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Effects of the Addition of Oxygen Plasma Treated Polyamide fibers on Some Properties of Room Temperature Vulcanized Maxillofacial Silicon Elastomers.

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ABSTRACT

In an attempt to improve the physical and mechanical properties of maxillofacial silicone variety of fillers were added, some showed improvement while others decline the properties. The aim of the study is to evaluate the effect of incorporation of oxygen plasma treated polyamide monofilament fibers with 2mm length to VST-50F (RTV) silicone elastomer on tear strength, tensile strength, elongation percentage, hardness and surface roughness. Based on pilot study plasma treated polyamide fibers with (1% and 1.5%) concentrations by weight were selected among other groups (0.5%, 1% and 1.5%) without oxygen plasma treatment and (0.5%) plasm treated fibers. Total 120 samples were prepared for the main study and divided into (4) groups, each one containing (30) samples according to the conducted tests, i.e. tear strength, tensile strength, hardness test, and surface roughness except for the elongation percentage test which was measured concurrently with the tensile strength test. Then, each group was subdivided into three subgroups according to the concentration of the fibers (0%, 1% and 1.5%) where n= 10. The samples were tested and the data were analyzed with a descriptive statistical analysis, one-way ANOVA, post-hoc tests (Bonferroni, Games-Howell). All plasma treated 2mm polyamide fibers with (1% and 1.5%) concentrations showed significant increase of tear strength, hardness and surface roughness. The 1.5% group showed the highest mean values among other groups. Tensile strength showed no significant change among all groups. Elongation at break showed significant decrease with increase concentrations. It was concluded that reinforcement of VST-50F maxillofacial silicone with 1.5% by weight (2mm) length plasma treated polyamide fibers can improve the mechanical properties

Keywords: Maxillofacial Silicone, Oxygen Plasma Treatment, Polyamide Fibers, Tear Strength

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INTRODUCTION

Cranio-maxillofacial injuries may occur as a result of explosive devices by terrorist attack, civilians and military people will be affected by these attacks and need to be prepared for rehabilitation (1).

Plastic surgery was preferred as a first approach of treatment but due to unfavorable condition rehabilitation with maxillofacial prosthesis is the best choice for improving patient esthetic and confidence and facilitating their return to society (2), silicon elastomers are considered the best choice of treatment for maxillofacial defect due to biocompatibility, easy manipulation, and realistic appearance (3).

Maxillofacial prosthesis are considers ideal when have physical and mechanical properties similar to the human part it replace and maintain these properties during service (4). Silicone elastomers have been used as maxillofacial prostheses since 1960s (3), Although its mechanical properties may not fulfill the ideal requirement so that additive, fillers and pigments were used to produce strong and elastic material to improve the physical, mechanical and clinical requirement (5)

Several polymers are widely applied within the body because they considered biocompatible and bio-stable materials (6).

Springer, Fleiner (7) Stated that polyamide fabrics have been chosen due to their mechanical stability and bioactivity. Polyamide monofilaments were used for manufacturing a non resorbable, long-lasting and stress-absorbent reinforcement for designing articular disc substitutes. Although the addition of Nylon (Nylon Art 2429- mesh type) to silicone elastomers A-2186 successfully reinforced and provide the silicone with better mechanical properties and augment strength especially for the delicate edge of maxillofacial prosthesis (5).

Plasma-surface modification (PSM) is an effective and economical surface treatment technique for many materials and of growing interests in biomedical engineering (8).

O2- Plasma treatment was founded to be able to control hydrophilicity and to produce various functional groups, leading to applications such as humidity sensors, enzyme immobilization and polymer bonding without the use of adhesives (9).

The aim of the study is to analyze the effect of addition of (2mm) length plasma treated polyamide fibers with (1% and 1.5%) by weight on tear strength, tensile strength, elongation at break, hardness and surface roughness of VST-50F maxillofacial silicone elastomer

MATERIALS AND METHODS

Tow concentrations of Polyamide (PA-6) fibers co-polymer (monofilament with diameter of 0.25mm and 2mm length) (Goodfellow, Cambridge limited, England) which treated with oxygen plasma and then were added to VST 50 F room temperature vulcanized silicone elastomer (Factor II Inc., Lakeside, AZ, USA)

The molds were fabricated according to test requirements using a laser engraving cutting machine (JL-1612, Jinan Link Manufacture & Trading Co., Ltd., China). Acrylic sheets with 2±0.05 mm and 6±0.05 mm thickness were fabricated which represent the depth of corresponding tests while 4±0.05 mm thickness was fabricated to make the bottom and the cover parts (10, 11)

A pilot study was conducted to compare the effect of polyamide fibers with and without oxygen plasma treatment by addition of (0.5%, 1% and 1.5%) for each type and using (0%) as a control group, tear strength and surface roughness tests were carried out, Results revealed that (1% and 1.5%) by weight oxygen plasma treated PA-6 fibers improve the mechanical properties of the silicone elastomer.
Surface modification of the fibers was performed by oxygen plasma treatment for (10) min (12), fibers were cut by scissors into average length of (15 cm) to be placed in the disc of plasma device that will be placed between the electrodes (Fig.1).

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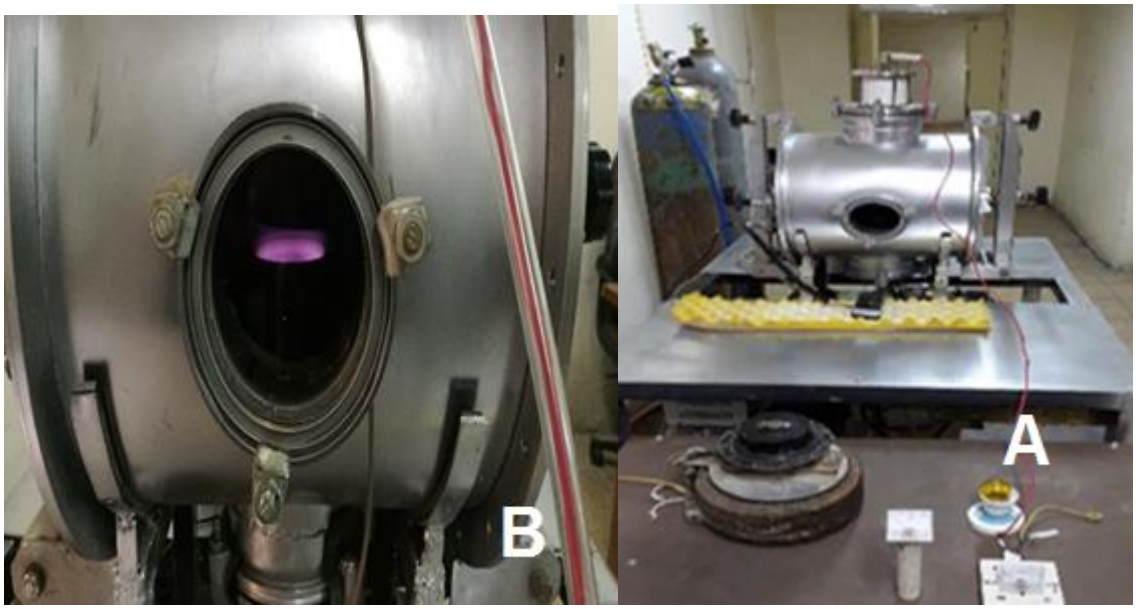

Figure (1): A. DC glow discharge system; B. Glow discharge during treatment

The VST-50F mixing ratio is 10:1 for the base and catalyst by weight according to the manufacturer instruction.for the control group the base was weighted using electronic digital balance then the catalyst added and mixed by a vacuum mixer (Multivac 3; Degussa, Germany) for 5 minutes at speed of 360 rpm and under vacuum of -10 bar (13).

Plasma treated polyamide fibers were cut by scissors into (2mm) length and then weighted using electronic digital balance followed by the addition of accurate weight of silicone base to prevent dispersion of the filler. The modified silicone was mixed by a vacuum mixer for 10 minutes; the vacuum was turned off for the first three minutes to avoid suction of the fibers and then turned on for the rest of the 7 minutes at 360 rpm speed and a vacuum value of -10 bar. The silicone cross linker was added to the silicone base or the modified silicone (base and PA-6 fibers) and mixed again in the vacuum mixer for 5 minutes to get a homogenous and free bubble mixture (Fig. 2) (11, 13).

Figure (2): vacuum mixing

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A separating medium was brushed inside the mold and left to dry then the silicone mixture was poured and the mold was closed with the aid of screws and G-clamps (10, 14). The silicone set for 2-3 hours and it should be cured at 75°F (24°C) and 50% relative humidity according to the manufacturer instructions (Fig.3).

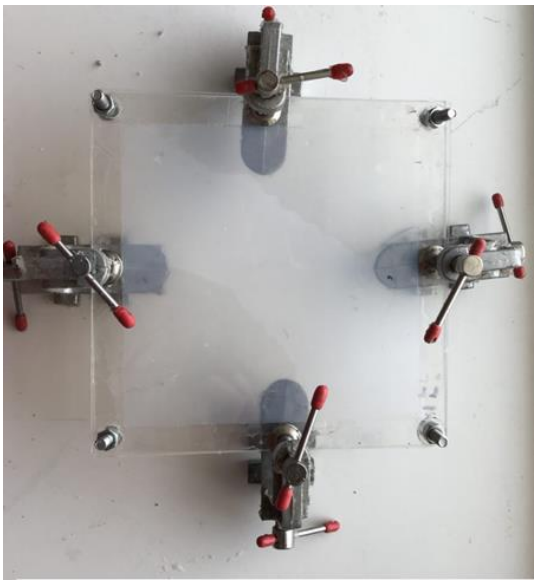

Figure (3): silicone poured and closing the mold

After the polymerization process was completed, the silicone sheet (15×15 cm) (11, 15) was separated from the mold cavity. The sheet was inspected for any abnormality or air bubbles then it was cut by a custom-made specimen cutting press (hydraulic jack of (3) tons capacity) (Lezaco, Syria) with the help of suitable cutting dies to ensure smooth cut surfaces (Fig. 4).

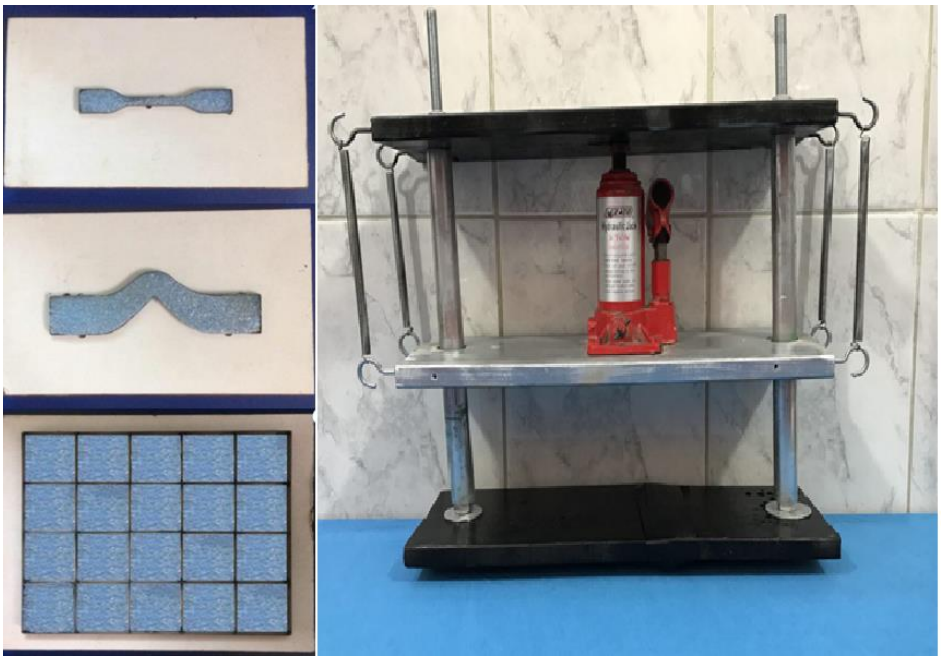

Figure (4): cutting dies and hydraulic jack

One hundred and twenty specimens of the modified silicone were fabricated and divided into 4 groups according to the conducted tests (tear strength test , tensile strength and elongation percentage tests,

Shore A hardness test and surface roughness test) with 30 specimens for each test except for the elongation percentage test which was calculated concurrently with the tensile strength test.

After cutting the specimens were inspected for internal defect or irregularities, the specimens were stored in a vaccine storage box (polar bag, china) for at least, 16 hours of favorable conditions before testing (14, 16)

Thirty specimens were prepared for tear strength test, all specimens were tested with a universal testing machine (WDW-20, Laryee Technology Co.Ltd., China) at 500 mm/min cross-head speed (17) .

According to ISO 37(18) , Type C which is an un-nicked specimen with a 90° angle on one side and with tab end specimens, 10 specimens were used as control group and the other 20 specimens after the addition of (1% and 1.5%) plasma treated polyamide fibers (Fig. 5). Specimens were mounted in a computerized universal testing machine with a 30±0.5 mm apart (17). The maximum load was calculated by the machine software then the tear strength according to the following equation:

$$\text{Tear strength} = F/D$$

Where:

F: The maximum force required for specimen to break (KN).
D: The median thickness of each specimen (m).

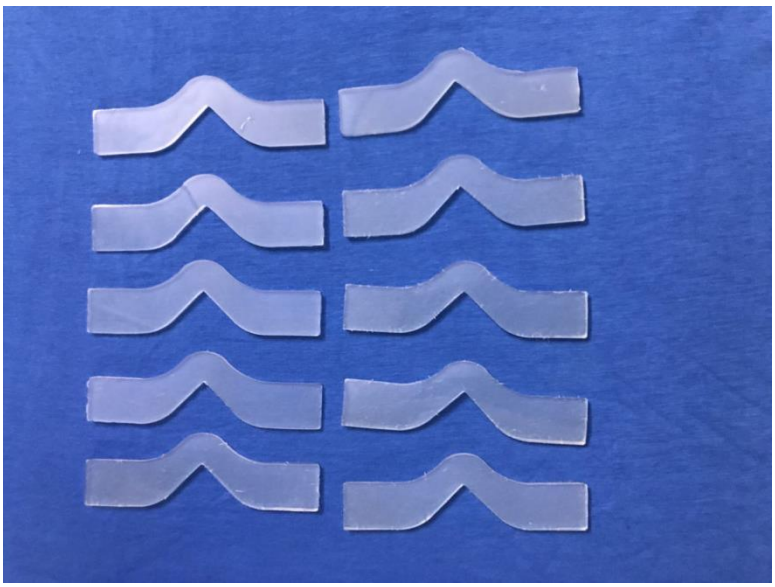

Figure (5): tear strength specimens

Thirty specimens were prepared for tensile strength and percentage elongation test, all specimens were tested with a universal testing machine at 500 mm/min cross-head speed (17) . According to ISO 37(18) Type 2 dumb-bell shape specimens were fabricated, 10 specimens were used as control group and the other 20 specimens after the addition of (1% and 1.5%) plasma treated polyamide fibers (Fig.6).Specimens were mounted in a computerized universal testing machine 25±0.5 mm apart(17) The tensile strength was calculated by the machine software according to the following equation:

$$\text{Tensile strength} = F/A$$

Where:

F: The maximum force recorded at break (N).
A: The original cross-sectional area of the specimen (mm2).

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At the same time the elongation percentage test was calculated with tensile strength test according to the following equation:

$$\text{Elongation at break} = [(L-L0)/L0]\times 100$$

Where:

L*o*: The original length (mm).
L: Extension at break (mm).

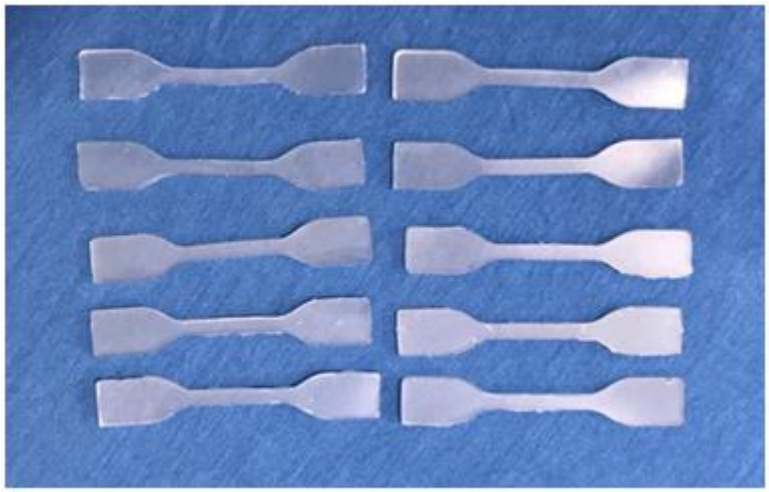

Figure (6): tensile strength and percentage elongation specimens

Sixty specimens of 25×25×6 mm for Shore A hardness and surface roughness tests were fabricated, 30 specimens for each test; 20 specimens were used as control group and the other 40 were tested after addition of (1% and 1.5%) plasma treated polyamide fibers, according to ISO 7619 (19)

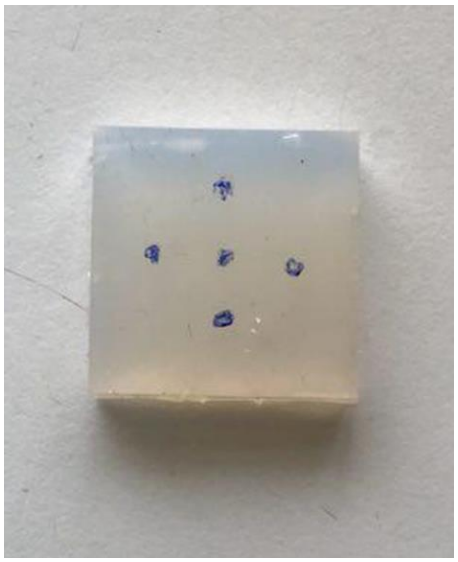

Figure (7): Hardness and surface roughness specimen

Shore A durometer with a blunt indenter of diameter of (1.25mm) was used for hardness test, it must be placed vertically over the surface of the specimen supported by a flat and rigid surface. Five points were marked with a (6 mm) distance between each other and the lateral margins of the test specimen; the average of these 5 readings was reported as the hardness value. Readings were taken after 1 second of stable contact over the specimen (13).

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The surface roughness test was carried out with the aid of a Portable digital roughness tester (Profilometer). The stylus of the device touches the surface of the specimen at three different points to obtain 3 readings. Then the mean value of the three readings was reported as roughness value (20)

Furthermore, SEM was performed on polyamide fibers before and after oxygen plasma treatment. VST-50F silicone elastomer was analyzed by both SEM and XRD before and after the addition of plasma treated PA-6 fibers.

RESULTS

SEM results of PA-6 fibers after oxygen plasma treatment that the surface of the fibers become rough which will increase its adhesion (Fig.8)

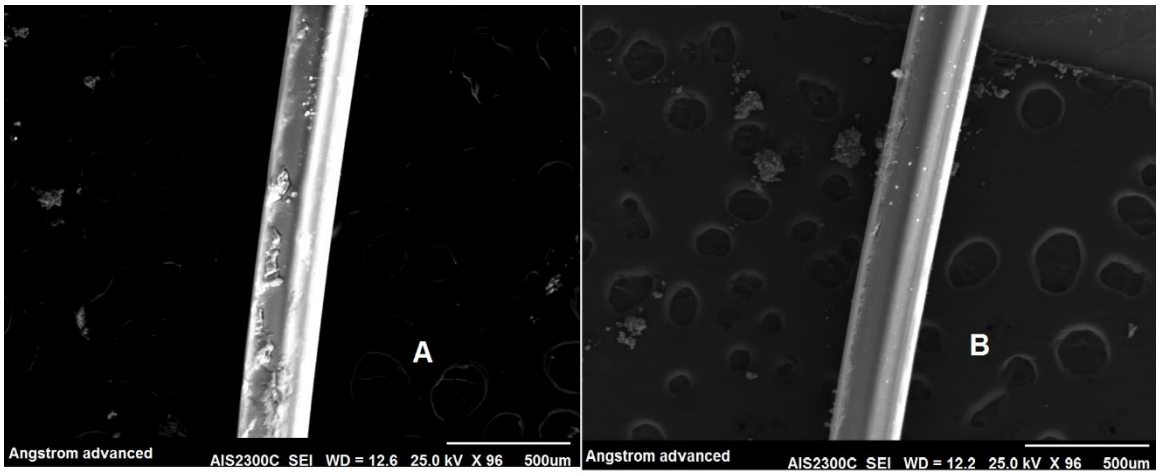

Figure (8): A. Oxygen plasma treated PA-6 fiber; B. untreated PA-6 fiber

SEM results among with XRD resutls of VST-50F silicone elastomer before and after addition of the fibers showed incorporation of the fibers with the matrix of the silicone Fig. (9 and 10) respectively.

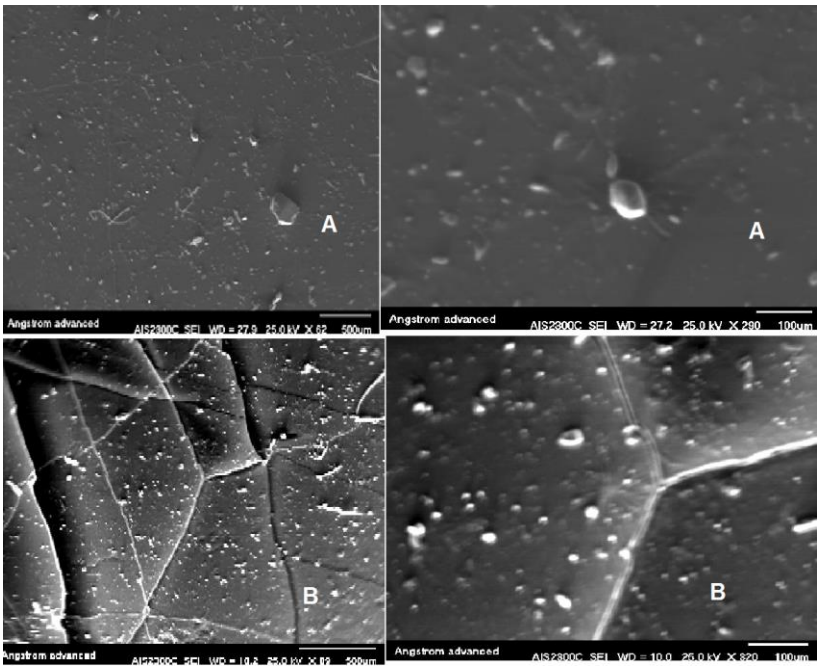

Figure (9): A. SEM results of VST-50F ; A without fibers, B with plasma treated PA-6 fibers

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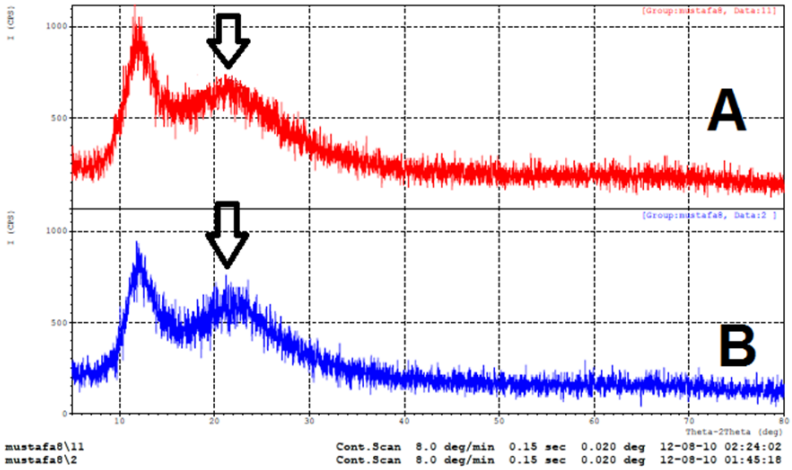

Figure (10): XRD results of VST-50F ; A without fibers, B with plasma treated PA-6 fibers

Tensile Strength Test

Results are listed in Table (1) that showed non-significant increase of tensile strength after addition of (1% and 1.5%) and a significant increase between the two concentrations

Table (1): Descriptive statistics, one way ANOVA test, Levene test, Bonferroni post hoc test for tensile strength test

	A Control	B 1% PA-6 fibers	C 1.5% PA-6 fibers	ANOVA F-test	P-Value	Levene test	Bonferroni		P-value	
N	10	10	10				A	B	0.280	NS
Mean	6.274	5.686	7.097	8.795	0.001	0.916	A	C	0.065	NS
SD	0.813	0.668	0.777		HS	NS	B	C	0.001	HS
Min	4.676	4.511	5.833							
Max	7.287	6.583	8.271							

Percentage elongation

Results are listed in Table (2) showed non-significant decrease of percentage elongation after addition of (1%) plasma treated PA-6 fibers ,highly significant decrease of percentage elongation after addition of (1.5%) plasma treated PA-6 fibers and a significant decrease between the two concentrations

Table (2): Descriptive statistics, one way ANOVA test, Levene test, Bonferroni post hoc test for percentage elongation test

	A Control	B 1% PA-6 fibers	C 1.5% PA-6 fibers	ANOVA F-test	P-Value	Levene test P-value	Bonferroni test		P-value	
N	10	10	10				A	B	1.000	NS
Mean	544.053	526.872	453.932	6.325	0.006	0.142	A	C	0.007	HS
SD	75.539	62.643	35.051		HS	NS	B	C	0.0035	S
Min	353.482	452.258	390.067							
Max	609.016	605.759	526.237							

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Tear strength

Results are listed in Table (3) showed highly significant increase of tear strength after the addition of (1% and 1.5%) plasma treated PA-6 fibers and a significant increases between both concentrations

Table (3): Descriptive statistics, one way ANOVA test, Levene test, Bonferroni post hoc test for tear strength test

	A Control	B 1% PA-6 fibers	C 1.5% PA-6 fibers	ANOVA F-test	P- Value	Levene test	Bonferroni		P-value	
N	10	10	10				A	B	0.000	HS
Mean	23.300	26.330	27.910	55.025	0.000	0.188	A	C	0.000	HS
SD	1.404	0.707	0.722		HS	NS	B	C	0.035	S
Min	21.500	25.200	26.500							
Max	26.000	27.400	28.700							

Shore A hardness test

Results are listed in Table (4) showed highly significant increase of hardness of VST-50F between all tested groups

Table (4): Descriptive statistics, one way ANOVA test, Levene test, Bonferroni post hoc test for shore A hardness test

	A Control	B 1% PA-6 fibers	C 1.5% PA-6 fibers	ANOVA F-test	P- Value	Levene test P-value	Bonferroni test		P-value	
N	10	10	10				A	B	0.009	HS
Mean	24.99	28.66	31.97	19.477	0.000	0.289	A	C	0.000	HS
SD	1.99691	2.06839	3.24210		HS	NS	B	C	0.019	HS
Min	21.50	26.00	25.50							
Max	28.40	32.10	36.30							

Surface roughness test

Results listed in Table (5) showed highly significant increase of surface roughness of VST-50F between all tested groups

Table (4): Descriptive statistics, one way ANOVA test, Levene test, Games-Howell post hoc test for surface roughness test

	A Control	B 1% PA-6 fibers	C 1.5% PA-6 fibers	ANOVA F-test	P-Value	Levene test P-value	Games-Howell test		P-value	
N	10	10	10				A	B	0.000	HS
Mean	0.352	0.423	0.437	449.183	0.000	0.003	A	C	0.000	HS
SD	0.002	0.008	0.009		HS	HS	B	C	0.003	HS
Min	0.350	0.412	0.422							
Max	0.355	0.432	0.452							

DISCUSSION

The mechanical and physical properties of the material determine the success of the prosthetic rehabilitation of the facial defect, variety of materials have been used for construction of facial prostheses unfortunately none of them fulfill all the ideal requirements for a satisfactory prosthesis **(21).**

Reinforcement by Fiber is depending on various variables including, fiber type, length, and form, and arrangement, percentages of fibers in the polymeric matrix and fiber matrix interaction and presence or absence of salination **(22)**

Decrease of tensile strength mean value when reinforced with 1% plasma treated PA-6 fibers is that the fibers may act as a flaw in the rubber matrix and cause early rupture **(23)**

Increase of tensile strength mean value when reinforced with 1.5% plasma treated PA-6 is that in short-fiber reinforced rubbers, the tensile strength was increased by the resistance of fibers for external loads because the space between fibers decreased with fiber aspect ratio and content. It is found that short-fiber incorporation has an important effect on tensile strength because of the acceleration in the crystallizing behavior of elastomers and this strain-induced crystallization will improve the tensile property **(24)**

The decrease in elongation percentage mean value after addition of 1.5% plasma treated PA-6 fibers may be due to that the fibers inhibit the orientation and flow of molecular chains which restrained the matrix of the material then the failure is initiated at multiple points and hence resulting in substantially lower elongation percentage values **(25, 26)**

The significant increase in tear strength when reinforced with plasma treated PA-6 fibers may be attributed to that the fibers bridged the tear and prevent or obstructed the propagating tear. When the propagating tear came in contact with these fibers, it may be arrested there or may branch there and proceed. Either of the processes may be the cause of the tear strength increase **(23, 27, 28).**also the fibers may act as a framework that support the matrix and increase the tear strength **(29)**

The increase in hardness mean value may be attributed to the variation of cross-link density with filler content, The addition of the fibers may decrease the distance between the crosslinks of the polymeric matrix thus the reduction of the softness of the material contributed to the polymer and filler interaction **(30)**

Finally the results reported increase of surface roughness which may be explained that the fibers were randomly distributed during sample preparation and may protrude from its surface. Random distribution in addition to fiber protrusion may attribute to increase of mean value of surface roughness **(26)**

CONCLUSION

Plasm treatment for PA-6 fibers is an efficient procedure to produce rough surface that improves its adhesion. The addition of plasm treated polyamide fibers can improve the mechanical properties of VST-50F

REFERENCES

[1] Covey DCF, James. Blast and fragment injuries of the musculoskeletal system. Orthopedics in Disasters: Orthopedic Injuries in Natural Disasters and Mass Casualty Events: Springer Berlin Heidelberg; 2016. p. 269-80

[2] Guiotti AM, Goiato MC, dos Santos DM. Evaluation of the Shore A hardness of silicone for facial prosthesis as to the effect of storage period and chemical disinfection. The Journal of craniofacial surgery. 2010;21(2):323-7.

[3] Goiato MC, Pesqueira AA, Moreno A, dos Santos DM, Haddad MF, Bannwart LC. Effects of pigment, disinfection, and accelerated aging on the hardness and deterioration of a facial silicone elastomer. Polymer Degradation and Stability. 2012;97(9):1577-80.

[4] Eleni PN, Krokida M, Polyzois G, Gettleman L, Bisharat GI. Effects of outdoor weathering on facial prosthetic elastomers. Odontology. 2011;99(1):68-76.

[5] Gunay Y, Kurtoglu C, Atay A, Karayazgan B, Gurbuz CC. Effect of tulle on the mechanical properties of a maxillofacial silicone elastomer. Dental materials journal. 2008;27(6):775-9.

[6] Ramakrishna S. HZ-M, Kumar G.V., Batchelor A.W., Mayer J. An Introduction to Biocomposites 1. 1st ed. London: Imperial College Press; 2004.

[7] Springer ING, Fleiner B, Jepsen S, Açil Y. Culture of cells gained from temporomandibular joint cartilage on non-absorbable scaffolds. Biomaterials. 2001;22(18):2569-77.

[8] Chu PK, Chen JY, Wang LP, Huang N. Plasma-surface modification of biomaterials. Materials Science and Engineering: R: Reports. 2002;36(5):143-206.
[9] Wu Z, Xanthopoulos N, Reymond F, Rossier JS, Girault HH. Polymer microchips bonded by O2-plasma activation. Electrophoresis. 2002;23(5):782-90.
[10] Atta Allah J, Muddhaffer M. Influence of Artificial Weathering on Some Properties of Nano Silicon Dioxide Incorporated into Maxillofacial Silicone. IJSR; 2017.
[11] Fatalla A, E AlSamaraay M, Jassim R. Effect of the Addition of Polyamide (Nylon 6) Micro-Particles on Some Mechanical Properties of RTV Maxillofacial Silicone Elastomer Before and After Artificial Aging2017. 1933-42 p.
[12] Jassim RK. Studying the properties of heat cure acrylic resin after reinforced with polyamide fibers treated with plasma (oxygen gas). Iraqi dental journal. 2015;37(3):83-9.
[13] S. Tukmachi M, Moudhaffer M. Ali M. Effect of Nano Silicon Dioxide Addition on Some Properties of Heat Vulcanized Maxillofacial Silicone Elastomer2017. 37-43 p.
[14] Fatalla A. Effect of Polyester Fiber Incorporation into RTV Maxillofacial Silicone Elastomer on Tear Strength, Tensile Strength, Surface Roughness and Shore 'A' Hardness: A P2018.
[15] Rai SY, Guttal SS. Effect of intrinsic pigmentation on the tear strength and water sorption of two commercially available silicone elastomers. The Journal of Indian Prosthodontic Society. 2013;13(1):30-5.
[16] dos Santos DM, Goiato MC, Sinhoreti MAC, Fernandes AÚR, do Prado Ribeiro P, de Carvalho Dekon SF. Color stability of polymers for facial prosthesis. Journal of Craniofacial Surgery. 2010;21(1):54-8.
[17] Zayed SM, Alshimy AM, Fahmy AE. Effect of surface treated silicon dioxide nanoparticles on some mechanical properties of maxillofacial silicone elastomer. International journal of biomaterials. 2014;2014.
[18] ISO-37. 37: 2011 Rubber, vulcanized or thermoplastic—Determination of tensile stress-strain properties. British Standards Institution: London, UK2011.
[19] ISO-7619–1. 7619–1: 2010 Rubber, vulcanized or thermoplastic—Determination of indentation hardness. Part 1: Durometer method (Shore hardness). British Standards Institution: London, UK. 2010.
[20] Jaber AN, Jassim RK, Moudhafar M, Fatihallah AA. Effect of Polyester Fiber Incorporation into RTV Maxillofacial Silicone Elastomer on Tear Strength, Tensile Strength, Surface Roughness and Shore â Aâ Hardness: A Pilot Study. International Journal of Medical Research & Health Sciences. 2018;7(2):92-101.
[21] Alqutaibi AY. Materials of facial prosthesis: History and advance. Int J Contemp Dent Med Rev. 2015;2015:4.
[22] Hachim TM, Abullah ZS, Alausi YT. Evaluation of the effect of addition of polyester fiber on some mechanical properties of heat cure acrylic resin. Journal of baghdad college of dentistry. 2013;25(special issue 1):23-9.
[23] Kumar RP, Thomas S. Short fibre elastomer composites: effect of fibre length, orientation, loading and bonding agent. Bulletin of Materials Science. 1995;18(8):1021-9.
[24] Lee D, Ryu S. The Influence of Fiber Aspect Ratio on the Tensile and Tear Properties of Short-Fiber Reinforced Rubber. ICCM12, Paris. 1999.
[25] Sreeja T, Kutty S. Studies on acrylonitrile butadiene rubber-short nylon fiber composites. Journal of Elastomers & Plastics. 2002;34(2):157-69.
[26] Leny M, Narayanankutty SK. Development of Elastomeric Hybrid Composite Based on Synthesised Manosilica and Short Nylon Fiber: Cochin University of Science and Technology; 2009.
[27] Murty V, De S. Short-fiber-reinforced styrene–butadiene rubber composites. Journal of applied polymer science. 1984;29(4):1355-68.
[28] Sreeja T, Narayanankutty SK. Studies on short nylon fiber-reclaimed rubber/elastomer composites: Cochin University of Science & Technology; 2001.
[29] Gunay Y, Kurtoglu C, Atay A, Karayazgan B, Gurbuz CC. Effect of tulle on the mechanical properties of a maxillofacial silicone elastomer. Dental materials journal. 2008;27(6):775-9.
[30] Liu Q, Shao L, Xiang H, Zhen D, Zhao N, Yang S, et al. Biomechanical characterization of a low density silicone elastomer filled with hollow microspheres for maxillofacial prostheses. Journal of Biomaterials Science, Polymer Edition. 2015;24(11):1378-90.