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RESEARCH AND EDUCATION

Effect of nonthermal plasma on the properties of a resinous liner submitted to aging

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ABSTRACT

Statement of problem. The properties, such as softness and viscoelasticity, of a resinous reliner can deteriorate and extrinsic elements can become incorporated, making surface protection of the reliner material essential.

Purpose. The purpose of this in vitro study was to evaluate the effect of low temperature plasma on Coe-Soft resinous reliner, submitted to aging in artificial saliva for up to 180 days. Sorption, solubility, Shore A hardness, surface energy, and topographic characteristics were analyzed by scanning electronic microscopy (SEM) and energy-dispersive spectroscopy (EDS).

Material and methods. Forty-four specimens were fabricated and distributed in 2 groups: nonplasma reliner (control group) and reliner with plasma (plasma group). The plasma was applied with a mixture of 70% hexamethyldisiloxane, 20% O, and 10% Ar. Total work pressure was maintained at a constant 20 Pa for 30 minutes of deposition. The specimens were analyzed before and after aging in an incubator with immersion in artificial saliva for 30, 90, and 180 days. The quantitative data were submitted to 2-way ANOVA and the Tukey test (α=.05), while qualitative data were compared visually.

Results. The control group presented lower Shore A hardness values only in the initial period, and surface energy increased with aging for both groups until 90 days. Greater sorption percentage values were encountered at 180 days in the plasma group. Greater solubility values were encountered in the control group in all periods.

Conclusions. Plasma is an option for the protection of the material studied because the deposited film remained on the surface of the reliner material after aging. (J Prosthet Dent 2018;119:397-403)

Patients with partial or complete edentulism require relining of their prostheses for improved adaptation, retention, and comfort, principally in situations where there is severe bone resorption.[1] In addition, silicone or acrylic resin soft lining materials can be used to treat trauma to the oral mucosa and in the obturation of oroantral communication in patients with oral defects.[2-7]

The softness and viscoelasticity of acrylic resin reliner materials are important for clinical application.[6,8] These properties help to absorb masticatory forces, reducing and distributing the forces over the supporting bone.[9,10] The reliner materials also require dimensional stability, biocompatibility, adequate tear strength, and adhesion to the prosthetic material.[10] However, over time, some properties deteriorate and extrinsic elements become incorporated.[10-12] Use in the oral cavity may harden the material[6,13-15] and increase the modulus of elasticity, one of the principal physical characteristics of soft relining materials.[10]

Saliva affects the properties of the soft relining materials.[15] Therefore, in vitro studies that attempt to reproduce the conditions of the oral environment and

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Clinical Implications

The application of low-temperature plasma may be an effective way of increasing the longevity of relined partial or complete removable dental prostheses by protecting the surface of the material from degradation in the oral cavity.

study the effects on physical properties (such as viscoelasticity and hardness) due to the leaching out of plasticizers during clinical use[8] should include immersion in artificial saliva.[15,16]

Alteration of physical properties can influence patient comfort by reducing the absorption of masticatory stress and decreasing protection from the inflammation and trauma promoted by reliner softness.[8,15] Therefore, techniques to increase the clinical life of prostheses,[1,5,15] such as the application of nonthermal plasma (NTP) to provide surface protection, are valuable.

NTP is an environment where ionized, reactive, and neutral particles are generated from gas, vapor, or a mixture of both. It reacts with, cleans, or conditions the material surface, depending on its composition, and promotes adhesion and the formation of a thin protective layer on substrate surfaces.[17-19] Improving reliner material barrier properties through surface layer alteration while preserving resin softness is important.

Plasma-deposited organosilicon films present low wettability and elastomeric material structure properties[20] attributed to the chemical composition and molecular structure of the coatings. Adhesion of these coatings, used as barriers against the permeation of water vapor and gases (O_2, N_2) for organic electronic[21] and food packaging[22,23] polymeric devices, is reported to be satisfactory. On the basis of these findings, organosilicon film deposition may be a useful approach to increasing the resistance of acrylic reliner to saliva while preserving the volume properties of the reliner material.[24]

Therefore, the objective of this in vitro study was to evaluate the effect of NTP on the sorption, solubility, Shore A hardness, surface energy, and topographic properties of the tested resinous reliner, submitted to aging for up to 180 days in artificial saliva. The null hypothesis was that NTP would not influence the sorption, solubility, Shore A hardness, surface energy, or properties of the resinous reliner.

MATERIAL AND METHODS

Forty-four round specimens of Coe-Soft (GC America Inc) resinous reliner material were fabricated by using 11 g of powder per 8 mL of liquid, according to the manufacturer's recommendations (http://www.gcamerica.com/products/operatory/coe-soft/). The specimens were distributed in 2 groups: nonplasma reliner (control group) and reliner with NTP (plasma group). Twenty specimens were used for the sorption and solubility test (45 mm diameter×1 mm thick), 20 for Shore A hardness and surface energy tests (30 mm diameter×3 mm thick), and 4 for analysis of surface properties (30 mm diameter×3 mm thick).

Plasma-treated specimens were treated with NTP by using a glass chamber in a reactor developed by the Plasma Technologies Laboratory (LaPTec, Engineering Faculty/UNESP). Plasma was applied at a base pressure of 2.67 Pa with a mixture of 70% hexamethyldisiloxane, 20% O, and 10% Ar. A power of 150 W at a radiofrequency of 13.56 MHz was applied on the inferior electrode (specimen supports) with the superior electrode grounded. The total work pressure was maintained at a constant 20 Pa for 30 minutes of disposition.

The specimens were immersed in artificial saliva (KCl [0.4 gL^{-1}], NaCl [0.4 gL^{-1}], $CaCl_2$-$2H_2O$ [0.906 gL^{-1}], NaH_2PO_4-$2H_2O$ [0.690 gL^{-1}], Na_2S-$9H_2O$ [0.005 gL^{-1}]) and maintained in an incubator at 37°C for artificial aging. They were identified and stored individually, and the saliva was changed daily.[25,26] The total aging period was 180 days. Sorption, solubility, Shore A hardness, and surface energy were analyzed in the initial period and after 30, 90, and 180 days. Twenty specimens (n=10)[25] were submitted to the desiccation test for sorption and solubility analysis by following specification #12 of the American Dental Association (ADA).[27] The specimens were stored in an incubator (Odontobras) in a vacuum-sealed dryer containing silica gel maintained at 37 ±2°C and were weighed daily on a precision digital scale (BEL Equipamentos Analitico) until the initial mass (W1) was acquired.

A new weight was measured (W2) and the specimens desiccated before the aging periods. Subsequently, the specimens were reweighed (W3) and the sorption and solubility degrees were calculated with the following formulas:

$$\text{Sorption percentage} = (W2 - W3)/W1 \times 100$$

$$\text{Solubility percentage} = (W1 - W3) \times 100.$$

The Shore A hardness test was performed on 20 specimens (n=10)[25] with the Shore A digital durometer (GSD 709; Teclock), according to the American Society for Testing and Materials D2240 specification.[28] The definitive hardness value was based on the penetration depth of the spring-loaded indenter,[25] and after each specimen had been measured 3 times, a constant load of 10 N was acquired as the mean.

Twenty specimens (n=10)[29] were used for surface energy analysis by using a goniometer and the sessile drop technique. Drops of 2 liquids with different polarities (deionized water and diiodomethane) were deposited on each specimen placed in the goniometer

Table 1. Two-way ANOVA of Shore A hardness of resinous reliner for analyzed groups

Characteristic	SS	*df*	MS	F	*P*
Treatment	31.626	1	31.626	9.628	.006
Between specimens	59.127	18	3.285		
Period	61.759	3	20.586	6.721	.001
Treatment×Period	20.937	3	6.979	2.279	.090
Intraspecimen	165.397	54	3.063		

SS, sum of squares; MS, mean squares. $P<.05$, significant statistical difference.

Table 3. Two-way (ANOVA) of surface energy of resinous reliner for analyzed groups

Characteristic	SS	*df*	MS	F	*P*
Treatment	.072	1	0.072	0.005	.947
Between specimens	280.004	18	15.556		
Period	3062.883	3	1020.961	60.072	<.001
Treatment×Period	85.960	3	28.653	1.686	.181
Intraspecimen	917.770	54	16.996		

SS, sum of squares; MS, mean squares. $P<.05$, significant statistical difference.

Table 5. Two-way repeated-measures ANOVA of sorption of resinous reliner for groups analyzed

Characteristic	SS	*df*	MS	F	*P*
Treatment	1.596	1	1.596	28.524	<.001
Between specimens	1.007	18	0.056		
Period	23.641	2	11.821	292.547	<.001
Treatment×Period	3.349	2	1.674	41.442	<.001
Intraspecimen	1.455	36	0.040		

SS, sum of squares; MS, mean squares. $P<.05$, significant statistical difference.

(Ramé-Hart 100-00; Ramé-Hart Instrument Co), and 10 readings per specimen were made by the same person (E.V.F.S.) in a temperature-controlled environment at 23°C, in accordance with American National Standards Institute/ADA #6872.[29]

The image of each drop was recorded and analyzed with software (DROPimage Standard; Ramé-Hart Instrument Co), and the surface energy was calculated in accordance with the Owens-Wendt-Rabel-Kaelble (OWRK) method, based on the contact angle formed by 2 tested liquids with different polarities.[29] Surface properties were evaluated with scanning electronic microscopy (SEM) (JSM 6010LA; JEOL Ltd) by using 4 specimens (n=2) for each evaluation. Images were registered for each test at ×10 000 magnification. Elemental chemical composition characterization of the specimen surface was performed in volumes of 1 μm^3 with energy-dispersive spectroscopy (EDS). Analyses were performed in the initial and definitive period of the study.

The quantitative data for sorption, solubility, Shore A hardness, and surface energy were submitted to 2-way repeated-measures ANOVA and the Tukey test (α=.05). The SEM and EDS qualitative results were compared visually.

Table 2. Shore A hardness (mean ±SD) of resinous reliner treated or nontreated with NTP before and after periods of aging

	Treatment	
Period	**Nonplasma**	**NTP**
Initial	26.13 ±1.43^{ABb}	28.60 ±2.05^{Aa}
30 d	26.80 ±1.72^{Aa}	27.52 ±0.89^{Aa}
90 d	25.47 ±1.33^{Ba}	25.34 ±1.26^{Ba}
180 d	26.67 ±2.67^{ABa}	28.63 ±2.12^{Aa}

NTP, nonthermal plasma. Means followed by same uppercase letter in column and lowercase letter in row did not differ ($P>.05$) by Tukey test.

Table 4. Surface energy (mean ±SD) of resinous reliner treated or nontreated with NTP before and after periods of aging

	Treatment	
Period	**Nonplasma**	**NTP**
Initial	44.52 ±3.81^{C}	42.29 ±4.73^{C}
30 d	55.37 ±4.96^{B}	58.80 ±3.73^{A}
90 d	59.79 ±3.23^{A}	59.35 ±3.65^{A}
180 d	52.14 ±2.99^{B}	51.61 ±4.98^{B}

NTP, nonthermal plasma. Means followed by same uppercase letter in column did not differ ($P>.05$) by Tukey test.

Table 6. Sorption (mean percentage values ±SD) for resinous reliner treated or nontreated with NTP after aging periods

	Treatment	
Period	**Nonplasma**	**NTP**
30 d	1.23 ±0.06^{Ca}	1.24 ±0.11^{Ba}
90 d	2.77 ±0.23^{Aa}	2.74 ±0.21^{Aa}
180 d	1.72 ±0.38^{Bb}	2.71 ±0.14^{Aa}

NTP, nonthermal plasma. Means followed by same uppercase letter in column and lowercase letter in row did not differ ($P>.05$) by Tukey test.

RESULTS

Treatment type (with or without plasma) (P=.006) and evaluation period (initial, 30, 90, and 180 days of aging) (P=.001) were verified as having influenced the relining material Shore A hardness (Table 1). Shore A hardness value was lower for the nonplasma group in the initial period compared with the plasma group (P=.006) and lower in nonplasma (P=.039) and NTP (P<.002) groups in the 90-day aging period compared with the other periods (Table 2).

The reliner material surface energy was influenced by the aging period (P<.001) (Table 3), and surface energy increased until 90 days of aging for nonplasma (P<.006) and NTP (P<.001) groups. In addition, surface energy was reduced in nonplasma (P<.001) and NTP (P<.001) groups at 180 days of aging (Table 4).

A significant interaction between treatment type and analysis period (P<.001) was found (Table 5). Sorption percentage values increased after 90 and 180 days of aging in both groups compared with the initial time

Table 7. Two-factor repeated-measures ANOVA of solubility of resinous reliner for analyzed groups

Characteristic	SS	*df*	MS	F	*P*
Treatment	1.520	1	1.520	8.574	.009
Between specimens	3.191	18	0.177		
Period	110.101	2	55.051	2675.582	<.001
Treatment×Period	.095	2	0.047	2.305	.114
Intraspecimen	.741	36	0.021		

SS, sum of squares; MS, mean squares. *P*<.05, significant statistical difference.

Table 8. Solubility (mean percentage values ±SD) of resinous liner treated or nontreated with NTP before and after aging periods

	Treatment	
Period	**Nonplasma**	**NTP**
30 d	1.99 ±0.13^{Ca}	1.78 ±0.12^{Cb}
90 d	3.88 ±0.23^{Ba}	3.55 ±0.26^{Bb}
180 d	5.40 ±0.29^{Aa}	4.99 ±0.45^{Ab}

NTP, nonthermal plasma. Means followed by same uppercase letter in column and lowercase letter in row did not differ (*P*>.05) by Tukey test.

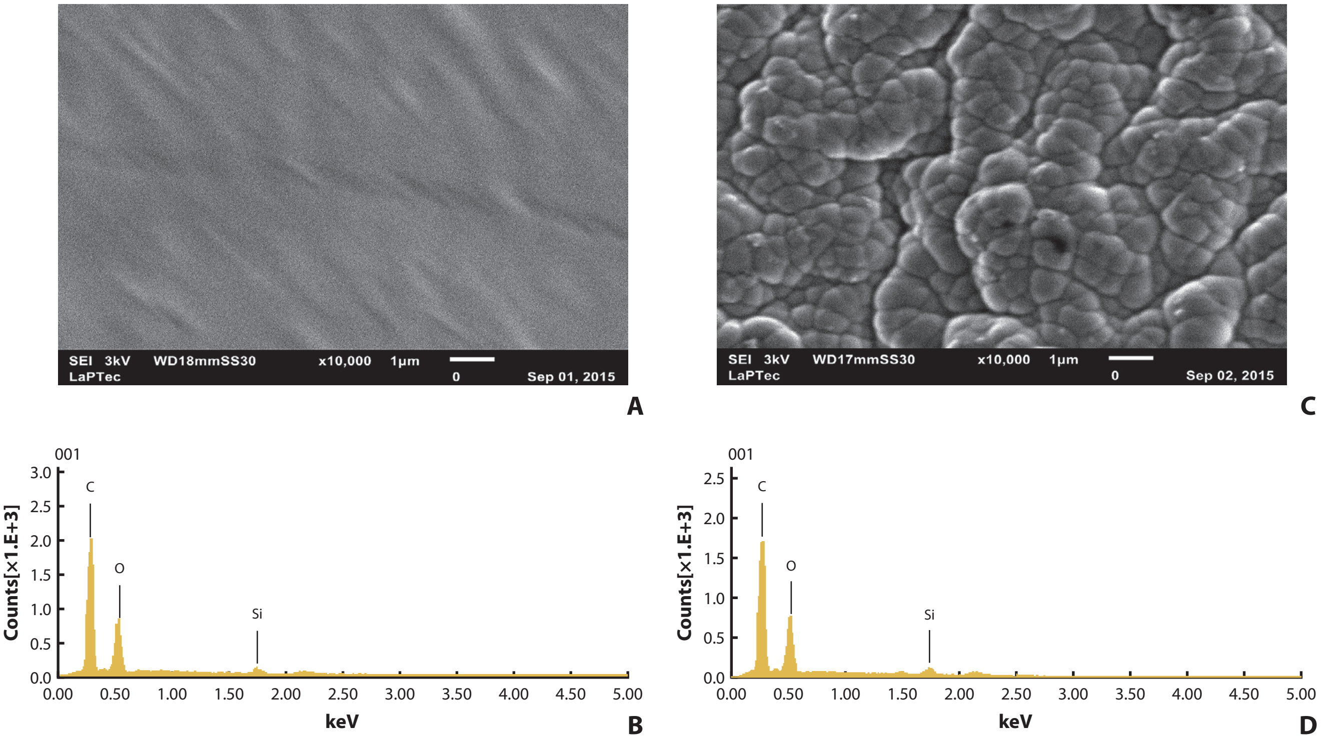

Figure 1. Initial SEM (original magnification, ×10 000) and EDS images without and with NTP application. A, Initial SEM images without NTP application. B, Initial EDS images without NTP application. C, Initial SEM images with NTP application. D, Initial EDS images with NTP application. SEM, scanning electron microscopy; EDS, energy-dispersive spectroscopy; NTP, nonthermal plasma.

period (Table 6). Treatment type (*P*=.009) and analysis period (*P*<.001) were shown to influence reliner material solubility (Table 7). Statistically greater solubility values were encountered for the nonplasma group in each analysis period compared with the plasma group. In addition, solubility values increased with aging in both groups (*P*<.001) (Table 8).

The SEM analysis identified differences between the groups in the initial (Fig. 1A, C) and 180-day aging periods (Fig. 2A, C). A smooth and homogenous surface was observed in the specimens without NTP, indicating the disposition of plasma films (Fig. 1C). Alterations were observed after 180 days of aging, suggesting surface degradation in groups with and without NTP compared with the initial period (Fig. 2A, C). The presence of C, O, and Si was observed in both groups in the initial period (Fig. 1B, D) with EDS analysis, and C and O were observed after aging (Fig. 2B, D).

DISCUSSION

The null hypothesis was rejected because Shore A hardness, sorption, and solubility were influenced by NTP; however, the surface energy results were not different. The Shore A hardness values were increased by NTP in the initial period. In the 180-day aging period, the sorption was greater in the plasma group, while solubility was lower in all analysis periods.

Only the initial results of the Shore A hardness analysis were influenced by the NTP application because lower Shore A hardness was presented by the nonplasma group in this period. However, no difference was shown between

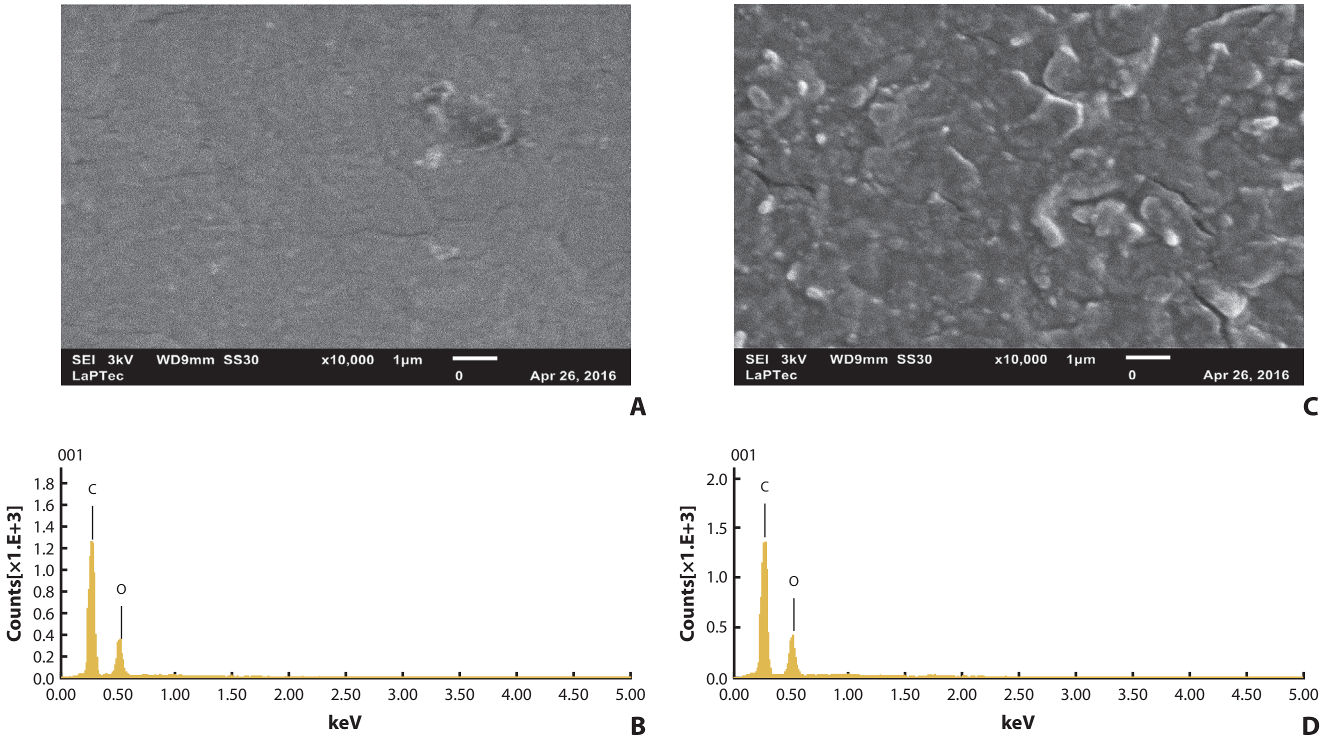

Figure 2. SEM (original magnification, ×10 000) and EDS images after 180 days without and with NTP application. A, SEM image after 180 days without NTP application. B, EDS images after 180 days without NTP application. C, SEM image after 180 days with NTP application. D, EDS images after 180 days with NTP application. SEM, scanning electron microscopy; EDS, energy-dispersive spectroscopy; NTP, nonthermal plasma.

the groups during aging. An alteration to surface hardness, and consequently viscoelasticity, is prejudicial to the clinical success of a soft reliner material.[6] The nonplasma group values were stable during all analysis periods; however, lower Shore A hardness was verified after 90 days of aging for the plasma group. According to the Shore A hardness scale, which varies from 0 to 100, the tested material was classified as soft because results between 25 to 29 Shore A hardness units were produced, corroborating the results reported in published studies.[5,8,12,15,25]

Surface energy variation of material is related to microorganism adhesion capacity because bacterial adhesion increases with the increase of energy.[19] In the present in vitro study, surface energy was influenced by the proposed aging period. Independent of the group, a progressive increase in energy values was verified with aging, being greater in the 90-day period, a fact that contraindicates the prolonged use of the material. However, a reduction was seen at 180 days in both groups. Yet on the basis of the results produced, the surface energy analysis results were not influenced by the application of the plasma.

Prolonged immersion of reliner materials in a liquid environment causes deterioration of their properties, principally the mechanical properties.[12] Sorption results were influenced by the treatment performed and the different evaluation periods. The plasma-treated group deteriorated significantly after 180 days of aging, and independently of the group evaluated, sorption results were greatest in the 90-day period. This factor can negatively influence material strength because of water absorption or interaction with other organic liquids present in the diet.[12]

Sorption and solubility are the most frequently encountered problems because of the humid environments of saliva, cleaning solutions, and storage in water.[4] Like sorption, solubility was influenced by the treatment performed and periods evaluated. However, contrary to the sorption results, better solubility results were found for the plasma-treated group compared with the nonplasma group. In the 180-day period, more unsatisfactory results were encountered independent of the group evaluated. The manufacturer recommends the use of resinous liner, under normal conditions, for 90 days (http://www.gcamerica.com/products/operatory/coe-soft/).

After a long storage period in saliva, the continuance of films deposited on the surface of the acrylic resin (methacrylate) specimens with plasma was verified. This demonstrates that thin films produced by NTP application can be maintained. However, surface deterioration was observed after 180 days of aging, indicating a structural alteration. Studies[10,11] have demonstrated that aging causes material to lose physical properties, principally through leaching of plasticizing substances, which can lead to greater surface roughness, promoting bacterial colonization,[14] patient irritation,[10] and greater material rigidness.[10,11]

The resultant layer promoted by NTP application is an organosilicon film, which according to the EDS results was composed of 74% C, 5% Si, and 21% O, with a stoichiometry closer to that of conventional polydimethylsiloxane, which has 50% C, 25% Si, and 25% O, compared with silica, which is 33% Si and 66% O. This similarity with conventional silicone is why these films are termed “silicone-like” materials. The slight increase in the Shore A hardness of the organosilicon film-covered surface was attributed to the formation of a more deformation resistant cross-linked structure compared with the resin relining material. These coating properties provide better mechanical and barrier properties to the surface while ensuring patient comfort.

Thus, given the results, the application of plasma is suggested to promote the surface protection of a methacrylate-based reliner material such as Coe-Soft. The initial Shore A hardness was greater in the plasma group; however, it was not sustained with aging. In addition, solubility was lower in the NTP group in all periods compared with the nontreated group.

NTP was shown to be effective in the superficial protection of the reliner material. Clinically, retention of the material properties results in greater patient comfort because of the protection from inflammation and trauma of the underlying denture area. Compliance and dimensional stability of the material, which are related to sorption and solubility properties, affect the clinical success of reliner treatment.[15,16]

On the basis of the limitations of this in vitro study, additional studies are suggested to evaluate the effects of prolonged immersion of the tested NTP-treated material when exposed to different possible staining solutions. The evaluation of tear strength and the effect of thermocycling on the maintenance of the physical properties of the tested material is also suggested. Trials aiming to assess the clinical effects of NTP on reliners should be performed because beneficial results were found in this in vitro study. In addition, the evaluation and use of new NTP compositions should be evaluated.

CONCLUSIONS

On the basis of the findings of this in vitro study, the following conclusions were drawn:

1. Nonthermal plasma was verified as a viable protection option of the material studied because the deposited film remained on the reliner surface after aging.
2. Initial Shore A hardness was greater in the plasma-treated group but was not sustained with aging.
3. Sorption was greater after 180 days of aging; however, solubility was lower in the plasma-treated group in all time periods compared with the nontreated group.

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Noteworthy Abstracts of the Current Literature

Response to antiseptic agents of periodontal pathogens in in vitro biofilms on titanium and zirconium surfaces

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Objective. The aim of this study was to develop in vitro biofilms on SLA titanium (Ti-SLA) and zirconium oxide (ZrO2) surfaces and to evaluate the effect of antiseptic agents on the number of putative periodontal pathogenic species.

Methods. An in vitro biofilm model was developed on sterile discs of Ti-SLA and ZrO2. Three antiseptic agents [chlorhexidine and cetyl-pyridinium-chloride (CHX/CPC), essential oils (EEOOs) and cetyl-peridinium-chloride (CPC)] were applied to 72-h biofilms, immersing discs during 1min in the antiseptic solution, either with or without mechanical disruption. Viable bacteria [colony forming units (CFU/mL)] were measured by quantitative polymerase chain reaction (qPCR) combined with propidium monoazide. A generalized lineal model was constructed to determine the effect of the agents on the viable bacterial counts of Aggregatibacter actinomycetemcomitans, Porphyromonas gingivalis and Fusobacterium nucleatum on each surface.

Results. The exposure to each antiseptic solution resulted in a statistically significant reductions in the number of viable target species included in the in vitro multi-species biofilm, on both Ti-SLA and ZrO2 ($p<0.001$) which was of up to 2 orders for A. actinomycetemcomitans, for P. gingivalis 2 orders on Ti-SLA and up to 3 orders on ZrO2, and, for F. nucleatum up to 4 orders. No significant differences were found in counts of the tested bacteria between in vitro biofilms formed on both Ti-SLA and ZrO2, after topically exposure to the antimicrobial agents whether the application was purely chemical or combined with mechanical disruption.

Significance. A. actinomycetemcomitans, P. gingivalis and F. nucleatum responded similarly to their exposure to antiseptics when grown in multispecies biofilms on titanium and zirconium surfaces, in spite of the described structural differences between these bacterial communities.

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