

Non-Thermal Atmospheric Plasma: Can it Be Taken as a Common Solution for the Surface Treatment of Dental Materials?*

Emre SEKER¹, Mehmet Ali KILICARSLAN², Serdar POLAT³, Emre OZKIR⁴,
Suat PAT⁵

¹Department of Prosthodontics, Faculty of Dentistry, Eskisehir Osmangazi University,
Eskisehir 26480, Turkey

²Department of Prosthodontics, Faculty of Dentistry, Ankara University, Ankara 06100,
Turkey

³Department of Prosthodontics, Faculty of Dentistry, Gazi University, Ankara 06500,
Turkey

⁴Department of Prosthodontics, Faculty of Dentistry, Afyon Kocatepe University,
Afyonkarahisar 03200, Turkey

⁵Faculty of Art and Science, Eskisehir Osmangazi University, Eskisehir 26480, Turkey

Abstract This study aimed to evaluate the surface roughness and wetting properties of various dental prosthetic materials after different durations of non-thermal atmospheric plasma (NTAP) treatment. One hundred and sixty discs of titanium (Ti) ($n:40$), cobalt chromium (Co-Cr) ($n:40$), yttrium stabilized tetragonal zirconia polycrystals (Y-TZP) ($n:40$) and polymethylmethacrylate (PMMA) ($n:40$) materials were machined and smoothed with silicon carbide papers. The surface roughness was evaluated in a control group and in groups with different plasma exposure times [1-3-5 s]. The average surface roughness (Ra) and contact angle (CA) measurements were recorded via an atomic force microscope (AFM) and tensiometer, respectively. Surface changes were examined with a scanning electron microscope (SEM). Data were analyzed with two-way analysis of variance (ANOVA) and the Tukey HSD test ($\alpha=0.05$). According to the results, the NTAP surface treatment significantly affected the roughness and wettability properties ($P < 0.05$). SEM images reveal that more grooves were present in the NTAP groups. With an increase in the NTAP application time, an apparent increment was observed for Ra, except in the PMMA group, and a remarkable reduction in CA was observed in all groups. It is concluded that the NTAP technology could enhance the roughening and wetting performance of various dental materials.

Keywords: dental material, atmospheric plasma, surface treatment, roughness, contact angle

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(Some figures may appear in colour only in the online journal)

1 Introduction

The adhesion capability and surface activation of restorative materials remain important to the reliable clinical performance of dental restorations. Durable and strong adhesive bonding between the luting agent and the framework or between different materials in the structure of prosthesis is necessary to withstand varied challenges in the oral environment [1]. Adequate bonding depends on both micromechanical interlocking and physicochemical treatments [2].

Dental alloys, such as Ti and Co-Cr, have been treated for surface activity in many different ways, including electrolytic etching [3], sandblasting [4–6], the

deposition of a layer of silica [7] and the application of alloy primers [8]. All of these techniques attempt to enhance the bond strength and the bond stability for long-term clinical performance. However, the mechanism underlying adhesion between adhesive materials and metal alloys remains unclear [8].

Conversely, the same efforts have been given to the surface treatment of ceramics with hydrofluoric acid followed by silane application [9]. Although this procedure has been proven effective for glass ceramic systems, more structurally resistant and inert surface ceramics, such as high strength Y-TZP, have required resin bonding cementation concepts to promote adequate surface characteristics [2,10]. Even though these methods pro-

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mote effective bonding to Y-TZP substrates, particle abrasion may create structural defects and mechanical damage, which might be detrimental for the long-term performance of the restoration [11,12]. Selective infiltration etching (SIE) [13] and chloro-silane combined with water vapor [14] for adhesive bonding to Y-TZP have been introduced, but further research on these materials is needed.

Since acrylic polymers are the most widely used base material for constructing removable dentures, many different efforts have been made to enhance their surface energy and wettability with various surface modification procedures [15]. Evaluations of the frequency of various denture repairs have revealed that tooth debonding is a commonly encountered problem for conventional prosthesis [16]. Additionally, with an increase in implant applications and the associated increase in the occlusal loads applied to the supported tissues by prosthetic construction, denture tooth debonding or denture base fracture may become a frequent clinical problem for implant-retained removable prosthesis [17]. Currently, the bond strength of PMMA and alloy is substantially more important, particularly in hybrid prosthesis. Therefore, various chemical treatments have been driven to improve the wettability and adhesive capability of the PMMA material, such as the use of monomers, nonpolymerizable solvents, dissolved PMMA, tribochemical silica coating + silanization, silicon tetrachloride or combinations of the above [15,18]. The acceptable bond strength depends on the wettability between the adhered surface and the adhesive, which is necessary to ensure adhesion [19]. Wetting is the first condition for providing adhesion [20]. The surface energy (SE) of the solid material should be higher than the surface energy of the adhesive to ensure a lower CA that represents effective wettability. Accordingly, the CA measurement is an appropriate indicator of the wetting ability of an adhered surface for a liquid adhesive [21]. The actual contact area determines the degree of wetting between the adhesive and the solid surface [20]. There is a need for a more universal and, perhaps, innovative approach to promote the appropriate bonding to all dental substructures, such as Ti, Co-Cr, Y-TZP and PMMA. It has been reported that the present methods of roughening the surface of alloys and ceramics provide better wettability by increasing surface area [22]. At the same time, the literature has emphasized that it is necessary to develop additional modifications and innovative applications for increasing the SE [21–23]. In this way, the effect of the plasma on surface roughening and SE may be analyzed.

Plasma is defined as the fourth state of matter in nature, in addition to the solid, liquid and gaseous states. Plasma surface treatment has more frequently been used to modify the surface of products in the industrial sector [24,25]. Plasma is an electrically neutral, ionized gas that consists of ions, electrons, neutral particles, vacuum ultraviolet and ultraviolet irradiation, free radicals and chemically reactive neutral particles;

under normal pressure conditions, no vacuum ultraviolet radiation is produced [25]. Atmospheric pressure plasma jets have undergone extensive development in recent years due to their use as simple experimental apparatuses [26]. They have been investigated for improving the mechanical properties of acrylic resin materials [15,27–30] and increasing the bond strength between composite luting agents and alloy [21,25,31] or ceramic surfaces [21,32,33] in dentistry. Appropriate plasma processes render surfaces hydrophilic and modify the oxide layer [27,34], thereby influencing the adhesion ability. Plasma irradiation does not cause environmental pollution because it does not require chemical additives [24]. It was reported that the NTAP technique is cost-efficient, safe and relatively simple to handle [26,27,34].

The aims of this investigation were the following: (1) determine the Ra of Ti, Co-Cr, Y-TZP and PMMA surfaces exposed to 1–5 s of NTAP treatment; (2) cross-check the surface rheology of Ti, Co-Cr, Y-TZP and PMMA specimens before and after NTAP application via AFM and scanning SEM images; and (3) measure the CA of distilled water dispensed on Ti, Co-Cr, Y-TZP and PMMA surfaces before and after plasma treatment.

The following two hypotheses were tested:

a. An increase in the Ra levels of Ti, Co-Cr, Y-TZP and PMMA surfaces is expected after 1 s, 3 s and 5 s of NTAP application.

b. A reduction in the CA of distilled water applied to Ti, Co-Cr, Y-TZP and PMMA substrates is expected after similar NTAP surface treatment.

2 Experimental setup

In total, 160 discs (10 mm × 2.0 mm) of Ti (*n*:40), Co-Cr (*n*:40), Y-TZP (*n*:40), and PMMA (*n*:40) materials were machined and smoothed consecutively with 600-, 800-, and 1200-grit silicon carbide papers (English Abrasives; London, England) under running water to obtain standardized surface roughness; they were then cleaned in an ultra-sonic bath (Sonorex, Bandelin; Berlin, Germany), that was filled with distilled water, for 5 min.

Each material group was separated into two parts: Ra (*n*:28) and CA (*n*:12) assessment parts. The Ra part was divided into the following 4 subgroups (*n*:7): 1 control group (no exposure) and 3 groups that had consecutive application times of the plasma exposures (1–3–5 s). The group material types and distribution are described in Table 1.

2.1 NTAP treatment

An atmospheric plasma torch (Plasmatrete GmbH, Bisamweg 10, Steinhagen, Germany) was performed for surface treatment. It does not require any special gas; additionally, it generates plasma at room temperature and operates at a frequency of 21 kHz with an applied voltage of 5 kV without revealing perceivable heat. The

treatment speed was 5 m/min, with a pressure of 2 bar, and the approximate jet power was 1 kVA, which was used for 1-3-5 s with a distance of 5 mm between the nozzle and the test surfaces. Plasma stability control was realized with plasma plume length. Also it was attempted to maintain the specimens within the plasma.

2.2 AFM evaluation

The Ra of the specimens was measured and characterized with an AFM device (Auto-probe CP-II; Veeco, Plainview, NY, USA). The radius of curvature of the scanning tip was 10 nm. Images were recorded with a scan rate of 1 Hz at a resolution of 512×512 pixels per image and scanning area of 10×10 μ m. Each specimen was measured at 3 different sites, and the mean was calculated. The average Ra of the specimens after different treatments was recorded in nanometers (nm).

2.3 CA evaluation

For wettability assessment, separate Ti, Co-Cr, Y-TZP and PMMA discs ($n:12$) were horizontally aligned in a KSV Attension Theta Lite Optical Tensiometer and the equilibrium (θ_e), advancing (θ_a) and receding (θ_r) CAs of distilled water drops were measured under air. A HAMILTON syringe with a volume of 1 mL was used to form liquid droplets on each control and NTAP

treated surface. Three CA readings were performed and averaged.

2.4 SEM evaluation

Two specimens (Control and 5 s NTAP-treated) from each group were examined under SEM (JSM-5600 Scanning Microscope; JEOL Ltd, Tokyo, Japan) to assess changes in the surface topography before and after treatment. Representative specimens were photographed at 500 X magnification.

2.5 Statistical analysis

The Ra and CA values were analyzed by a two-way ANOVA using the Minitab 16 package software. According to the assumption of homogeneity of variance, the post-hoc Tukey's multiple comparison test was used. Statistical significance was set at the 0.05 probability level.

3 Results and discussion

The results of two-way ANOVA (Table 2) indicated that the surface treatment methods significantly affected the roughness and wettability values. Tables 3 and 4 show the mean values, standard deviations and statistical results for both Ra and CA measurements.

Table 1. Material properties

	Origin and Experimental Groups
Titanium Grade 5 (Ti-6Al-4V)	Biotech srl, Povolario di Dueville, Italy TC (Control): no NTAP T1:1 s NTAP T3: 3 s NTAP T5: 5 s NTAP
Co-Cr (Magnum Lucens, Type 4)	MESA, di Sala Giacomo&C. S.n.c. Travagliato, Italy CC (Control): no NTAP C1:1 s NTAP C3: 3 s NTAP C5: 5 s NTAP
Y-TZP	3M ESPE Dental Products, St. Paul MN/USA ZC (Control): no NTAP Z1:1 s NTAP Z3: 3 s NTAP Z5: 5 s NTAP
PMMA	Bloomden Bioceramic Co. Ltd, Hunan, China PC (Control): no NTAP P1:1 s NTAP P3: 3 s NTAP P5: 5 s NTAP

Table 2. ANOVA results for mean surface roughness (Ra) and mean contact angle (Ca) values

	Ra (nm) ^a					Ca (degree) ^b				
	SS	df	MS	F	P	SS	df	MS	F	P
Material type	105326	2	52663	827.16	<0.001	5058.0	3	1686.0	1718.97	<0.001
Duration	407592	3	135864	2133.97	<0.001	16324.4	3	5441.5	5547.84	<0.001
Material type X duration	94405	6	15734	247.13	<0.001	3129.2	9	347.7	354.49	<0.001
Residual	4584	72	64			31.4	32	1.0		
Total	611908	83				24543.0	47			

^aR-Squared = 99.25%, Adjusted R-Squared = 99.14%; ^bR-Squared = 99.87%, Adjusted R-Squared = 99.81%

Table 3. Mean $\pm$ SD of the surface roughness (Ra) values

Application time	n	Ti	Co-Cr	Y-TZP	PMMA*
Control	7	51.0 $\pm$ 1.39 ^G	42.8 $\pm$ 2.74 ^G	28.1 $\pm$ 3.24 ^H	198.43 (Mean)
1 s	7	114.5 $\pm$ 13.8 ^D	95.7 $\pm$ 12.6 ^E	57.0 $\pm$ 7.64 ^G	133.86 (Mean)
3 s	7	115.8 $\pm$ 5.33 ^D	193.4 $\pm$ 10.5 ^C	75.3 $\pm$ 4.23 ^F	-
5 s	7	324.7 $\pm$ 8.51 ^A	237 $\pm$ 9.83 ^B	128.4 $\pm$ 2.87 ^D	-

Means that do not share a letter are significantly different for post-hoc Tukey test (P<0.05).

* The number of measurements is incomplete for statistical evaluation.

Table 4 Mean $\pm$ SD of the Contact Angle (Ca) Values

Application time	<i>n</i>	Ti	Co-Cr	Y-TZP	PMMA
Control	3	8.1 $\pm$ 1.2 ^A	91.8 $\pm$ 0.71 ^B	65.5 $\pm$ 0.47 ^F	76.8 $\pm$ 0.41 ^D
1 s	3	84.9 $\pm$ 1.05 ^C	53.2 $\pm$ 0.09 ^H	43.2 $\pm$ 0.04 ^I	73.2 $\pm$ 0.23 ^E
3 s	3	39.2 $\pm$ 0.04 ^J	39.3 $\pm$ 0.6 ^J	33 $\pm$ 0.06 ^K	60.8 $\pm$ 3.42 ^G
5 s	3	37.5 $\pm$ 0.01 ^J	31.5 $\pm$ 0.1 ^K	20.9 $\pm$ 0.08 ^L	53.6 $\pm$ 0.2 ^H

Means that do not share a letter are significantly different for post-hoc Tukey test ($P < 0.05$).

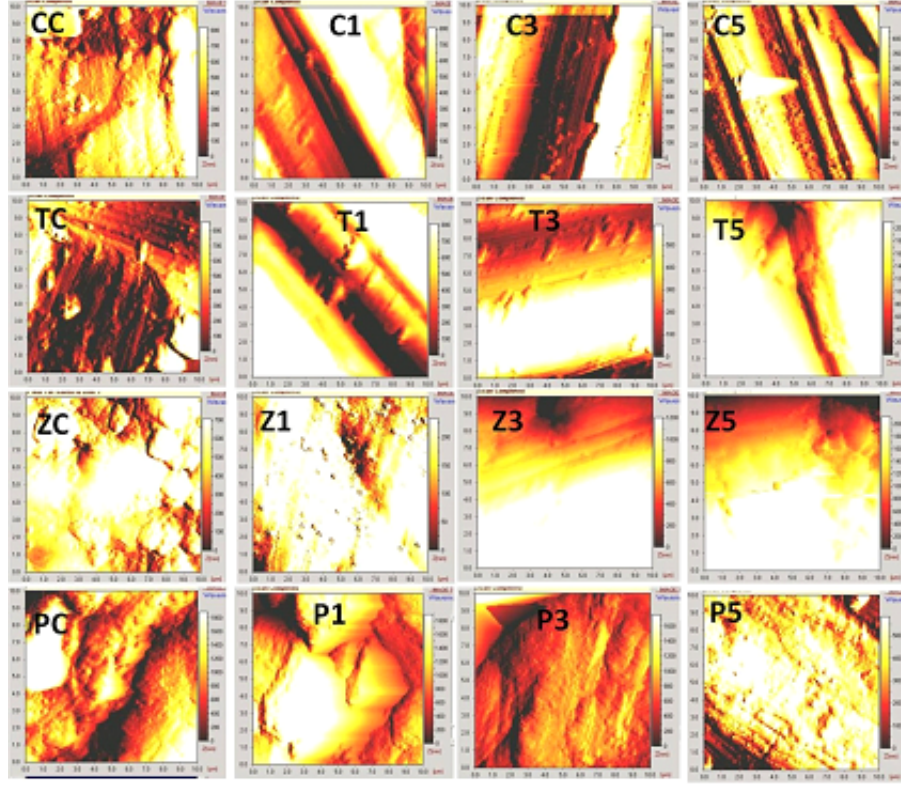


Fig.1 Representative AFM images and Ra of the untreated control (TC-CC-ZC- PC) and the plasma treated surfaces with different exposure durations (1-3-5 s)

3.1 Surface roughness measurements

The highest (324.7 ± 8.51 nm) and the lowest (28.1 ± 3.24 nm) surface roughness values were determined for T5 and ZC samples, respectively, in all groups (Table 3). However, the number of measurements for the PMMA samples is incomplete for statistical evaluation; therefore, all PMMA specimens were disqualified from the Ra investigation. With respect to the other three material groups, there were significant differences ($P < 0.05$) between each group, except between T1 and T3 and among TC, CC and Z1 ($P > 0.05$), for the Ra.

The effects of NTAP treatment on the surface morphology were clearly observed on three-dimensional AFM images with well-defined macro-sized elevated pit and groove areas (Fig. 1). However, images of the latter two applications could not be assessed for surface burning in the PMMA samples.

3.2 Contact angle measurements

The highest ($98^\circ \pm 1.2^\circ$) and the lowest ($20.9^\circ \pm 0.08^\circ$) contact angle values were determined for the TC and

Z5 samples, respectively, in all groups (Table 3). Representative images of the CA before and after plasma treatment for Ti, Co-Cr, Y-TZP and PMMA are presented in Fig. 2. There were significant differences ($P < 0.05$) between each group, except between T3, T5 and C3 and between C5 and Z3 ($P > 0.05$), for the CA. There were also distinctive decreases with increasing application time in the overall CA values.

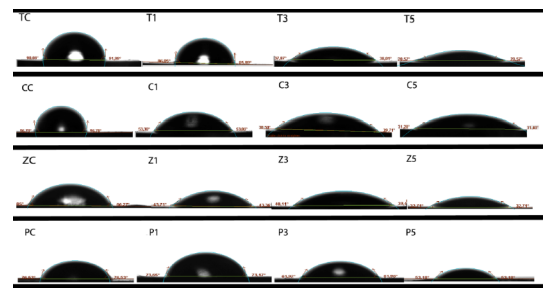


Fig.2 Series of images illustrating the contact angle of drops of distilled water on the untreated control (TC-CC-ZC-PC) and the plasma treated surfaces with different exposure durations (1-3-5 s)

3.3 SEM findings

SEM examination showed that groups with different values have varied surface topography compared with the control (Fig. 3). All of the different material groups differ in terms of their topography, and they were very rough due to the deep macro cavities and porosities covered with micro gaps, which possibly resulted from the high impact of abrasive papers. The original control surface exhibited pronounced grinding marks, which appeared rough with irregular, embedded alumina and silica particles. SEM images reveal that more grooves were present in the NTAP groups. In the NTAP groups, the superficial fissured coarse surface had carbon spots, but the morphological characteristics of the sample surface did not differ in any important way from the control group, except in the case of PMMA samples. Unlike other samples, the PMMA samples had surface burns with increases in the application time (3 s and 5 s).

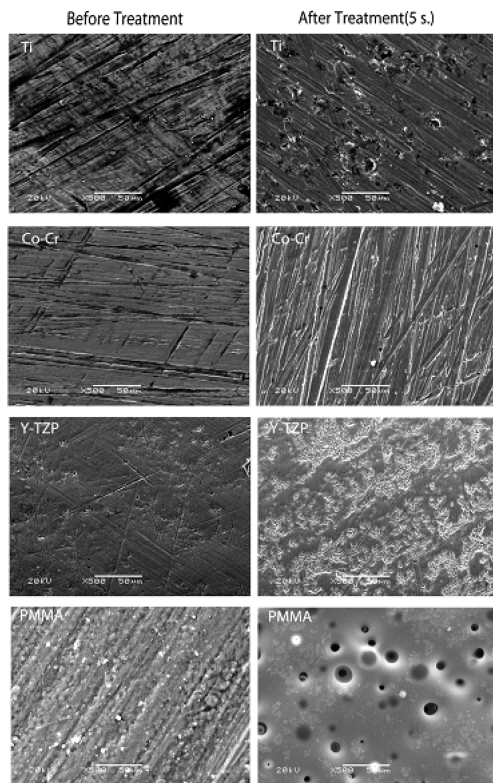


Fig.3 Representative SEM images of samples before and after NTAP surface treatment

Surface modification of biomaterials continues to be investigated to improve biomaterial-biomaterial interactions and compatibility, particularly in restorative dentistry [35]. The most commonly used method of roughening a dental material's surface is airborne-particle abrasion, which is expected to enhance the bond strength by providing mechanical interlocking that is considered a significant factor influencing wetting and increasing surface area [4–6]. Some publications have mentioned that micromechanical roughening cleans and activates, thus enabling the better wetting of the Y-TZP surface [36,37]. Nevertheless, several studies

have reported that airborne-particle abrasion contaminates the Ti surface [4,5] and creates structural defects and mechanical damage on the Y-TZP surface [11–13], which might arise from the compromised materials' corrosion resistance and biocompatibility and, in turn, be detrimental for the long-term performance of the restoration. The contamination [4,6] and distortion [5] of the substrate are undesirable, and this method remains sensitive to the technique used. At the same time, a number of studies have evaluated the effects of atmospheric plasma on certain types of dental material surface for polymers [15,27–31], zirconia [12,21,33], and Ti [21,25,31]. To the best of our knowledge, the effect of plasma surface treatment on Co-Cr alloys and the aforementioned comparison of the materials have not been previously investigated, making this article noteworthy.

Some authors have reported that appropriate plasma processes render surfaces hydrophilic and modify the oxide layer, which improves wettability and thus allows for the formation of stronger bonds and produces chemically active sites for the adhesion of other molecules [15,21,24,27]. It has also been reported that with the use of atmospheric gases, such as He, O₂ and N₂, NTAP treatment increases the number of oxygen-containing hydrophilic polar groups, including C–O–C and O=C–O, and reduces C–C links on the material surface [28]. Furthermore, by breaking the C–H and C–C bonds, the use of NTAP also has cleaning potential [37], which may play a highly effective role in enhancing the adhesion capacity.

An overall assessment of studies that compare the plasma technique with other surface treatments is presented here. Derand et al. revealed that pretreating zirconia ceramic surfaces with plasma can successfully increase the adhesion to composite resin cements compared with silane application [10]. Valverde et al. obtained significantly higher bond strength values in all NTAP groups compared with their respective non-NTAP groups on the Y-TZP surfaces [33]. Additionally, Nishigawa et al. reported that plasma treatment was more efficient than the adhesive primer application in increasing the bond strength of self-curing PMMA to heat-cured PMMA resin [24,29]. Ozden et al. concluded that plasma treatment offers a durable (up to at least 60 days) wettability by altering the surface of the PMMA resin [15]. They also emphasized no contamination effect after plasma treatment, which is a surprising and promising result. In the present study, after using silicon carbide papers (control groups), the PMMA samples had the highest and Y-TZP samples the lowest roughness values compared with the Ti and Co-Cr samples (Table 3). Moreover, the lowest value of both roughness and contact angle was observed for the Y-TZP measurements in all NTAP groups (1-3-5 s). Unexpectedly, the PMMA samples were damaged in the last two steps so that roughness could not be assessed in these stages. However, no problems were encountered in the measurement of the CA.

With respect to a general comparison, the highest and the lowest values of all measurements varied for each group according to the type of the material. This finding might be related to the difference in hardness among the substrates, and it can be explained by the material hardening and the decrease of roughness value. Additionally, each material has different hydrophilic and hydrophobic affinities in terms of the CA values. When all materials were compared, regardless of the rate of increase in Ra, this experimental study revealed that the Ti, Co-Cr and Y-TZP control groups were 6.3-, 5.6-, and 4.5-fold increased, respectively, after 5 s of NTAP treatment. Conversely, in case of CA, the Ti, Co-Cr, Y-TZP and PMMA control groups were 2.6-, 2.9-, 3.2- and 1.4-fold decreased, respectively, after 5 s of NTAP treatment. This observation was supported by the AFM and tensiometer findings, which revealed the efficacy of the different material types in plasma surface treatments (Figs. 1 and 2).

Although some publications have reported that the Ra could increase the stress concentration at the material surface and produce sharp angles, which could cause voids at the metal-cement interfaces and prevent complete wetting [4,6,38], the Ti samples in the present study had a greater degree of roughness ($324.7 \pm 8.51 \mu\text{m}$) following a 5-s application compared with the other, previously mentioned groups (Table 3). Ti is a highly reactive element, and oxygen has a high affinity for Ti; this interaction thus spontaneously results in an oxidized surface, which is beneficial for the bonding mechanisms. In this study, this characteristic feature might explain the high wettability results of the Ti samples.

In this study, when considering each material individually, we found that CA decreases with increasing roughness. This finding is in agreement with several studies [21,33]. If we compare different materials, the Y-TZP samples have the lowest Ca values with the lowest roughness values for all application times. However, the opposite was true for the PMMA samples. We can clearly conclude that the degree of wettability depends on the molecular structure of the material. In the present study, the CA values for all specimens were decreased with increasing execution time. Our findings are in agreement with the following authors. Fang et al. reported that an increase in the execution time and applied power of plasma discharge would increase the hydrophilic polar groups on a material's surface [30]. Duske et al. evaluated the effect of NTAP on the Ti surface wettability with the use of distilled water and reported that the CA of all specimens were significantly decreased after plasma treatment, from 70° - 120° to 0° - 10° , as compared with untreated discs [25]. Additionally, Silva et al. evaluated the effect of NTAP on the Y-TZP and Ti surface wettability using an MDP primer and reported that the CA decreased from 15° to 0° for Y-TZP and a 0° CA was obtained on the Ti surface before and after plasma treatment for Ti specimens [21]. Of note, all CA values were significantly

different ($P < 0.05$), except for the T3 and T5 groups. Therefore, the application time will not make a difference for Ti specimens when it is longer than 3 s.

The outcomes of the physical and chemical treatment on the surface hydrophilicity and wettability were investigated by measuring their CA. In the present study, the mean CA measurement values were significantly less than those in previous, short-time applications for all material types. The additional NTAP treatment decreased the value of the CA from ($98.1^\circ \pm 1.2^\circ$) to ($37.5^\circ \pm 0.01^\circ$), from ($91.8^\circ \pm 0.71^\circ$) to ($31.5^\circ \pm 0.1^\circ$), from ($65.5^\circ \pm 0.47^\circ$) to ($20.9^\circ \pm 0.08^\circ$), and from ($76.8^\circ \pm 0.41^\circ$) to ($53.6^\circ \pm 0.2^\circ$) for Ti, Co-Cr, Y-TZP, and PMMA, respectively (Table 4). With this finding it should not be surprising that higher wettability was determined relative to short time treated or non-treated specimens [25,39]. Our results are in agreement with previous NTAP studies on the increase in surface wettability of different substrates. Conversely, some studies have reported that there is no significant difference in the SE values for Y-TZP and Ti after different plasma exposure times (5 s, 10 s, or 20 s) [21].

The effects of the physical and chemical treatment on surface morphology are illustrated in three-dimensional topographic AFM and SEM images (Fig. 1, Fig. 3). The AFM images clearly show that large morphological changes occurred after treatment. In addition to the exposure time, the range and waveform of the applied power and the distance between NTAP nozzle and sample surface affected the number of active species that interact with the material surface [21,28,30]. However, the applied power and application distance were constant for all experiments in this study. Our study may be limited in this respect. The role of NTAP on the surface adhesion capability remains to be investigated because the present results only revealed surface changes. Bonding behaviors with various cements may be investigated in future studies. Before recommending these materials for clinical use, the effects of thermal cycling and long-term experiments in water should be performed.

Although the surface treatment of biomaterials can be performed by either chemical or physical methods, the physical method could provide more precise surface modifications without requiring rigorous and complicated process control. This advantage eliminates surface damage and subsequent surface modification problems. No chemical solutions are involved in the plasma process, and there is no need for disposal of waste liquids and toxic by-products in this fast, cost-effective and environmentally friendly approach [40].

4 Conclusions

The present study shows that the novel NTAP technology can enhance roughening and wetting performance to different dental materials. Further studies will address the effect of NTAP on the long-term durability

of bonding various luting cements to denture material surfaces.

a. This treatment may be considered an alternative to conventional surface treatment methods.

b. The time for treatment is an important factor in the NTAP treatment.

c. NTAP treatment is more effective for low-hardness materials.

d. NTAP treatment may damage the surface properties of PMMA samples.

With respect to the Ra, an apparent increment was observed for all specimens with increased application times. These increased Ra values are significant except for T1s and T3s groups. These findings support hypothesis (1). With respect to the CA, a remarkable reduction was observed for all specimens with increasing application times. This reduction is significant, except in the T3s and T5s groups, thus supporting hypothesis (2).

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E-mail address of Emre SEKER:

emreseker@hotmail.com