

RESEARCH AND EDUCATION

Aging effect of atmospheric air on lithium disilicate ceramic after nonthermal plasma treatment



Daniela Micheline dos Santos, DDS, DMD, PhD,^a Emily Vivianne Freitas da Silva, DDS,^b Aljomar José Vechiato-Filho, DDS,^c Paulo Francisco Cesar, DDS, DMD, PhD,^d Elidiane Cipriano Rangel, PhD,^e Nilson Cristino da Cruz, PhD,^f and Marcelo Coelho Goiato, DDS, DMD, PhD^g

Dental ceramics have high biocompatibility and satisfactory mechanical and esthetic properties. They are popular in practice because of their good clinical performance.¹⁻⁶ However, achieving a stable and lasting adhesion between the restoration surface, resin cement, and tooth structure is a major clinical challenge.^{7,8}

Hydrofluoric (HF) acid etching is recommended for some dental ceramics such as lithium disilicate ceramic to selectively dissolve their glassy phase.⁹⁻¹² Surface coating with silane improves the bond strength between the ceramic surface silica and the luting cement.^{4,13,14} The ceramic surface modification promoted by HF acid etching increases the ceramic surface roughness and the mechanical imbrication with the resin cement. However, the surface defects created by this treatment may negatively affect the mechanical properties of the etched ceramic.^{9,15-17}

ABSTRACT

Statement of problem. Nonthermal plasma (NTP) treatment is an alternative technique for promoting the adhesion of resin cement to lithium disilicate ceramic. However, no study has evaluated whether the surface modifications are affected by atmospheric air aging.

Purpose. The purposes of this in vitro study were to characterize the lithium disilicate surface after depositing an organosilicon film with NTP treatment and to verify the surface energy before and after atmospheric air aging up to 30 days.

Material and methods. Sixteen lithium disilicate disks (10×3 mm) were prepared, and their surfaces were treated with a mixture of hexamethyldisiloxane and argon, followed by oxygen plasma treatment, both for 30 minutes. The lithium disilicate surface was characterized through scanning electron microscopy, energy-dispersive spectroscopy, and atomic force microscopy. Surface energy analysis was performed before (T0) and immediately after NTP treatment (T1) and after atmospheric air aging for 7 (T2), 15 (T3), and 30 days (T4). Data were submitted to analysis of variance followed by the Tukey HSD test ($\alpha=0.05$).

Results. Carbon, oxygen, and silicon were identified on the disilicate surface after NTP treatment, suggesting organosilicon film adhesion. Air aging did not modify the film morphology. At T1, the surface energy was significantly higher compared with other periods, and the water contact angle on the disilicate surface was reduced to 0 degrees. Similar surface energy was observed for T0, T2, T3, and T4.

Conclusions. On the basis of the results of this in vitro study, NTP treatment can promote bonding to lithium disilicate surfaces because of its high surface wettability. However, after air aging, the wettability was not durable. (J Prosthet Dent 2016;115:780-787)

This is due to the larger amount of glass phase surrounding the lithium disilicate crystals.¹⁸

The nonthermal plasma (NTP) technique is an alternative to HF acid etching that can increase the bond

^aProfessor, Department of Dental Materials and Prosthodontics, Aracatuba Dental School, Sao Paulo State University (UNESP), Sao Paulo, Brazil.

^bPostgraduate student, Department of Dental Materials and Prosthodontics, Aracatuba Dental School, Sao Paulo State University (UNESP), Sao Paulo, Brazil.

^cPostgraduate student, Department of Dental Materials and Prosthodontics, Aracatuba Dental School, Sao Paulo State University (UNESP), Sao Paulo, Brazil.

^dProfessor, Department of Biomaterials and Oral Biology, Faculty of Dentistry of Sao Paulo University, Sao Paulo University, Sao Paulo, Brazil.

^eProfessor, Technological Plasma Laboratory (LaPTec), Experimental Campus of Sorocaba, (UNESP), Sao Paulo, Brazil.

^fProfessor, Technological Plasma Laboratory (LaPTec), Experimental Campus of Sorocaba, (UNESP), Sao Paulo, Brazil.

^gProfessor, Department of Dental Materials and Prosthodontics, Aracatuba Dental School, Sao Paulo State University (UNESP), Sao Paulo, Brazil.

Clinical Implications

Nonthermal plasma treatment is an alternative technique for promoting the adhesion of resin cement to lithium disilicate ceramic. Because the surface energy was statistically higher in the period immediately after the organosilicon film deposition, the cementation process should be performed as soon as possible after this procedure.

strength between dental ceramics and resin cements.^{4,13,19} NTP consists of a partially ionized gas containing a mixture of ions, electrons, and neutral species and can be applied to ceramic surfaces.^{4,8,13,20,21} The NTP treatment produces a thin layer that chemically modifies the ceramic surface, increasing the surface hydrophilicity and cement wetting capacity, and forms chemically active sites for bonding with other molecules.^{8,13,22-26}

To facilitate the clinical use of NTP treatment, the maintenance of the hydrophilic surface during atmospheric air aging is essential because there is a lapse between plasma deposition in the dental laboratory and the ceramic cementation process.²² This maintenance is a challenge because inorganic impurities may be deposited on the hydrophilic surface, decreasing its bonding capacity.²⁷⁻²⁹ Therefore, the behavior of these organosilicon hydrophilic films should be evaluated when they are applied on lithium disilicate ceramic surfaces, with the goal of establishing a protocol for their application in the dental clinic.

Until recently, few studies had characterized the surface of lithium disilicate ceramic after the deposition of organosilicon films by NTP treatment.²² Furthermore, no study has reported the effect of atmospheric air aging on these films. The purpose of this study was to characterize the lithium disilicate surface after the deposition of an organosilicon film by NTP treatment and to verify the surface energy before and after atmospheric air aging up to 30 days. The null hypotheses were that the deposition of the organosilicon film would not resist atmospheric air aging and that no significant reduction would be found in the surface energy after the aging periods.

MATERIAL AND METHODS

Sixteen lithium disilicate glass-ceramic specimens (IPS e.max Press; Ivoclar Vivadent AG) were fabricated with the lost-wax technique from autopolymerized acrylic resin disks (Duralay; Polidental) obtained from a metallic circular-shaped pattern measuring 10 mm in diameter and 3 mm in thickness. The materials used in this study are listed in [Table 1](#). The ceramic disks were sequentially polished with metallographic abrasive papers with constant water irrigation and cleaned in ultrasonic baths.²²

After specimen fabrication, the initial analyses of surface characterization and surface energy were performed (T0). Then an organosilicon thin film was deposited by NTP treatment. Immediately after its application (T1) and after atmospheric air aging for 7 (T2), 15 (T3), and 30 days (T4), new analyses were performed. After NTP treatment, the specimens were placed in a Petri dish with a lid and maintained at a controlled temperature (33°C) in the same environment where the initial tests were performed for aging.

For NTP treatment, the specimens were positioned in a cylindrical glass chamber (Technological Plasma Laboratory, LaPTec, Experimental Campus of Sorocaba, Sao Paulo State University) that was evacuated to a background pressure of 12.0 Pa.²⁴ Initially, organosilicon films were deposited from a mixture of 85% hexamethyldisiloxane vapor and 15% argon (Ar) under a pressure of 8.0 Pa, resulting in a constant work pressure of 20.0 Pa. This mixture was then stopped, and a surface treatment with oxygen plasma (O₂) was admitted under a pressure of 1.3 Pa, resulting in a work pressure of 13.5 Pa. The treatments were performed by applying radiofrequency power (13.56 MHz, 50 W) for 30 minutes. The parameters for the NTP technique were determined according to a previous study²² and on the basis of the desired degree of hydrophilicity.

The disilicate surface with an organosilicon film was characterized through scanning electron microscopy (SEM) and atomic force microscopy (AFM). The main chemical elements of the disk surface were identified through energy-dispersive spectroscopy (EDS). For SEM analysis (JSM 610LA; JEOL), 5 specimens were used, 1 for each tested period (T0, T1, T2, T3, and T4). For surface characterization, each specimen was metallized before the reading procedure. Images were recorded at $\times 300$, $\times 10\,000$, and $\times 25\,000$ magnifications. Simultaneously with the SEM analysis, EDS analysis was performed at the order of $1\ \mu\text{m}^3$.

One specimen was submitted to AFM analysis at the 5 cited periods (T0, T1, T2, T3, and T4). The images obtained were transferred to the Gwyddion 2.33 program (Department of Nanometrology, Czech Metrology Institute) to obtain 3-dimensional images. The images were standardized at $5\times 5\ \mu\text{m}$ to enable a visual comparison between the surface topography profiles.

The surface wettability was evaluated through surface energy analysis of the lithium disilicate disks. The surface energy of 10 disks was measured for the tested periods T0, T1, T2, T3, and T4. The sessile drop technique was used in a controlled-temperature room, and 10 measurements were obtained per specimen. Drops of 2 liquids with different polarities, deionized water, which represents polar components and diodomethane, featuring dispersive components, were deposited in a controlled way on the specimen surface and horizontally

Table 1. Material, composition, commercial brand, manufacturer, and batch number of materials used

Material	Composition	Product Name	Manufacturer	Batch No.
Lithium disilicate	SiO ₂ , Li ₂ O, K ₂ O, MgO, ZnO, Al ₂ O ₃ , P ₂ O ₅ and other oxides	IPS e.max Press	Ivoclar Vivadent AG	N24492
Hexamethyldisiloxane (CH ₃) ₃ SiOSi(CH ₃) ₃	C, Si, O, and H	-	Sigma Aldrich	USLY001027
Ar (argon)	Ar	-	White Martins	15116/12
O (oxygen)	O ₂	-	White Martins	07036/12

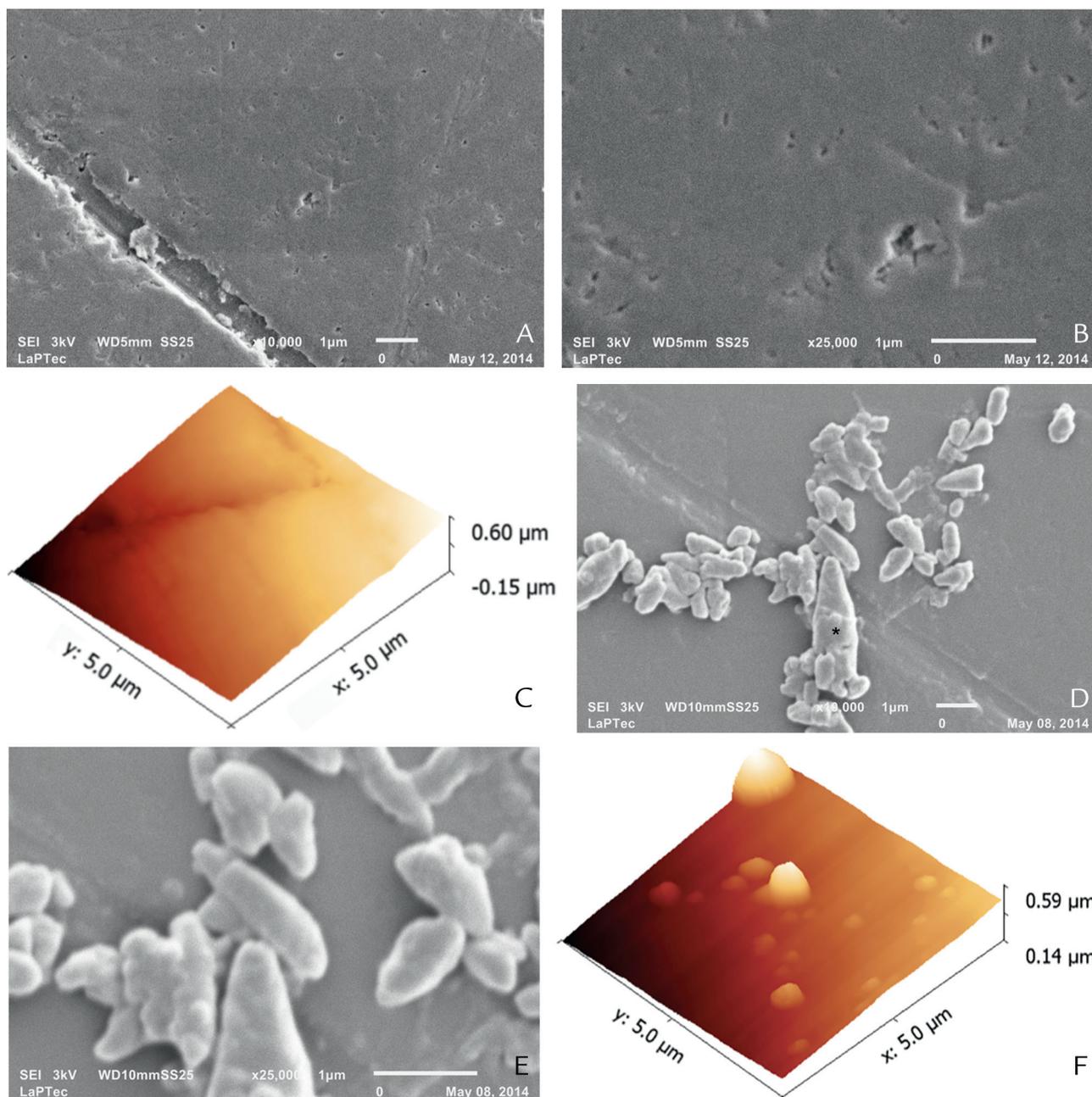


Figure 1. A, Representative SEM image (original magnification, $\times 10\,000$) of lithium disilicate disks before NTP treatment. B, Representative SEM image (original magnification, $\times 25\,000$) of lithium disilicate disks before NTP treatment. C, Representative AFM image of lithium disilicate disks before NTP treatment. D, Representative SEM image (original magnification, $\times 10\,000$) of lithium disilicate disks immediately after NTP treatment. *Areas where organosilicon film is agglomerated in rod-shaped form. E, Representative SEM image (original magnification, $\times 25\,000$) of lithium disilicate disks immediately after NTP treatment. F, Representative AFM image of lithium disilicate disks immediately after NTP treatment. (Continued on next page.)

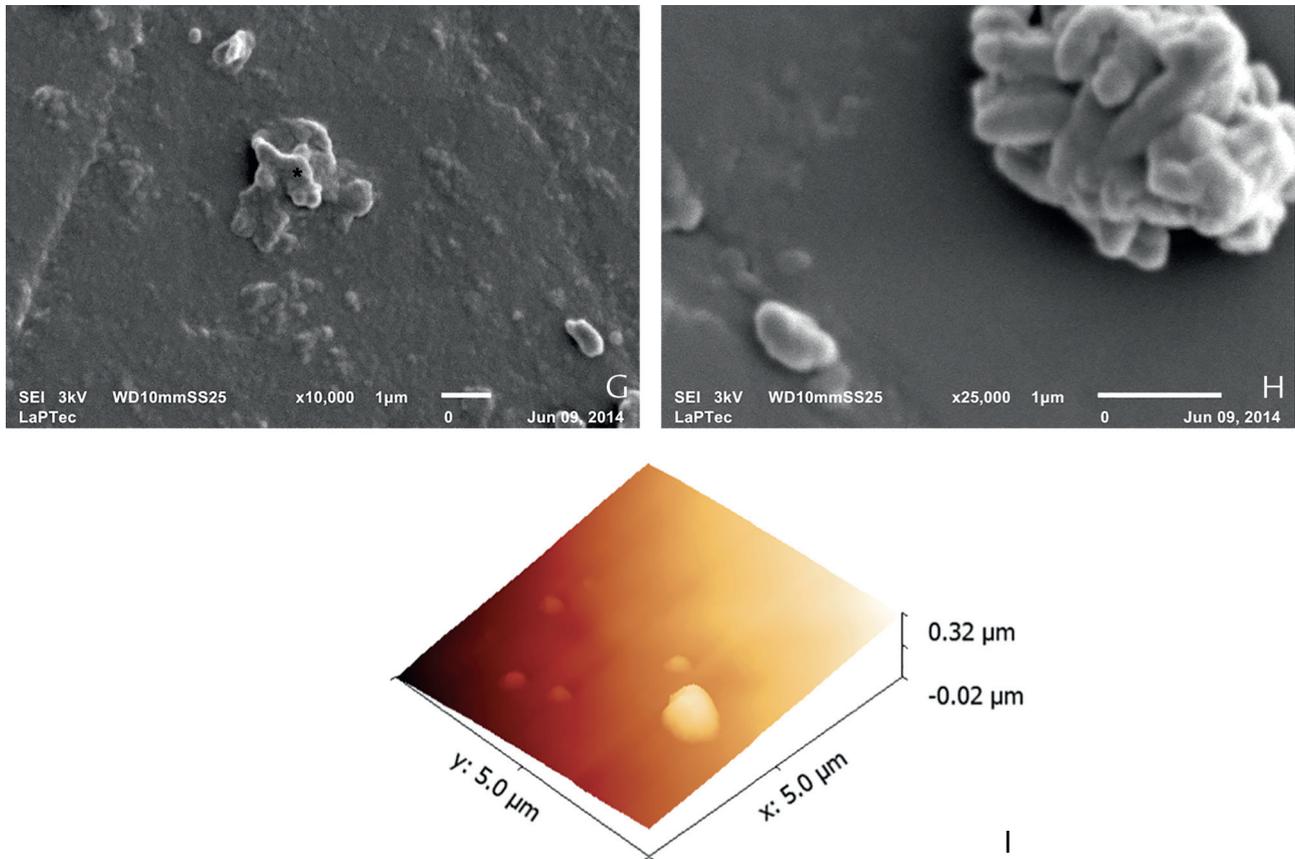


Figure 1. (Continued). G, Representative SEM image (original magnification, $\times 10\,000$) of lithium disilicate disks after 30 days of NTP treatment aging. *Areas where organosilicon film is agglomerated in rod-shaped form. H, Representative SEM image (original magnification, $\times 25\,000$) of lithium disilicate disks after 30 days of NTP treatment aging. I, Representative AFM image of lithium disilicate disks after 30 days of NTP treatment aging.

placed on a goniometer (Ramé-Hart 100-00; Ramé-Hart Instrument Co). Through software (DROPimage Standard; Ramé-Hart Instrument Co), the contact angle formed between the drop of each liquid to the disilicate surface was measured for surface energy analysis. The calculation of surface energy was performed according to the Owens-Wendt-Rabel-Kaelble (OWRK) method.⁷

The quantitative data of surface energy were submitted to 1-way repeated-measures analysis of variance (ANOVA), followed by the Tukey HSD test ($\alpha=.05$). Qualitative data were compared visually among the tested periods (T0, T1, T2, T3, and T4).

RESULTS

Topographic modifications on the lithium disilicate surface were identified through SEM images (Fig. 1) observed at $\times 10\,000$ and $\times 25\,000$ magnifications. The NTP treatment produced a homogeneous film on the ceramic surface. Additionally, areas where the organosilicon film agglomerated in a rod-shaped form were observed, which is observed under certain conditions of deposition of this material.³⁰ Similarly, agglomerated

areas were observed through AFM images (Fig. 1), forming sharp peaks without the presence of irregularities similar to surface deteriorations. These results indicate the presence of an organosilicon film with proper stability adhered to lithium disilicate surfaces.

The main chemical composition of the ceramic surface was identified through EDS analysis (Fig. 2). The initial EDS mapping results (T0) detected the presence of zinc (Zn), aluminum (Al), carbon (C), oxygen (O), and silicon (Si), commonly observed in lithium disilicate ceramic. After NTP treatment (T1, T2, T3, and T4), high quantities of C, O, and Si were observed, suggesting organosilicon film adherence on the ceramic surface and its maintenance during aging in atmospheric air. The initial chemical elements Zn and Al were not observed on the ceramic surface, suggesting that the film thickness is similar or slightly higher than the maximum depth reached by the EDS method.

Regarding the wettability, the time factor significantly affected the surface energy ($df=1$; $F=7834.255$; $P<.001$ by ANOVA). The surface energy was statistically higher in the period immediately after the organosilicon film deposition (T1) compared with periods T0, T2, T3, and T4

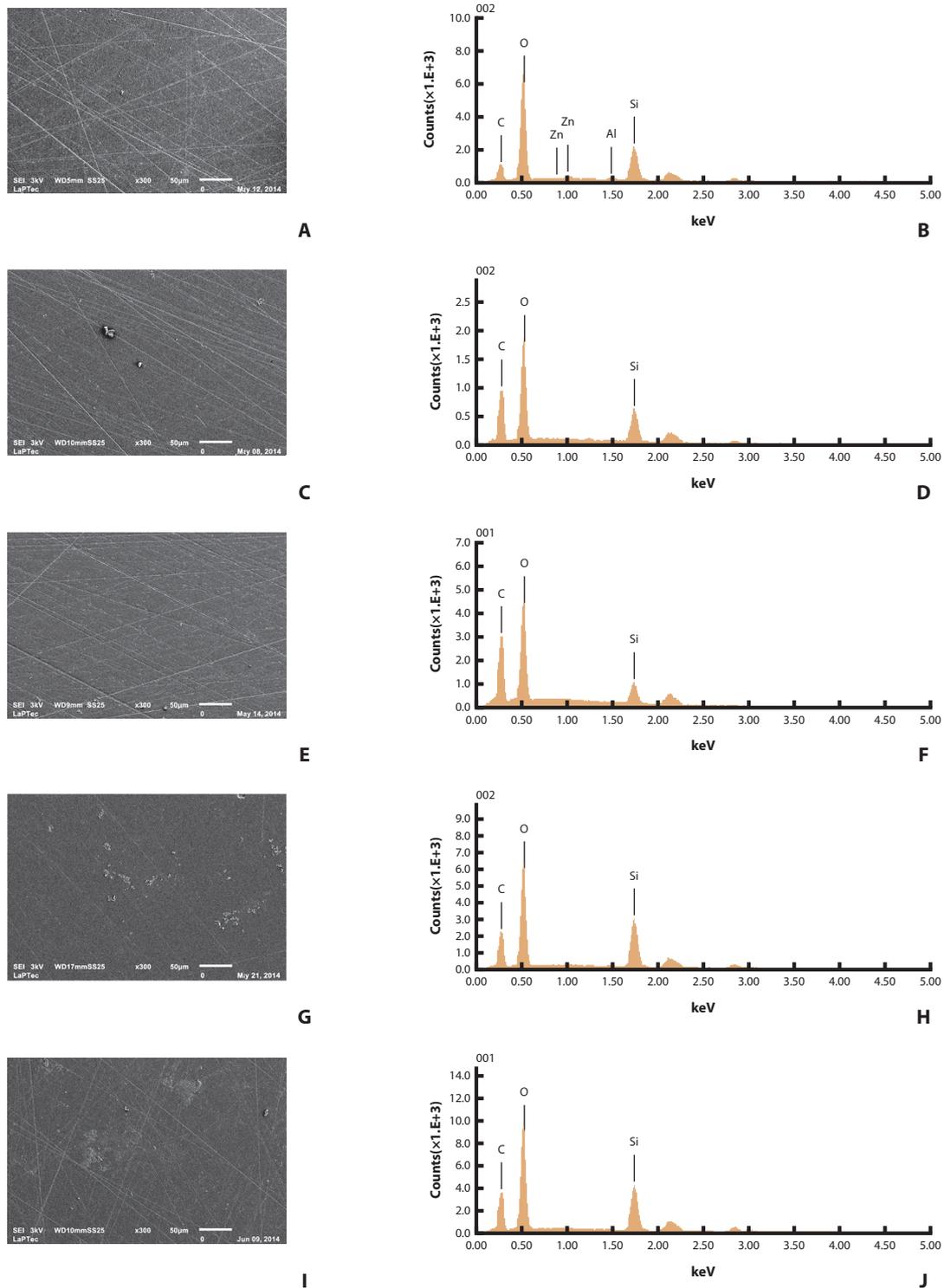


Figure 2. A, Representative SEM image (original magnification, $\times 300$) of lithium disilicate disks before NTP treatment. B, Representative EDS image of lithium disilicate disks before NTP treatment. C, Representative SEM image (original magnification, $\times 300$) of lithium disilicate disks immediately after NTP treatment. D, Representative EDS image of lithium disilicate disks immediately after NTP treatment. E, Representative SEM image (original magnification, $\times 300$) of lithium disilicate disks after 7 days of NTP treatment aging. F, Representative EDS image of lithium disilicate disks after 7 days of NTP treatment aging. G, Representative SEM image (original magnification, $\times 300$) of lithium disilicate disks after 15 days of NTP treatment aging. H, Representative EDS image of lithium disilicate disks after 15 days of NTP treatment aging. I, Representative SEM image (original magnification, $\times 300$) of lithium disilicate disks after 30 days of NTP treatment aging. J, Representative EDS image of lithium disilicate disks after 30 days of NTP treatment aging.

Table 2. Ratios C/Si, O/Si, and O/C determined from proportions of elements identified by EDS spectra of lithium disilicate disks before NTP treatment, immediately after NTP treatment, and after atmospheric air aging for 7, 15, and 30 days

Atmospheric Air Aging Period	C/Si	O/Si	O/C
Initial	0.50	3.25	6.50
Immediate	1.43	2.57	1.80
7 d	3.00	4.50	1.50
15 d	0.75	2.00	2.67
30 d	0.94	2.44	2.60

(Fig. 3). Through representative images of the water contact angles (Fig. 4), a reduction of the contact angle immediately after NTP treatment (0 degrees) was observed compared with other periods. This indicates a slight increase in the polar components on the ceramic surface immediately after NTP treatment (T1) and their decrease with aging, resembling the baseline results (T0). The surface energy was similar in periods T0, T2, T3, and T4 ($P > .05$).

Table 2 shows the ratios C/Si, O/Si, and O/C determined from the proportions of the elements identified by EDS spectra. The ratio O/Si was approximately 2.0 for the results obtained immediately after NTP treatment, which is typical of silica, with 2 O for each Si. This result explains the high surface energy of these specimens. However, the O can also be connected to the C (SiO_2C). Furthermore, a tendency to increase the proportion of C and O on surfaces aged for 7 days was observed (SiO_4C_3), consistent with surface energy reduction. An opposite trend was observed with increasing aging time, in which the atomic ratios tend toward those obtained immediately after NTP treatment.

DISCUSSION

The null hypotheses that the organosilicon film would not resist atmospheric air aging and that no significant reduction in the surface energy after aging periods were partially accepted because, although the film topographic characteristics were not altered by atmospheric air aging, the maintenance of the hydrophilic surface was not sustained with aging. The surface energy increased significantly at T1.

The organosilicon film on the ceramic surface maintained its topographic characteristics in all tested periods after its deposition without exhibiting signs of degradation or detachment (Fig. 1). This fact can be confirmed by SEM and AFM analyses, which enable surface observation of several ceramic systems with high precision and 3-dimensional investigation of surface roughness.^{25,26}

Regarding the chemical analysis of the surface, the initial EDS mapping results reflect the lithium disilicate ceramic composition (Table 1)^{10,16,17} because Zn and Al

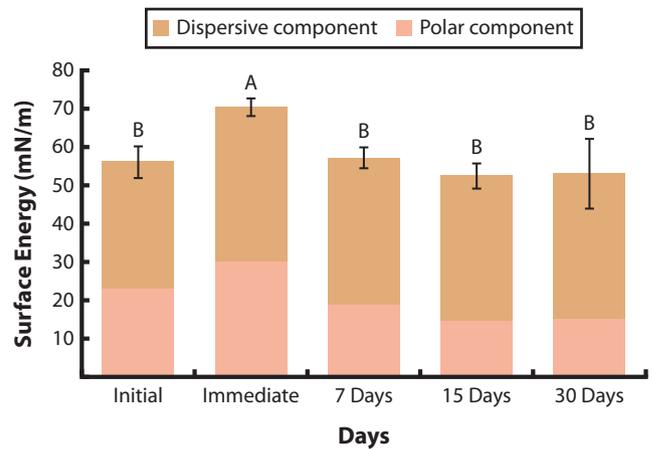


Figure 3. Surface energy of polar and dispersive components of lithium disilicate disks before NTP treatment, immediately after NTP treatment, and after atmospheric air aging for 7, 15, and 30 days. Different uppercase letters denote statistically significant differences ($P < .05$).

were detected (Fig. 2). After NTP treatment, high quantities of C, O, and Si were identified. The presence of these components indicated that the film was predominantly organosilicon.

The results of the present study showed that the organosilicon film recovered the ceramic surface and remained intact during aging. Furthermore, EDS mapping results indicated that the organosilicon film thickness was relatively small because this analysis can be used to determine the material surface composition up to a depth of 1 μm ,^{5,6,22} and Zn and Al elements, initially identified on the ceramic surface, were not detected after the film deposition. A thin film is advantageous because it would not interfere with the seating of the ceramic restoration.⁴

Ceramic surface wettability promotes resin cement flow, optimizing adherence.¹⁹ Therefore, the higher the ceramic surface energy, the greater the physical-chemical interaction between the bonding material and the prosthesis. In the present study, the contact angle formed between the drop of water and the disilicate surface was approximately 0 degrees, lower than those obtained in other tested periods (Fig. 4). Organosilicon films, unlike silicon oxide films, have high ratios of carbon,²⁴ as detected in EDS analysis (Fig. 2). This material has an essentially inorganic skeleton (Si-O) with methyl groups attached to the silicon forming methylsilyl, dimethylsilyl, and trimethylsilyl on the surface. The polar Si-O groups are surrounded by the nonpolar groups (CH_3). The electrostatic shield promoted by the organic groups reduces the water affinity of this material.²⁴ However, a highly receptive water surface seems to be created by oxygen plasma treatment after deposition. In this treatment, abundant atomic oxygen with a high affinity to organic groups is formed. The interaction between activated oxygen plasma and the surface removes

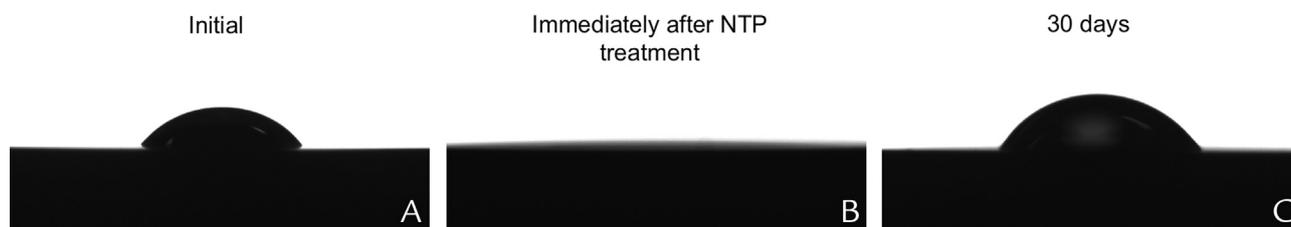


Figure 4. A, Representative image of configuration assumed by deionized water drops deposited on lithium disilicate surface before NTP treatment. B, Representative image of configuration assumed by deionized water drops deposited on lithium disilicate surface immediately after NTP treatment. C, Representative image of configuration assumed by deionized water drops deposited on lithium disilicate surface after 30 days of NTP treatment aging.

the methyl groups attached to the silicon of the organosilicon film, exposing the polar inorganic skeleton. Thus, the superhydrophilicity ($\theta < 10$ degrees) of the ceramic surface after NTP treatment is caused by the removal of the methyl groups from the organosilicon surface that shields the interaction between the water molecules and the polar groups of this structure.^{7,8,22,23,27} The increase in the polar component of surface energy supports this proposal (Fig. 3). Similar results have been reported for zirconia recovered with organosilicon film.⁸

The significant reduction of surface energy through atmospheric air aging indicates that impurities are incorporated into the surface, altering its chemical nature (Fig. 3). These results are consistent with those of Yi et al,²⁷ who found that the hydrophilicity resulting from oxygen plasma treatment was stable for only a few days or hours of air aging. The incorporation of hydrocarbons and the formation of new connections with components such as C and O made the surface tend once again toward an organosilicon film. The increasing trend of these components can be observed in EDS analysis after aging in atmospheric air for 7 days (Fig. 2, Table 2).²⁷

Additionally, the surface energy in the organosilicon film could have been reduced because of the migration/rotation of the polar groups incorporated after oxygen plasma treatment from the films outermost to innermost layer. The movement shields highly reactive polar groups from the nonpolar components that take their place on the surface.²⁵

Organic or inorganic contamination of the ceramic surface negatively interferes with its adhesive cementation.¹⁷ Good adhesion between the ceramic surface and the tooth structure is required for restoration success after definitive cementation.¹⁴ To increase the clinical longevity of the lithium disilicate ceramic, a durable and widely used dental ceramic system,⁵ contamination must be avoided during cementation.^{7,8}

In the present study, the aging periods tested were chosen to simulate the time between the surface treatment in the dental laboratory and the ceramic cementation process. The surface energy was statistically higher in the period immediately after the organosilicon film

deposition, suggesting that the restoration should be cemented as soon as possible after this procedure.

The present study has some limitations. Aging intervals between the period immediately after the organosilicon film deposition and after atmospheric air aging for 7 days were not evaluated. In addition, bond strength was not analyzed, and NTP treatment was not compared with those obtained by etching the ceramic surface with HF acid. Additional studies should be performed with shorter aging periods to identify when the effect of the plasma treatment changes with the reduction of the superhydrophilicity of the ceramic surface.

CONCLUSIONS

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

1. Plasma treatments with hexamethyldisiloxane associated with argon and followed by oxygen plasma significantly increased the surface energy of lithium disilicate ceramic.
2. Although atmospheric air aging did not modify the film morphology, the surface energy decreased significantly. The wettability was not durable after atmospheric air aging immediately after NTP treatment (T1).

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Corresponding author:

Dr Daniela Micheline dos Santos
 Department of Dental Materials and Prosthodontics
 Aracatuba Dental School, Sao Paulo State University (UNESP)
 Jose Bonifacio St, 1153, Vila Mendonca, Aracatuba
 Sao Paulo, 16015-050
 BRAZIL
 Email: danielamicheline@foa.unesp.br

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