; C: control - washing and immersion in water; HP2%: washing and immersion in hydrogen peroxide at 2%; T0.15%: washing and immersion in triclosan at 0.15%.

The decomposition processes initiating around 347–361°C (dt1) for MDX4-4210 and 329–336°C (dt1) for Silpuran 2420. MDX4-4210 showed increasing stability over time, with final decomposition temperatures (dt5) in the WP group rising from 563°C (T0) to 651°C (T2). Similarly, the IEP group exhibited enhanced stability, with dt5 increasing from 576°C (T0) to 603°C (T2). For Silpuran 2420, the WP group demonstrated a slight increase in stability, with dt5 rising from 534°C (T0) to 549°C (T2). The IP and IEP groups showed comparable trends, with final decomposition temperatures at T2 reaching 549°C and 548°C, respectively. Thermogravimetric analyses (TGA) indicated progressive increases in stability. For MDX4-4210, the WP group's primary decomposition point (tg5) increased from 533°C (T0) to 543°C (T2). The IEP group also showed growth, with tg5 rising from 540°C (T0) to 553°C (T2). Silpuran 2420 displayed smaller increases in tg5 across groups, with values reaching 522°C (WP), 522°C (IP), and 518°C (IEP) at T2. Decomposition curves revealed processes beginning near 300°C and extending to approximately 550°C. Both silicones left residues (35–57% of mass) composed primarily of SiO^2 and SiC. The main exothermic DSC peak corresponded to silicone polymerization, with minor peaks indicating secondary processes, including siloxane hydrolysis. Pigmentation not influenced in this comportment (WP = 562°C; IP = 563°C; IEP = 566°C). Time did not influence stability until T2, where samples exhibited slightly higher stability (582°C) (Table 4). FTIR (Figure 3) confirmed these findings, with preserved Si-O-Si stretching peaks at 1082 cm^{-1} and 707 cm^{-1}, as well as Si-CH3 and CH2-stretch modes around 1260 cm^{-1} and 2970 cm^{-1}. The spectra revealed that pigmentation and time had minimal impact on polymer structures, maintaining relative peak intensities in all groups.

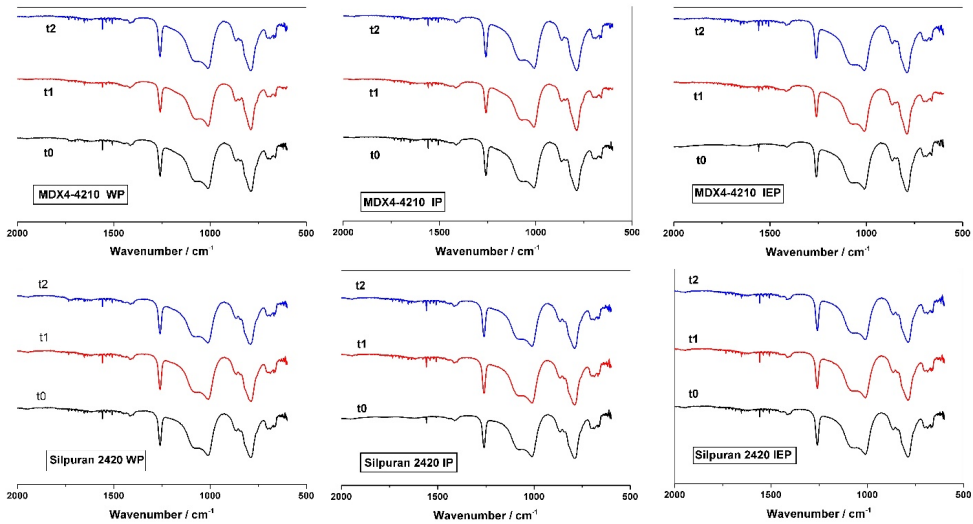


Figure 3. Fourier Transform Infrared Spectroscopy (FTIR) curves of hygiene protocols and pigmentation groups.

Table 4. Temperatures of the thermoanalytical curves of the Differential Scanning Calorimetry (DSC) and Thermogravimetry (TGA) tests of the silicone MDX4-4210 and Silpuran 2420 of the colorless (WP), with intrinsic pigmentation (IP), and with intrinsic+extrinsic pigmentation (IEP) at the T0, T1 and T2 times.

	Groups	Times	Points of the DSC curves						Points of the TGA curves				
			dt1	dt2	dt3	dt4	dt5	dt6	tg1	tg2	tg3	tg4	tg5
MDX4-4210	WP	T0	347	434	484	537	563	635	367	432	458	481	533
		T1	358	412	438	463	568	637	372	407	436	497	529
		T2	351	439	482	574	651	*	369	425	447	466	543
	IP	T0	354	*	441	502	585	638	378	438	*	489	553
		T1	354	431	448	490	574	651	372	413	430	497	574
		T2	354	435	471	575	615	661	381	433	452	470	570
	IEP	T0	361	427	446	547	576	645	374	422	451	491	540
		T1	358	433	473	564	579	628	379	431	460	498	529
		T2	353	446	474	576	603	*	377	426	456	473	553
Silpuran 2420	WP	T0	330	413	436	486	534	649	355	409	434	485	508
		T1	336	426	445	491	524	638	360	416	441	466	494
		T2	329	423	438	474	534	642	354	422	439	475	504
	IP	T0	332	417	441	483	523	611	360	398	413	440	455
		T1	336	427	452	504	536	625	360	413	451	488	525
		T2	335	416	441	494	549	643	362	413	441	491	522
	IEP	T0	335	420	459	501	548	654	358	388	454	492	518
		T1	334	413	447	477	548	637	359	411	445	485	522
		T2	335	415	442	495	544	637	360	417	443	494	516

*Absent

Discussion

The null hypothesis was rejected. For both silicones, the IP promoted greater color stability over time, suggesting that pigment incorporation was favorable. In general, there was color change for the WP group with T0.15%, and for the IEP group at T1 and T2[4,9,10,12,19]. According to the NBS scale, WP and IEP groups of both silicones showed clinically unacceptable color changes when exposed to T0.15% at T1 and T2, regardless of the hygiene protocol. The IP groups of both silicones showed values within the clinically acceptable range, regardless of time and hygiene protocol. The results are promising, as 2% hydrogen peroxide and 0.15% triclosan has good antimicrobial action[24,27] and are easy to access. Color degradation for the colorless group indicates that color instability is inherent to the material[7,8]. On the other hand, the color degradation of the IEP specimens may be related to substances absorption, degradation reactions such as hydrolysis, and thermal and humidity changes of the extrinsic pigment[10]

as well as modification or removal of the external pigment layer caused by hygiene protocols over the months.

Due to average hardness values, MDX4-4210 can be classified as soft silicone and Silpuran 2420 as extra soft[33]. The literature is controversial regarding the ideal Shore A hardness of silicone for facial prostheses. For some authors, hardness values should be between 25 and 35 units[1,2]. However, the choice of a material with higher or lower hardness depends on the clinical condition of the organ to be rehabilitated and its tissue characteristics[1,7,18,19]. The increase in the hardness can promote a negative effect on patient acceptance due to misfit of the edges[1]. The maximum change in hardness of the materials studied was 3.78 points, and considering the initial values, this change would hardly affect the quality of the prosthesis. Both silicones with IEP showed shore A hardness changes smaller than or equal to the IP group, corroborating with the literature[19]. However, other studies show that extrinsic pigmentation promoted a greater change in hardness when compared to intrinsic pigmentation and colorless silicone[5,9]. Although the authors justify this result as consequence of double polymerization, the use of the make-up powder and not a pigment suitable for silicone can had influenced. The HP2% and T0.15% promoted similar or lower hardness values than control group, independent of pigmentation. This can be considered a good result.

It is important to mention that triclosan was banned from soap products (liquid, gel, foam, bar) by the U.S. Food and Drug Administration in 2016 and from all human hygiene biocidal products by the European Union in 2017, due to the detection of triclosan in skin, urine, and plasma after mucosal contact, raising concerns about its potential toxicity and harmful effects on both the population and the environment[34]. However, there is a lack of mechanism-based human studies in terms of both number and scope[35]. Most cohort and *in vitro* studies have predominantly assessed the toxic effects of triclosan exposure without an in-depth analysis of its specific mechanisms of action and cellular targets. The literature emphasized the need for further investigation into the biological effects of triclosan exposure at the metabolic level *in vivo*. Furthermore, evaluation of the DNA damage, cytokinetic defects, proliferative potential, and cell death caused by mouthrinses (chlorhexidine, triclosan, and essential oils in ethanolic solution) didn't show evidence of genotoxic effects from the analyzed mouthrinses[36]. In this study, triclosan was tested at a concentration of 0.15% and was also recommended for denture immersion, as this method avoids direct mucosal contact, minimizing the risk of toxicity.

Pigmentation and time might influence silicone surface roughness. The smallest variation occurred with colorless silicones[7,19], however, this group showed a greater change in roughness in T2 when compared with T1[7,14,19,20]. In regard to hygiene solutions, the literature presents different options[2,6,13,15,19,21,26], but the majority of the studies evaluated antimicrobial action without analyzing the effect on material properties, specifically surface roughness, which is related to biofilm adhesion[19,20]. Our results indicate that there was an increase in roughness after hygiene protocols and that the use of neutral soap resulted in less variation for both materials in the colorless form[20].

Infrared vibrational spectrometry showed that the vibrational peaks remained preserved for both materials. Similar chemical groups were identified among the samples, regardless of pigmentation and time, indicating that the addition of pigments did not interfere in the polymerization process and composition of the samples[4]. Thermal analysis corroborates with the vibrational spectroscopy, which indicates that the variation factors applied to the materials did not result in structural alterations. Both materials started the continuous decomposition process at around 300°C, generating a loss of 35% to 57% mass of silicon dioxide (SiO_2) and silicon carbide (SiC) residue. This can be explained by the difference in proportions in quantity of carbon and oxygen in the composition of the two types of silicone. It is also noted that the initial mass loss is not perfectly monotonic, and there is the presence of minor oxidative processes, suggesting that presence of occluded oxygen in the specimens in bubble form. The mass loss in an anoxic atmosphere was consistent with the release of siloxane hydrolysis products with the formation of water and carbon dioxide. The main exothermic peak, around 500ºC, corresponded to the degradation processes of the two types of silicone and, when comparing all the specimens, the variation in temperatures was very small, with the values of MDX4-4210 around 10% higher compared to those of Silpuran 2420, suggesting similarity between them. However, it should be noted that the temperatures required for the degradation of both types of silicone were much higher than those experienced in everyday life. This variation in values does not seem to be related to the presence of pigment, suggesting that there was integration of the pigments added to the silicone.

One limitation of this study is the artificial aging process used, which differs from natural aging. Due to time constraints, a 12-month consecutive material analysis was not feasible, so accelerated aging was necessary to simulate long-term conditions. Additionally, the absence of previous studies on Silpuran 2420 limits the ability to directly compare the results. However, this also highlights the innovative nature of the findings. Future studies could be analyzing other properties of the materials, such as tensile strength, tear strength, and sorption and solubility. Furthermore, surface analysis using confocal microscopy, extended aging times, and studies evaluating the behavior of Silpuran 2420 regarding biofilm adhesion and antimicrobial response under various hygiene protocols are necessary. Moreover, analyses to identify whether residues of the solutions remain on the prostheses after washing are important to eliminate possible adverse effects.

This study demonstrated that intrinsic+extrinsec pigmentation impacted color stability over time in both MDX4-4210 and Silpuran 2420, with notable variations, particularly when exposed to HP2% and T0.15%. Changes in hardness did not compromise the quality of the materials. Chemical and thermal comportment was not affected by pigmentation or hygiene protocols. IEP silicones showed lower variation in surface roughness, suggesting greater resistance to surface wear, but still exhibited increases in roughness with prolonged exposure to hygiene protocols.

Conflict of interests

The authors affirm that they have no conflicts of interest to declare.

Data Availability

Datasets related to this article can be found at: https://www.teses.usp.br/teses/disponiveis/58/58131/tde-23102023-145726/pt-br.php. Hosted at: https://www.teses.usp.br/

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Author Contribution

Carla Maria de Almeida Prado Magdalena: Data curation, Investigation, Methodology, Writing – original draft. **Arthur Augusto Martins e Silva**: Writing – original draft, Writing – review & editing. **Priscila Lai Liu**: Methodology, Writing – original draft. **Ana Paula Macedo**: Data curation, Formal Analysis, Methodology, Writing – original draft. **Grégoire Jean Francois Demets**: Formal Analysis, Methodology, Writing – original draft. **Cláudia Helena Silva-Lovato**: Conceptualization, Data curation, Formal Analysis, Funding acquisition, Methodology, Project administration, Resources, Supervision, Validation, Writing – original draft, Writing – review & editing. All authors revised and approved the final version of the manuscript.

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