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Comparison of some mechanical and physical properties of heat cured soft denture liner after the addition of polyamide nylon-6 micro particle

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Abstract

The longevity of soft denture liners depend on it is properties, that is why, defects in the material properties may reduce the service life of soft lining materials and necessitate its replacement. The aim of this study was to evaluate the effect of adding different concentrations of Polyamide-6 (Nylon-6) on the peel bond strength, tensile strength and wet ability of acrylic base heat cure soft liner. 120 samples were fabricated by the addition of 0%, 1%, 2% and 3% by weight PA-6 micro-particles powder to acrylic soft liner. The study samples were divided into three(3) groups according to test to be performed, each group containing 40 samples, 10 specimens for each concentration of PA-6 micro particle (0%,1%,2% and3%). The data were analyzed with a descriptive statistical analysis, one-way ANOVA, and Bonferroni multiple comparison test. The mean values of peel bond and tensile strength of 1% PA-6 reinforcement group increased significantly when compared to control group and higher than the mean values of other experimental group. On other hand, there was non-significant elevation in wettability property after addition of different percentage of PA-6 micro particle powder(1%,2% and 3%). The 1% PA-6 reinforcement improved peel bond as well as tensile strength with higher value among other percentages (2% and 3%) , also improved the wettability property of acrylic soft liner.

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Introduction

Permanent soft denture liners have great value in dentistry because of their viscoelastic properties , these materials serve as shock absorbers that minimize and distribute the stress on denture-bearing tissues, in addition to improve the intaglio denture surface (Sertgöz *et al.,* 2002). These liners clinically beneficial for treatment patient with atrophic ridge, bruxism, xerostomia and bone undercuts.

However, the use of soft lining materials have several disadvantages, one of the major drawback is lack of durable bond to denture base (Pisani *et al.,* 2009). Debonding between soft lining materials and denture base results in potential surface for microbial growth, plaque and calculus formation. So, frequent evaluations and periodic replacement of soft liners are needed (Kawano *et al.,* 1992). Different methods such as peel bond test have been applied to measure the bond strength (Wood *et al.,* 1993). The peel bond strength test simulates the relining procedure more accurately, with an even distribution of force over the bonding area (Waters and Jagger 1999). Other properties like tensile property are fundamental and considered as a general guide to the quality of rubber materials. Also wettability of denture soft-lining materials are essential , as they give an information about the ease of saliva to spread over their surfaces (Wright 1980). Wetting measurements also indicate the degree of denture retention (Monsenego *et al.,* 1989).

The purpose of the present study was to evaluate the effect of adding different weight percentage (1%, 2%, 3%) of Polyamide-6 (Nylon-6) on the peel bond, tensile strength and wettability properties of acrylic based heat cure soft liners.

Materials and methods

This study investigated the peel bond , tensile strength and wettability property of acrylic based heat cure soft liner (Vertex™Soft, Vertex-Dental, Netherlands) before and after the addition of polyamide-6 (Nylon-6) micro-particles powder (average particle size 15-20 micron) (Goodfellow, Cambridge Limited, England) in different percentages (1%,2%,3% by weight). A total of one hundred twenty specimens were prepared and divided into three groups according to the test to be performed each test has forty specimens, ten for each concentration (0%,1%,2% and 3%).

Peel bond strength

The preparation of the peel bond strength test specimens was made according to ASTM D903-93 (ASTM. D903-93). However, the size of the specimens given in this specification is very long, thus half of length and width was considered to be sufficient for testing (Demir *et al.,* 2011). Rectangular stainless steel plates with dimensions of 100 x 10 x 2 mm for PMMA, and 150 x 10 x 2 mm for soft liners holes inside them were fabricated.

Special stainless steel flask was designed by (Auto CAD 2015) computer software according to the test specimen's specifications. It was fabricated by wire cut device (Figure 1).

The first step in specimen preparation include packing of heat cure acrylic resin (Sanchez-Aliaga *et al.,* 2016), this material was proportioned and mixed according to the manufacturer's instructions(P/L 2.3g powder to 1 ml of liquid) then inserted into the empty space for acrylic in the flask .

The flask was closed and placed under hydraulic press with slow application of pressure to allow even flow of soft liner dough until reach (100 MPa) and left under this load for 5 minutes (Yassir 2017). After that, the specimens were immersed in water path for 20 min according to the manufacturer's instructions. After polymerization, the flasks were kept for bench cooling for 30 min followed by cooling under running water for 15 min (Sanchez-Aliaga *et al.,* 2016).

The acrylic strips were deflasked and trimmed away. The surfaces of acrylic that bonded with soft liners were smoothed using 240-grit silicone carbide paper, cleaned, and dried. Then the acrylic specimens were reflasked (Figure 2).

Before packing the heat cure soft liner a part of the acrylic specimen surface of all specimens was covered with a piece of tinfoil to ensure that only 70mm length of the lining material was bonded and the remaining length was unattached (Demir *et al.,* 2011). The proportioning and mixing of the soft liner powder and liquid followed the manufacturer's instruction. P/L ratio (1.2g /1 ml).For experimental group, the quantities of polyamide powder according to percentage of 1%, 2% and 3% by wt. PA-6 were first weighted in the mixing bowl over the electronic scale then added to the liquid of soft liner and mixed with sonication apparatus for 20 second (Muttagi and Subramanya 2017). The powder of soft liner after subtraction the weight of polyamide (to keep the same P/L ratio according to the manufacturer's instruction) were added and mixed , the mixture then covered and left for 15 min with 23±1°C according to manufacturer's instruction. The soft lining material was inserted into the hollow space in the plate designed for soft liner. This assembly was covered with another plate 5mm thick .The flask was placed under hydraulic press with slow application of pressure to allow even flow of soft liner dough until reach (100MPa) and left for 5 minutes (Yassir 2017). The excess material was removed. The flask then immersed in water path with average temperature 20° °C then increases the temperature to 70° C for 90 min. After that the temperature was increased to reach 100° C for the next 30 min. for curing soft liner according to manufacturer's instructions. After polymerization of soft liner, the flasks were bench cooled as done before with acrylic specimen, the flask then opened. The specimens were removed from the flask and any flash was trimmed with a sharp blade or scissor.

The peel bond strength test was analyzed according to ASTM D903-93 (ASTM. D903-93) in a universal testing machine at an angle of 180° and speed of 152 mm/min. The non-relined portion of the heat-cured acrylic resin was clamped on the upper clutch of the equipment while the free portion of soft lining material was folded and fixed in the lower clamp of the equipment (grip about 25mm of soft liner), Maintain the specimen during test approximately in the plane of clamps.

This done by holding the specimen against an alignment plate as in (Figure 4).After the specimens were tested and removed from the testing device, the nature of the bonding failure was evaluated by naked eye, and categorized as cohesive, adhesive or mixed. Cohesive failure refers to tearing within the soft liner material, adhesive failure refers to total separation at the interface between the soft liner and acrylic resin, and mixed failure refers to both (Demir *et al.,* 2011).

The peel bond strength was calculated by using the following equation where the peeling angle was considered 180°.

Peel strength = average load / width of the sample (Kutay 1994)

Tensile strength test

Samples were made by using wooden model with type 2 dumb-bell shape that fabricated according to ISO 37:2011 specifications (ISO 37. 2011) (Figure 5) which were invested in stone to form stone mould(Figure 6) . The soft lining material was mixed, packed and cured as instructed by manufacturer, and as for experimental samples, PA-6 was added to the liner liquid and mixed with sonicater (Muttagi and Subramanya 2017). The samples then finished, polished and ready for testing.

The specimens were tested using Instron testing machine (Instron 1195, England) at cross head speed equal to (500mm/min) (Waters and Jagger 1999). The tensile strength was calculated from the maximum stretching force at break divided by the original cross sectional area of the narrow portion of the specimen (width × thickness) using the following formula:

$T = F / A$

Where:

F: The maximum force recorded at break (N).

A: The original cross-sectional area of the specimen (mm^2)

Wettability test

To evaluate the wettability of soft denture lining material, Rectangular shaped specimens were prepared with dimensions of (30*10*1) length, width, depth respectively (Alcibiades *et al.,* 2001). Static sessile drop method was utilized in this study. A digital microscope (Dino-lite digital microscope pro - Taiwan) was used. This device was applied to take a magnified pictures with (45X magnification) of the profile of the specimens with a drop of liquid on its surface, the size of the drop (40µl) was standardized with the use of micropipette then the contact angles were determined by analyzing the picture in the specialize software (Dino Capture) of the digital microscope by drawing a tangent. The angle between the baseline of the drop and the tangent at the three-phase-line solid/liquid/air was measured (Al-Shaikhli and Khamas 2012) (Figure7).

Results

Figure 8 shows the SEM analysis results indicate some degree of the PA-6 micro fillers agglomeration into the soft liner matrix.

Peel bond test

The mean values of peel bond strength of acrylic base heat cure soft liner before and after addition 1%,2% and 3% PA-6 fillers are present in (Table 1) The experimental group (1% by wt. PA-6) showed highest mean value ,while the lowest mean value was found for the control group. Descriptive statistics, One-way analysis of variance (ANOVA) and Bonferroni multiple comparison test of the peel bond strength values are presented in(Tables 1and 2). The results of peel bond strength test indicated highly significant difference (p≤0.01) between all tested groups (Table 1).

Table 1. Descriptive statistics and ONE -way Analysis of variance (ANOVA) for peel bond strength test.

groups	N	Mean	Std. Deviation	Min	Max	Anova F-test	P-value	Sig.
control	10	2.03	0.442	1.30	2.90			
1%	10	4.59	0.525	3.80	5.30			
2%	10	3.81	0.404	3.30	4.50	51.428	0.000	HS
3%	10	3.39	0.508	2.50	4.10			

Table 2. Bonferroni multiple comparison test of the peel bond strength.

Compared study groups	Mean Difference (I-J)	P-value.	Sig.
control - 1%	-2.56	0.000	HS
control -2%	-1.36	0.000	HS
control - 3%	-1.78	0.000	HS
1% - 2%	1.20	0.000	HS
1% - 3%	0.78	0.004	HS
2% - 3%	-0.42	0.328	NS

Tensile strength

The mean values of tensile strength of acrylic base heat cure soft liner before and after addition 1%,2% and 3% PA-6 fillers are present in (Table 3) The experimental group (1% by wt. PA-6) showed highest mean value ,while the lowest mean value was found for experimental control(3% by wt. PA-6). Descriptive statistics, One-way analysis of variance (ANOVA) and Bonferroni multiple comparison test of the tensile strength values are presented in(Tables 3and 4). The results of peel bond strength test indicated highly significant difference (p≤0.01) between all tested groups (Table 4).

Wettability test

The mean values of static contact angle for distilled water on acrylic soft liner before and after addition 1%,2% and 3% PA-6 fillers are present in (Table 5)

The control group showed highest mean value ,while the lowest mean value was found for experimental control(3% by wt. PA-6). Descriptive statistics and One-way analysis of variance (ANOVA) of static contact angle values are presented in(Tables 5). The results of wettability test indicated non-significant difference ($p \geq 0.05$) between all tested groups (Table5).

Table 3. Descriptive statistics and ONE -way Analysis of variance (ANOVA) for tensile strength test.

groups	N	Mean	Std. Deviation	Min	Max	Anova F-test	P-value	Sig.
control	10	3.00	0.408	2.30	3.60			
1%	10	3.41	0.488	2.70	4.20			
2%	10	3.26	0.365	2.60	3.90			
3%	10	2.71	0.310	2.30	3.20	5.950	0.002	HS

Table 4. Bonferroni multiple comparison test of the tensile strength.

Compared study groups	Mean Difference (I-J)	P-value.	Sig.
Control - 1%	-0.41	0.164	NS
Control -2%	-0.26	0.921	NS
Control -3%	-0.29	0.675	NS
1% - 2%	0.15	1.000	NS
1% - 3%	0.70	0.002	HS
2% -3%	0.55	0.023	S

Discussion

Peel bond analysis:

Peel bond testing is considered as the best model of the clinical environment to test soft denture liners concerning the bond failure of soft lining materials (Mutluay *et al.,* 2007). The result of this study revealed that there was significant increase in peel bond strength of experimental groups with various concentrations (1%, 2% and 3% by wt. PA-6 micro-fillers) in comparison with control group, with highest value for 1% by wt. PA-6 group and the lowest value for 3% by wt. PA-6 group. Experimental groups exhibited a high level of elastic deformation before peeling started and that indicated a more realistic bond strength and more elastic property for these groups.

Table 5. Descriptive statistics and ONE -way Analysis of variance (ANOVA) for static contact angle (distilled water).

Groups	N	Mean	Std. Deviation	Min	Max	Anova F-test	P-value	Sig.
Control	10	50.2	6.069	40	59			
1%	10	48.5	9.675	33	61	1.564	0.215	NS
2%	10	45.1	5.195	40	56			
3%	10	43.6	8.809	29	61			

The elevated level of elastic deformation values in experimental specimens are due to the addition of PA-6 micro-fillers to soft liner which reduce the degree of cross-linking as it penetrate inter macromolecular chains of polymer, this can be attributed to the reduced degree of cross-linking leads to increase in segmental mobility of polymeric chains which support the theory supposing that the high degrees of cross-linking resulting in large localized stress concentrations, so the material will break before the extension is sufficient for crystallization, where partial alignment of molecular chains occur, which prevents early breaking. The polymers, generally can crystalize when cooling from melt or mechanical stretching. The strength of the micro-filler PA-6/polymer bonding also has an influence on peel

bond property. Stronger filler/polymer bond increases the values of this property (Waters and Jagger 1999).

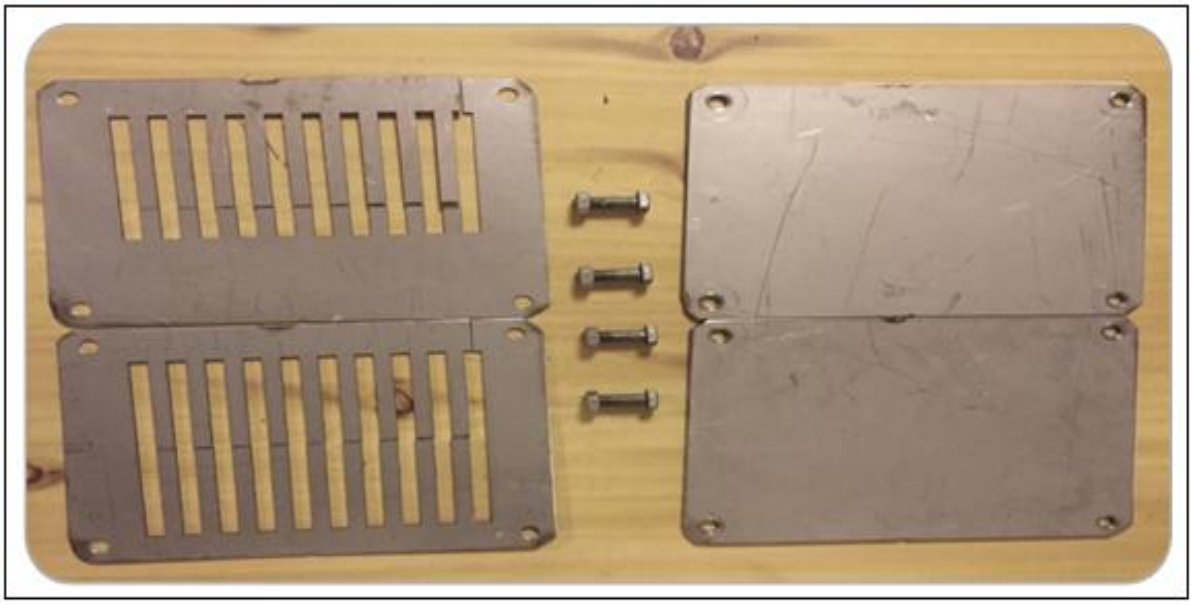

Fig. 1. Prepared plates for peel bond strength specimens.

The experimental groups showed a tendency to fail adhesively in 10% of specimens (only 3 specimens of experimental groups) after the addition of the PA-6 micro-filler powder, this may be related to the bonding surface swelling due to the absorption of water by the soft denture liner (because of hydrophilic action of polyamide) and stress may increase in the interface between soft denture liners and denture base acrylic resin leading to adhesive failure rather than cohesive failure.

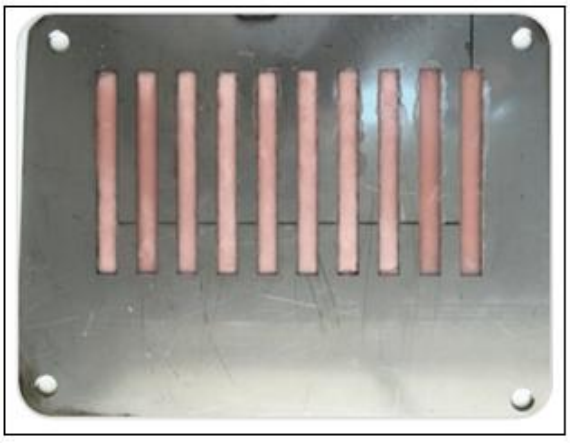

Fig. 2. Reflasked acrylic specimens.

In controversy, cohesive Failure in control group was predominant, due to its poor tear resistance.

Fig. 3. Peel bond strength test specimens ready for testing.

The bond between acrylic based soft liner and PMMA resin is obtained by interpenetrating polymer network formation which is one of the causes of cohesive failures (Oguz *et al.,* 2007).

The present results cannot be compared directly with previous studies because of variations in techniques, sample sizes, conditioning of samples, filler types and speed of separation. However, these results generally agreed with that has been noted previously when peel bond between soft liner and denture base tested by Waters and Jagger (Waters and Jagger 1999).

Fig. 4. Peel bond specimen under testing.

Even that, all specimens of control group would rupture at the interface (cohesive failure) but with forces below that needed to cause adhesive or cohesive failure of experimental group with various concentrations of PA-6 micro-fillers.

As noticed in the result of peel bond strength test, there were gradual reduction in peel strength as the PA-6 micro-fillers concentration increased however, they remained higher than control group that may be due to excess PA-6 micro-fillers would fill the intra macromolecular chains space, in addition the inter macromolecular chains space lead to separation of polymer chains and weak force between them cause reduction in peel property.

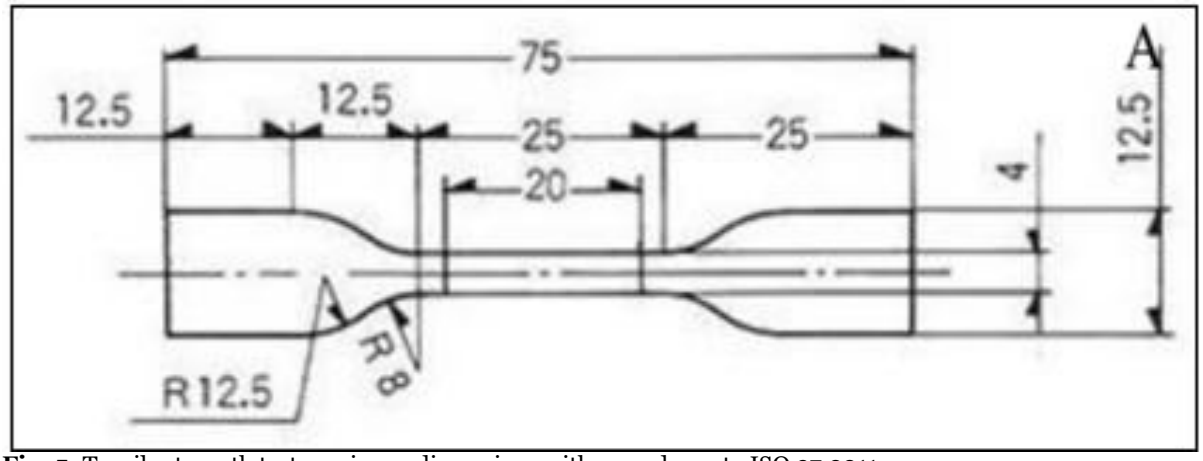

Fig. 5. Tensile strength test specimen, dimensions with accordance to ISO 37:2011.

Also reduction in peel bond strength may be due to aggregation of PA-6 (as noticed by SEM results) because of higher surface energy, this aggregation caused micro fracture that weakened the polymer structure as the percentage increased.

Tensile strength analysis

Among the several preferable mechanical properties of soft lining material, high tensile strength is of great importance to final prosthesis (Fatihallah and Alsamaraay 2017).

The result of this study revealed that significant increase in mean values of experimental groups by using concentration of 1% and 2% by wt., however the highest increase was noticed in 1% by wt. concentration; unfortunately there was decrease in tensile strength in 3% by wt. concentration in comparison with control group.

Fig. 6. Mold preparation for tensile strength test specimens.

The superior tensile strength shown by 1% and 2% by wt. experimental specimens in comparison with control specimens can also be explained in the same way mentioned in peel test.

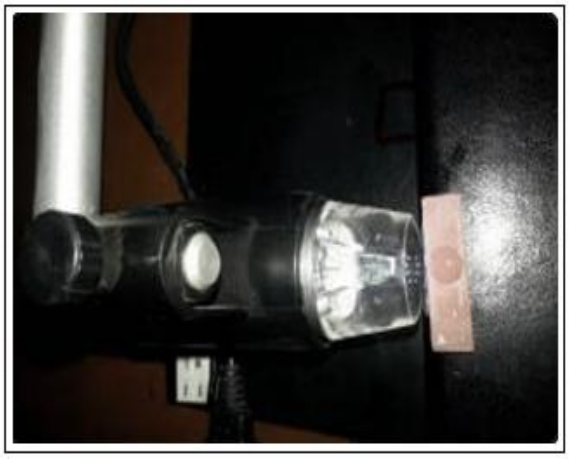

Fig. 7. The Dino-Lite and specimen during testing.

The results were agreed with the results of Waters and Jagger in 1999. Similarly AL-Samaraay in 2017 reported an increasing in tensile strength values after adding PA-6 to maxillofacial silicone elastomeric materials. At 3% by wt. polyamide micro-fillers concentration there was reduction in tensile strength below the control group, this may be due to excess PA-6 micro-fillers would fill the intra and inter macromolecular chains space that lead to separation of PMMA chain and weak force between them cause reduction in tensile property.

Also reduction in tensile strength at 3% by wt. concentration may be due to aggregation of PA-6 because of higher surface energy; this aggregation caused micro fracture that weakened the polymer structure at this percentage.

Another explanation, the higher concentration of polyamide 3% interfered with formation of the polymeric matrix, reducing the tensile strength of soft liners.

Wettability analysis

The direct relation between denture retention and wettability property make this feature of great importance.

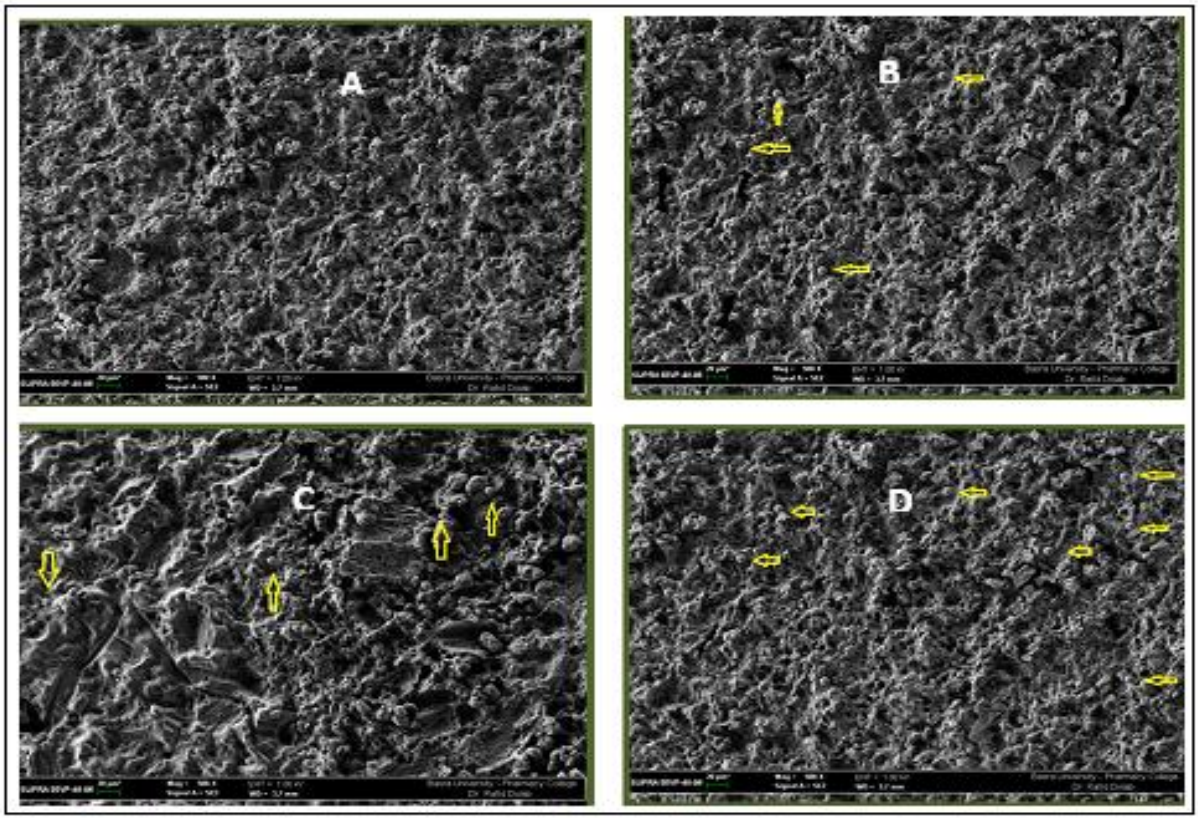

Fig. 8. SEM of acrylic soft liner. A: before the addition of PA-6 fillers; B, after the addition of 1% PA-6 fillers; C: after the addition of 2% PA-6 fillers and D: after the addition of 3% PA-6 fillers (Arrow pointed to PA-6 particle).

The contact angle measurement method is probably the most definitive way to determine the hydrophobicity of material surfaces. Low water contact angle values indicate a hydrophilic surface with high wettability property, whereas high water contact angle are indicative of a hydrophobic surface with low wettability property (Al–Nema 2011). A non-significant elevation in the wettability property in experimental groups of various concentrations (1%, 2%, and 3% by wt. PA-6 micro-fillers) as compared with control group was observed in the present work. This is related to the addition of polyamide micro-fillers which was hydrophilic material and also this material will increase roughness of soft liner, so rise the wettability. The results agreed with Muttagi and Subramanya (2016).

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