Effect of plasma treatment on the bond of soft denture liner to conventional and high impact acrylic denture materials

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ABSTRACT

Background: The main drawback of soft lining materials was that they debonded from the denture base after a certain period of usage. Therefore, the purpose of this research was to determine the impact of oxygen and argon plasma treatment on the shear bonding strength of soft liners to two different kinds of denture base materials: conventional acrylic resin and high impact acrylic resin.

Materials and Methods: Heat cure conventional and high impact acrylic blocks (40 for each group) were prepared. A soft liner connected the final test specimen of two blocks of each acrylic material. Shear bond strength (SBS) was assessed using universal testing machine. Additional blocks were also prepared for analyzing Vickers microhardness, contact angle, FTIR and AFM. The results were statistically analyzed using paired-sample T-test and independent-samples T-test ($\alpha=0.05$).

Results: The results showed a highly significant increase in SBS following plasma treatment with the highest mean value observed in plasma treated high impact acrylic specimen. Along with a significant rise in wettability, while microhardness was preserved.

Conclusion: In conclusion, oxygen and argon plasma treatment was significantly effective in enhancing the SBS between soft liner and acrylic materials.

Keywords: Plasma treatment, high impact acrylic, shear bond strength, microhardness, soft liner. **(Received: 1/6/2021, Accepted: 4/7/2021)**

INTRODUCTION

Acrylic resins are the most popular choice for fabricating denture bases due to their ease of processing, low cost, and aesthetic appeal.(1) The high frequency of fractures necessitated the need of methods for enhancing fracture resistance of denture bases. High impact strength acrylic was developed for this purpose.(2) Chemical modification of acrylic resin by incorporating rubber in the form of butadiene styrene has been proved effective in terms of enhancing fracture resistance and impact strength of the denture bases against unexpected high forces.(3)

While retention is critical for a good denture over time, dentures can become ill-fitting due to residual ridge resorption causing discomfort and pain to the patient.(4) This issue can be addressed by the use of resilient denture liners. Soft liners provide cushioning effect assisting in the distribution and reduction of the functional forces, as well as helping the tissue in recovering from trauma giving comfort to the patient.(5)

Soft liners serve in restoring the fit of the denture base in a variety of other clinical situations, including dentures that oppose natural dentition, xerostomia, and bony undercuts.(6) However, they suffer from a serious shortcoming, which is their debonding from the denture base following prolonged use, which may create a favorable environment for the growth of bacteria, thereby speeding up the decomposition of the material. (7,8)

Various methods of surface modification have been tested out to overcome the problem of debonding between acrylic resins and soft liners. One of these methods is treating the surface with plasma.(9) Plasma is made up of electrons and ions as well as neutrals, atomic and molecular species that behave collectively in the presence of an electromagnetic field.(10) It has been discovered that plasma treatment with oxygen increases the hydrophilicity of polymer surfaces, thus increasing their surface energy.(11) On the other hand, it has been reported that plasma treatment of polymers with argon gas induces polymer cross linking properties.(12)

There are a variety of generally accepted measures for determining the soft liner's mechanical properties, including tensile, peel, and shear bond strength. Al-Athel & Jagger (1996) claimed that shear bond strength (SBS) test had the best approximation of the situation that is present in the oral cavity in terms of the direction of forces which result in debonding of soft liner.(13)

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Therefore, the objective of this research was to study the effect of oxygen and argon plasma treatment on shear bond strength of soft liner to two types of denture base materials; conventional heat cure and high impact acrylic. The null hypothesis suggested that plasma treatment would have no positive impact on the shear bond strength.

MATERIALS AND METHODS

Preparation of specimens

The same procedure was used to prepare test specimens for both of the acrylic materials: conventional acrylic (n=20, for each test) and high impact acrylic (n=20, for each test). Twenty specimens for testing shear bond strength were prepared. Each specimen consisted of two blocks with dimensions of (75 × 13 × 13 mm) length, width, thickness, respectively, with a 3 mm depth stopper.(14) One block is fixed on top of the other, leaving a gap in between for the application of the soft liner material. Plastic blocks were constructed with the dimensions mentioned earlier; to be duplicated into acrylic. Laboratory silicone putty (Zetalabor, Zhermack, Italy) was used to aid in the duplication process (Figure 1). The putty was prepared by mixing its base and catalyst (according to the manufacturer's instructions, it was kneaded until it became homogenous, the plastic blocks were then invested in the silicone. After setting of the putty, the excess was sliced off a sharp knife. The final piece of putty was then inserted in stone in a regular flask. Following setting of stone, the plastic block was removed leaving a space for molding acrylic material.

Conventional acrylic (SpofaDental, Czech) was mixed as directed by the manufacturers; in a ratio of 2.2 g:1 ml. While high impact acrylic (Vertex, Netherlands) was mixed with a ratio of 2.1 g:1 ml. Each acrylic was then packed in dough stage into the silicone molds, the upper and lower parts of the flask were re-assembled until edge-to-edge contact was achieved, and placed under pressure using hydraulic press (100 kPa) to ensure even distribution of the material, and left there under pressure for 5 minutes. The flask was mounted into a clamp and submerged in boiling water in a digital water-bath, the heat was maintained at 70°C for an hour and a half, then the temperature was raised to 100°C for half an hour. After bench cooling of the flask for 30 minutes, the acrylic specimens were collected, finished and polished in the regular way. To create a smooth, flat surface, the targeted treatment surface was polished using gradually finer grades (600-1200) of silicon carbide paper. A digital vernier was used to verify the size of the acrylic blocks. The blocks were then stored in plastic containers with distilled water.

Specimens for testing microhardness were prepared in the same way (12 × 12 × 3 mm). (15) Specimens for testing wettability were also prepared (20 × 15 × 2 mm).(16) All specimens were thereafter cleaned using a 1% detergent solution (liquid soap and water) and then with distilled water in an ultrasonic cleaner for 15 minutes. After that, they were dried in the air, and immediately fixed in the sample holder inside the plasma chamber.(17)

Plasma treatment

Plasma was applied to ten specimens for each test of each group. Plasma treatment was carried out with the aid of a DC-glow discharge plasma system (locally manufactured at Ministry of Science and Technology, Iraq); the apparatus was equipped with a direct current (DC). The gas utilized was a combination of oxygen and argon at a ratio of 1:1. Bonding surfaces of the SBS specimens and the other test specimens were mounted on the center of the cathode surface at a right angle to the gas flow with a 4 cm distance. Gas pressure was kept constant at 4×10^{-2} mbar. The plasma was excited using a DC voltage supply operating up to 650 V and a maximum DC of 30 mA. A uniform glow could be seen directed to the samples. All of the specimens were exposed to plasma for 5 minutes. At the end of plasma exposure period, the chamber was kept locked for an additional 15 minutes to allow the gas to be evacuated; the specimens were then retrieved and isolated using a cling film.

Preparing Final SBS Samples

Two acrylic specimens were placed facing each other for each SBS test sample, creating a gap between them measuring (13 × 13 × 3 mm) width, length, and depth, respectively. The two specimens were taped together and then fully submerged in laboratory putty (Zetalabor, Zhermack, Italy) and left to set completely. To facilitate flasking, a flask custom-made to the size of the samples was constructed.

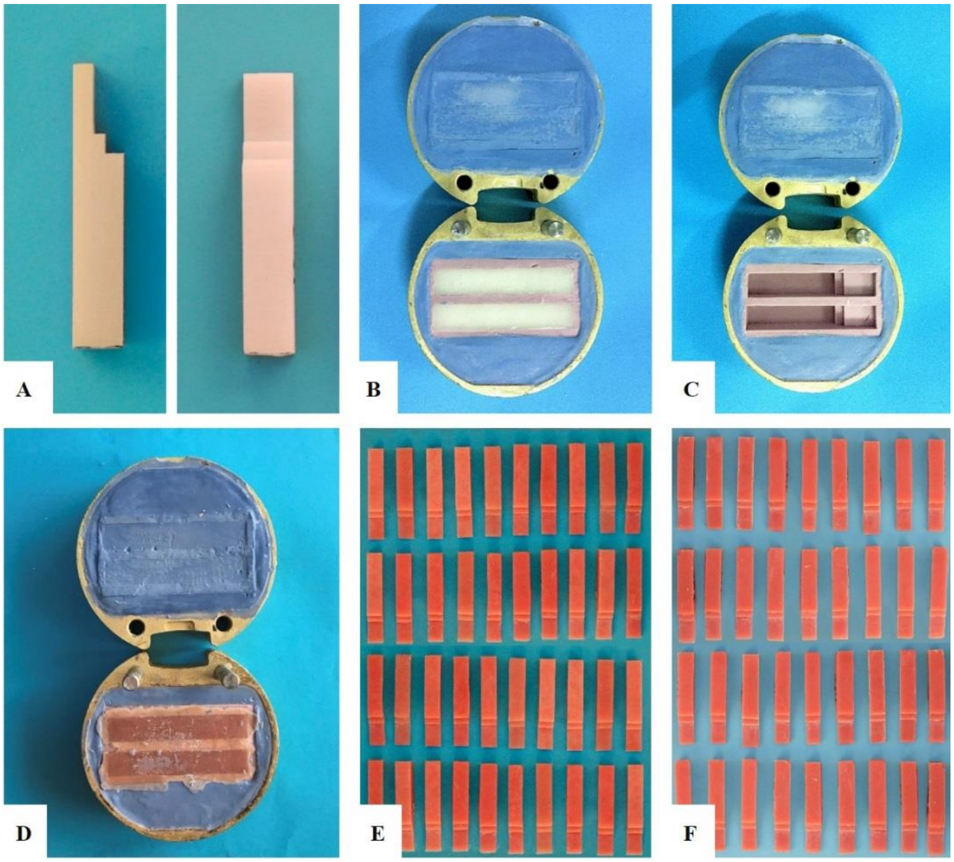


Figure 1: Preparing SBS acrylic specimens: A, Plastic block; B, Plastic blocks invested in silicone putty then in stone; C: Retrieving the blocks; D, Following curing of acrylic material; E, 20 pairs of conventional acrylic specimens; F, 20 pairs of high impact acrylic specimens.

The silicone containing the sample was then invested in stone inside the custom-made flask and allowed to set. Following that, the samples were extracted from the silicone to remove the tape and reinserted into the silicone.

Heat-cure soft liner (Vertex-soft, Netherlands) was prepared with a mixing ratio of 1.2 g: 1 ml. Once the material has reached its dough stage, with a metal mixing spatula, it was gently placed and condensed into the gap between each two blocks; the space was overfilled, the flask was then covered under pressure (1kg) and firmly screwed until edge-to-edge contact was achieved. The curing cycle was performed by heating water in a digital water bath up to 70°C for an hour and a half, then elevated up to 100°C for half an hour. Following curing, the flask was removed from the water bath and was left on the bench for 30 minutes to cool down, followed by 15 minutes of cooling under running tap water to ensure complete cooling.(18) The flask was opened and the samples were extracted and finished using a sharp blade to cut any excess and then stored in a container filled with distilled water.

Shear Bond Strength test

A universal testing machine (Laryree technology co.LTD, China) with a load cell capacity of 100 kg and a cross head speed of 0.5 mm/min was used to perform the shear bond strength test. Readings obtained from the machine represent the maximum load of failure. The machine's readings show the maximum load of failure. Bond strength was calculated by dividing the greatest load of failure by the cross-section area of each sample (13 × 13 mm = 169 mm^2), as recommended by ASTM specification D-638 (1986).(19)

Vickers Microhardness Testing

A Vickers microhardness tester (Brinell Rockwell Time Group Inc., China) was used to carry out the Vickers microhardness test. The square-base indenter was used to press a diamond indenter into the specimen surface and optically measure the diagonal length by a built-in scaled microscope. To determine the microhardness of all of the acrylic samples, they were loaded with a 30 g weight for 30 seconds. The final number was taken as the average microhardness of the indentation measured at four points for each sample.

Wettability Testing

Contact angle of the treated and untreated acrylic surfaces was measured using an optical tensiometer (TL 1000, Theta Lite, OneAttension, Biolin Scientific, Lichfield, UK). At room temperature, a drop of distilled water was used. In this procedure, a graduated syringe with hydrophobic needle deposits a drop; after 5 seconds the contact angle is captured with 60 images per second over 10 seconds. The images captured were analyzed using the special software of the microscope. This software drew a tangent automatically; the angle located at the three-phase-lines air/solid/liquid was calculated to give the contact angle value.

Chemical Surface Analysis (FTIR Analysis)

To gain a better understanding of the chemical surface changes that occurred on acrylic denture base materials following plasma treatment, the specimens' surfaces were investigated using FTIR analyzer (Fourier Transform Infra-Red Spectrophotometer, Bruker, Germany). Specimens with the exact measurements of wettability test specimens were prepared (20 × 15 × 2 mm).

Atomic Force Microscopy (AFM) Analysis

Atomic force microscopy was used to study the surface topography/morphology of untreated and plasma-treated acrylic polymer specimens. Specimens with the same measurements of wettability test specimens (20 × 15 × 2 mm) were prepared.

Statistical analysis

To conduct statistical analyses, statistical analysis software (IBM SPSS Statistics 26) was used. The pair-sample T-test and independent-samples T-test were used to analyze and compare the mean values. Statistical significance was considered for all comparisons when the p-value was less than 0.05.

RESULTS

SBS, microhardness, and contact angle findings were analyzed for the untreated and treated samples, for conventional acrylic, and high impact acrylic (Table 1). Comparative analysis for each group was individually performed using paired-samples T-test to determine the significance of plasma treatment effect. The results showed that SBS was significantly increased following plasma treatment ($p<0.001$) for acrylic of both types when compared to their respective control groups. The mean values of microhardness of regular and high impact acrylic had a non-significant change following plasma treatment ($p>0.05$). Table 1 also shows a significant decrease of contact angle mean values after plasma

Table 1: Comparative analyses of the mean values of SBS, microhardness and contact angle tests for conventional and high impact acrylic

	Test	Group	N	Mean	Standard deviation	Min.	Max.	T-test	df	Sig.
Conventional acrylic	SBS (N/mm2)	Control	10	0.825	0.0891036	0.722	0.976	-28.807	9	0.000
		Treated	10	1.540	0.0450955	1.459	1.600			
	Microhardness (HV)	Control	10	22.239	1.082707	20.46	24.18	0.075	9	0.942
		Treated	10	22.202	1.185962	20.44	24.26			
	Contact angle (°)	Control	10	74.264	1.59953	71.23	76.65	10.547	9	0.000
		Treated	10	66.363	1.76196	63.46	68.36			
High impact acrylic	SBS (N/mm2)	Control	10	1.359	0.0989452	1.191	1.475	-17.799	9	0.000
		Treated	10	2.180	0.1142171	1.982	2.335			
	Microhardness (HV)	Control	10	21.018	1.113701	19.37	23.30	1.099	9	0.300
		Treated	10	20.719	1.012384	19.43	22.97			
	Contact angle (°)	Control	10	69.765	1.90348	66.33	72.37	17.135	9	0.000
		Treated	10	58.151	1.75363	55.46	60.59			

treatment (p<0.001), indicating a significant increase in the wettability.

Independent-samples T-test was used for the comparison between the readings of SBS, microhardness and wettability of the two acrylic materials, conventional and high impact acrylics (Table 2).

Analyses of the chemical composition of the surfaces of each of the control and treated groups were performed using an FTIR analyzer (Figure 2). The two- and three-dimensional images obtained by the AFM analysis are shown in Figure 3. AFM analysis of plasma treated conventional and high impact acrylic surface has shown a more uniformly distributed granular film when compared to that of their untreated surface (Figure 4). Average surface roughness was increased following plasma treatment for both of the acrylic materials.

DISCUSSION

In practical usage, soft liner materials are often subject to tearing and shear stresses, resulting in their debonding from the denture base after a period of use.(4) Numerous ways of surface modification have been investigated in the literature; plasma treatment was found to greatly increase surface hydrophilicity without impairing the surface chemical characteristics.(12) As such, in the current research, the impact of oxygen and argon plasma treatment on SBS of soft liner to denture bases of two different materials was evaluated.

Table 2: Comparison of mean values of SBS, microhardness and contact angle tests between the two groups of acrylic materials

Test	Group	Acrylic Material	T-test	df	Sig.
SBS (N/mm2)	Control	Conventional	-12.685	18	0.000
		High impact			
	Treated	Conventional	-16.477	18	0.000
		High impact			
Microhardness (HV)	Control	Conventional	2.486	18	0.023
		High impact			
	Treated	Conventional	3.008	18	0.008
		High impact			
Contact angle (°)	Control	Conventional	5.722	18	0.000
		High impact			
	Treated	Conventional	10.446	18	0.000
		High impact			

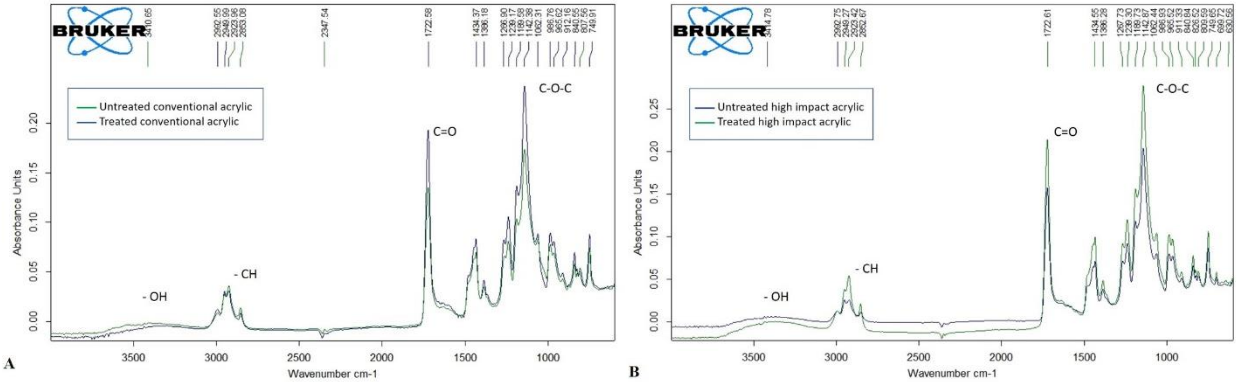
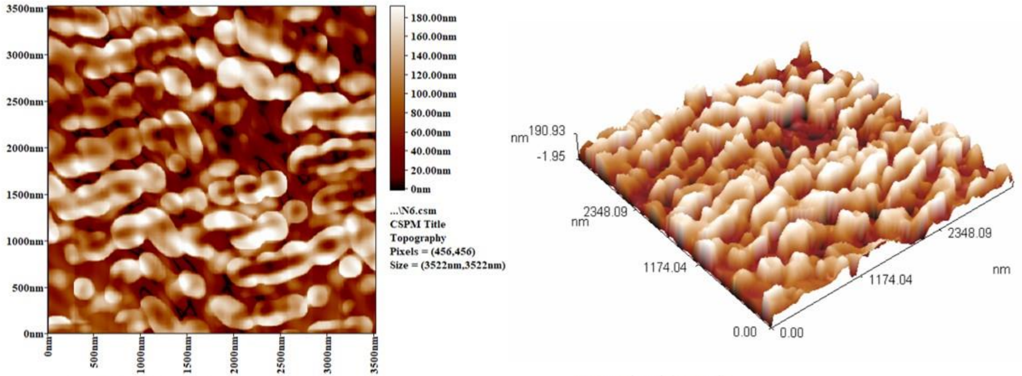
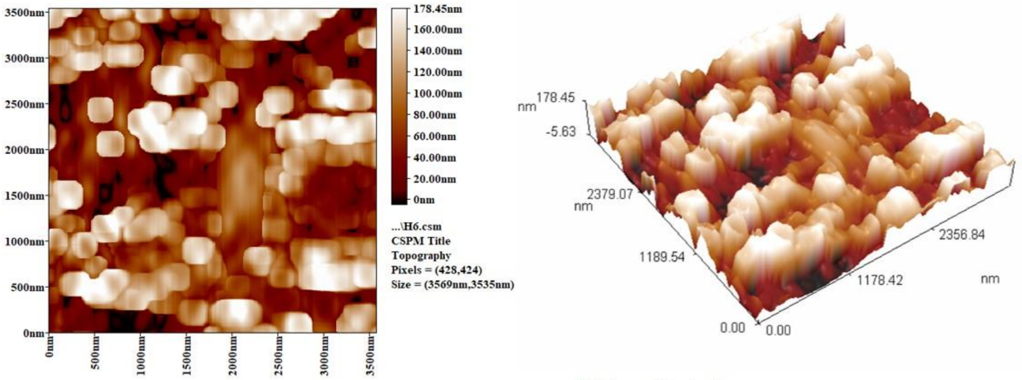


Figure 2: FTIR spectra before and after O_2 and Ar plasma treatment: A, Conventional acrylic; B, High impact acrylic.



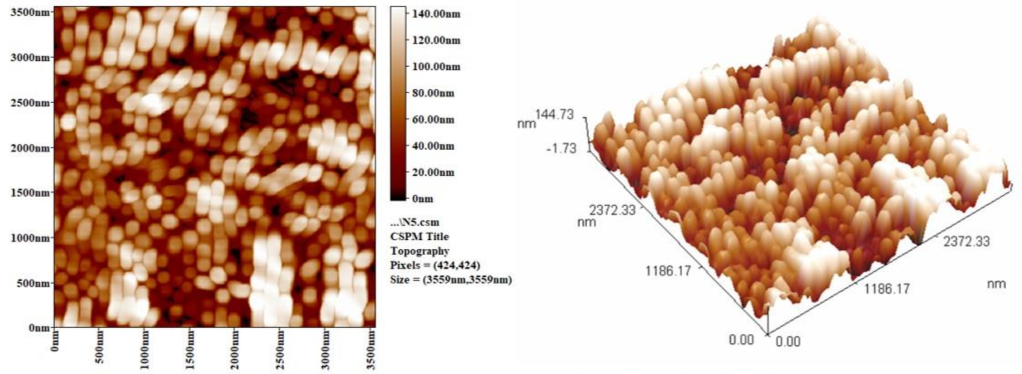
Conventional (control)

Conventional (control)



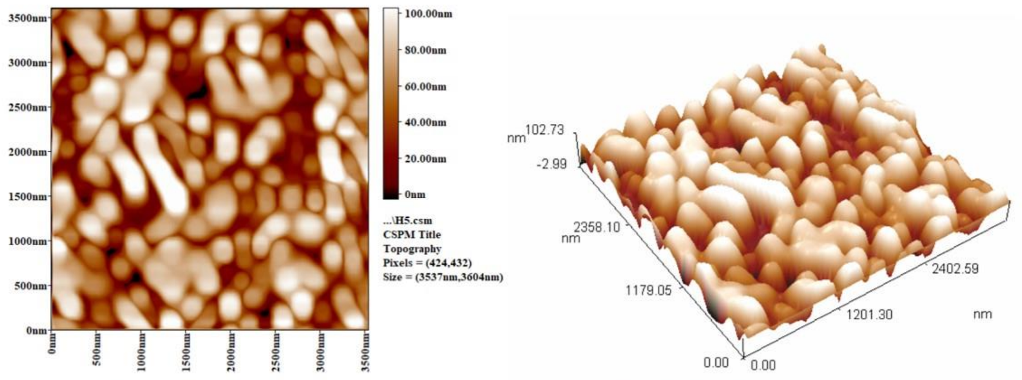
High impact (control)

High impact (control)



Conventional (plasma treated)

Conventional (plasma treated)



High impact (plasma treated)

High impact (plasma treated)

Figure 3: AFM two- and three-dimensional images of conventional and highs impact acrylic

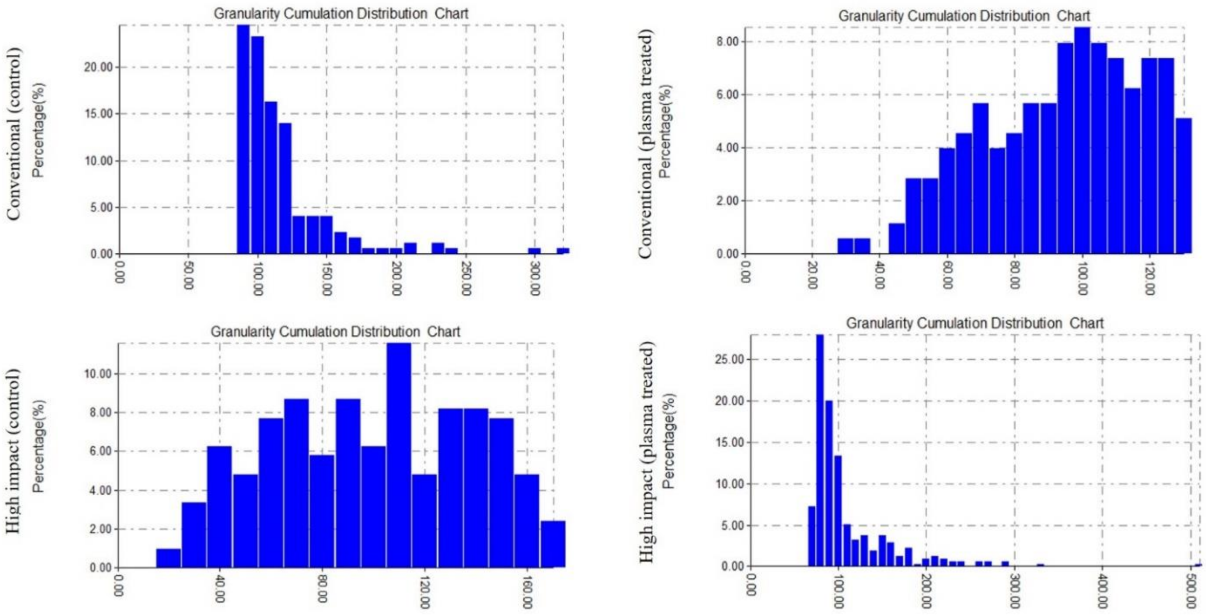


Figure 4: Bar charts illustrating granularity cumulation distribution of nanograins of the control and study groups of conventional and high impact acrylic

The null hypothesis that plasma treatment does not enhance SBS was rejected, as SBS was significantly improved following plasma treatment of both conventional heat-cure acrylic and high impact acrylic. This improvement may be attributed to the fact that oxygen gas in plasma treatment promotes an etching process by chemically removing particles from the surface material. Additionally, new functional groups such as O-H, C-O, and C=O are generated on the surface through the chemical oxidation reaction. It also enhances the surface energy, thereby allowing the soft liner to penetrate deeper into the irregularities,strengthening the bond between the two materials.(20) Even though argon is an inert gas and inert gas plasma treatments cannot generate any new reactive functional groups onto the polymer surface, treatment of polymers with inert gases could induce formation of free radicals on the acrylic surface via ultraviolet radiation and ion bombardment.(12) Furthermore, an inert gas, argon is combinedwith an active gas, such as oxygen in plasma, boosts oxygen functionality.(21) This was supported by measurements of contact angles, which showed that the contact angle of the treatment groups was substantially lower than that of the control groups. The additional polar functional groups that have been grafted onto the surface may have contributed to this reduction in contact angle. Functional groups break bonds on the surface, boosting surface energy and, as a result, wettability.(22) High impact acrylic showed significantly higher SBS than conventional acrylic in the control and treated groups. This may be due to the fact that high impact acrylic initially exhibits greater surface wettability, along with the presence of rubber particles in high impact acrylic. These particles are grafted into methyl methacrylate so as to bind them well to the acrylic heat polymerizing matrix. This coincides with the findings of Mittal et al. (2016), who found that the tensile bonding strength was greater between silicone-based soft liner and high impact acrylic than that with conventional acrylic.(6)

For both acrylic materials, plasma treatment showed a non-significant change on microhardness, which coincides with the conclusion of Dos Santos et al. (2016), that there was no effect on microhardness of acrylic denture resin when treated with plasma, making it an acceptable method for surface modification when compared to other treatments.(23) The results of FTIR analysis may provide an explanation for preservation of microhardness. No variation in peak positions was observed using FTIR surface chemical analysis, indicating that the hybridization state and electron distribution within the molecular bond have remained stable. This means that plasma treatment did not affect the chemical structure of these acrylic materials, which coincides with the FTIR results of Mustafa's study.(14) However, the peak intensities of the functional groups C=O, C-H, and C-O have increased, implying an increase in the amount (per unit volume) of these functional groups.(24)

AFM analysis of the control groups of both materials revealed that the surface granular film was distributed unevenly, when compared to that of the treated groups which showed a more even distribution along with a reduction in the average grain diameter and a rise in the number of grains. These observations indicate that plasma treatment removes the materials with lower attachment energy to the surface and diminishes the irregularities of the surface to bear polar groups.(25) These changes in surface morphology are suggested to be primarily produced by the surface being bombarded by high-energy ions present in the plasma, indicating that the phenomena of cross-linking has become enhanced.(26)

The test settings may not be representative of the actual clinical situation, as the test specimens comprised many adhesive surfaces, whereas dentures have just one adhesive surface in clinical practice. Thus, in vivo trials should be conducted as well. Meanwhile, the findings of this study may serve as a starting point for future research into novel materials and other factors affecting bond strength.

CONCLUSION

Within the confines of this study, the following conclusions were reached :

1-5-minutes oxygen and argon plasma treatment was successful in enhancing the shear bond strength of soft liner material to both of conventional acrylic and high impact acrylic denture materials.

2-High impact acrylic showed higher SBS initially and following plasma treatment when compared to conventional acrylic.

3-Plasma treatment had no significant effect on microhardness and chemical structure of the tested acrylic materials.

Conflict of interest: None.

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المستخلص

الخلفية: العيب الرئيسي لمواد التبطين اللينة هو أنها تنفصل عن قاعدة طقم الأسنان بعد فترة معينة من الاستخدام. لذلك، كان الغرض من هذا البحث هو تقييم تأثير معالجة بلازما الأوكسجين والأرجون على قوة الالتصاق القصّي للمبطنات اللينة لنوعين مختلفين من مواد قاعدة أطقم الأسنان: الأكريليك التقليدي المعالج حرارياً، والأكريليك عالي الصدمات.

طرائق العمل والمواد: تم تحضير اربعين قطعة من العينات لكل من الاكريليك التقليدي والاكريليك عالي الصدمات، لاختبار قوة الالتصاق القصّي. ثم تمت معالجة عشرة أزواج من العينات من كل مجموعة أكريليك بالبلازما لمدة 5 دقائق. بعد ذلك، تمت إضافة مادة التبطين الليّنة المعالجة بالحرارة إلى كل زوج من العينات. بعدها، تم تحليل قوة الالتصاق القصي باستخدام آلة اختبار عالمية. كما تم تحليل صلابة فيكرز الدقيقة وقابلية البلل وFTIR وAFM. تم تحليل البيانات إحصائيًا باستخدام T-test للعينات المزدوجة وللعينات المستقلة، ($\alpha = 0.05$).

النتائج: أظهرت النتائج زيادة ملحوظة في قوة الالتصاق القصّي بعد العلاج بالبلازما بأعلى متوسط قيمة (2.3355 نيوتن / مم ^{2}) لوحظ في عينات الأكريليك عالي الصدمات المعالجة بالبلازما. إلى جانب الارتفاع الكبير في قابلية البلل، ولم تتأثر الصلابة الدقيقة بفرقٍ ملحوظ بعد المعالجة بالبلازما.

الاستنتاج: في الختام، كانت المعالجة ببلازما الأوكسجين والأرجون فعالة بشكل كبير في تعزيز قوة الالتصاق القصي بين المبطنات اللينة ومواد الأكريليك.