Effect of Thermocycling on Surface Roughness and Shear Bond Strength of Acrylic Soft Liner to the Surface of Thermoplastic Acrylic Treated with Ethyl Acetate

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Abstract

Objective: To enhance bonding strength between thermoplastic denture base and acrylic soft liner through ethyl acetate surface treatment.

Materials and Methods: Modifications of thermoplastic acrylic denture base surface were investigated with SEM. FTIR was used to detect whether there was a chemical bond between thermoplastic acrylic and the organic solvent. A total of 80 samples were prepared and divided into 20 samples for the surface roughness test and 60 samples for the shear bond strength test. Failure type was assessed visually.

Results: Shear bond strength and surface roughness values of un treated samples were lower in comparison to surface treated groups; the greatest post thermocycling bond strength value was recorded for the samples treated with ethyl acetate following 1500 cycles.

Conclusions: Bonding strength was improved following ethyl acetate surface treatment.

***Key words**: Thermoplastic acrylic, ethyl acetate. toxicity*

Introduction

There are various types of thermoplastic denture base materials used in dentistry; polyamide, polyester, acrylic resin, and polycarbonate[1]. Thermoplastic acrylic resins have a main advantage over the classical acrylic resins which is the absence of residual monomer. The material is non-toxic and it is stable in water, alcohol, saliva and stomach acids. It has high wear resistance, mechanical strength and aesthetics [2].

Gradual oral tissue changes allow alveolar ridge sensitivity to the applied functional pressure during functional activities[3, 4]. Therefore, using of soft liners for removable prostheses will provide cushioning layer on the denture intaglio surface resulting in decreased transmission of traumatic occlusal forces[4, 5].

Resilient denture liners can be divided into acrylic based liners, [such as: poly (methyl methacrylate)] and silicone based, (such as poly dimethyl siloxanes) known as silicone[6]. Ideal denture relining strongly dependent on the high bond strength between the two bonded materials which is an essential requirement to attain an intact interface; it can overcome leaching out of relining material from the surface of denture base. In contrast, poor bond strength will cause mechanical strength deterioration[7, 8].

Several attempts were made to enhance adhesion of soft liners including chemical and mechanical surface modifications[9]. Among the chemical surface preparations were organic solvents, such as ethyl acetate[6]. Ethyl acetate is a popular organic solvent that is not listed in the classification of the International Agency for Research on Cancer (IARC) and selected as a safe surface treatment mediator that can swell the denture base surface increasing the bond between the denture base and soft liner[10].

The denture base with soft liner is subjected to thermal variations intraorally which affect negatively on bonding strength. Therefore, thermocycling is used to evaluate bonding durability of adhesive interface against thermal stress as it represents oral function in vitro[11].

This study aimed to assess the effect of surface preparation using ethyl acetate on surface roughness and shear bond strength of thermoplastic acrylic denture base to acrylic soft liner.

Materials and Methods

Heat polymerized acrylic soft liner (vertex–soft / Netherlands), injection molded thermoplastic acrylic denture base material (Deflex \ Argentina) and ethyl acetate solvent were used in this study.

In order to select the most effectual time, pilot study of shear bond strength and surface roughness tests were performed and 4 application periods of ethyl acetate were used (60, 120, 180 and 240 sec.). According to the results of pilot study for each test, 180 sec of ethyl acetate surface treatment was the most appropriate time because it showed improvement in shear bond strength between thermoplastic acrylic and soft liner.

SEM (Inspect S50, FEI, Japan) was taken to detect topographical changes to the denture base material surface following ethyl acetate surface treatment.

FTIR analysis (Tensor 27, Bruker, Germany) was also accomplished to explore the chemical interaction between ethyl acetate solvent and the surface of thermoplastic acrylic material.

Samples grouping

Twenty Samples of injection molded thermoplastic acrylic denture base were fabricated to perform surface roughness test. Injection molded thermoplastic acrylic and heat cured acrylic soft liner was used to fabricate 60 samples used for shear bond testing. According to the number of thermal cycles, thermoplastic acrylic samples were divided into 3groups and these groups were segmented into 2subgroups of 10 samples depending on ethyl acetate surface treatment. The first group was tested for shear bond strengths without thermal cycling while other two groups were thermocycled for 1500 cycles and 3000 cycles in water before testing.

Samples design

20 rectangular samples of thermoplastic acrylic with dimensions of (65mm x 10 mm x 2.5 mm) were fabricated for surface roughness testing according to ADA Specification No.12, (1999).

For shear bond testing, acrylic blocks with the measurements of (75mm x 25mm x 5) mm with a stopper of 3mm depth were constructed. Two acrylic blocks were put over each other to produce one sample with a space of (25mm x 25mm x 3 mm) between blocks[4].

Samples preparation

In order to obtain the acrylic blocks for shear bond and surface roughness test, plastic and metal patterns were constructed in accordance with dimension of each test and invested in silicon duplicating material (Addition Silicon, putty consistency Zhermack- Italy) .Silicone molds were then invested in dental stone in a flask. Preformed wax tubes were then connected to the pattern to allow material injection. Wax tubes were removedby performing wax elimination process. According to manufacturer's instruction, injection of the thermoplastic acrylic samples was done under (5-7 Bar) for (15 min) and at temperature (265ºC ± 10ºC). The finished acrylic samples were stored in distilled water at 37ºC for 48 hours according to ADA Specification No.12, (1999).

Surface conditioning

Surface roughness samples were cleaned ultrasonically [6]and were then air dried for 15 minutes[12, 13].

Shear bond test samples were abraded by using 400-grit silicon carbide paper[14]**.** This procedure was performed manually [15, 16], and under running water to overcome heat generation[17]. . Samples were then cleaned ultrasonically [13] and air dried for 15 minutes[12, 13].

Surface treatment with ethyl acetate

Thermoplastic acrylic blocks were surface treated by its immersion a closed clean glass beaker of ethyl acetate solvent for 180 sec. Samples were then left to dry for 2 min[18]**.**

Soft liner mixing and application

Each shear bond test sample consisted of two thermoplastic acrylic blocks which were secured together in a way that a space dimensioned 25mm length, 25mm width and 3mm depth was formed. Silicon putty duplicating material was used to invest the sample which was then invested in dental stone inside a custom-made flask.

The soft lining material was proportioned and mixed in a clean glass container and left till the dough stage and then applied in custom flask with gradual application for achieving even distribution. After flask closure, curing process was done using thermostatically controlled water bath by heating up to 70ºC for 90 minute and then boiled up to 100ºC for 30 minutes.

Thermocycling procedure

40 samples were thermocycled (20 samples for each number of cycles) in a thermocycler at 5°C and 55°C (± 2°C) with 1 minute dwelling time for each temperature. The 1500 cycles were performed within 3 days while the 3000 cycles were done within 6 days [19]**.**

Testing procedure

The surface roughness samples were tested by the profilometer (Time 3200 / TR200, China) .3 readings for each sample were obtained and the mean value of the 3 readings was reported as roughness value[20].

Shear bond test was done by using universal testing machine (Laryee, China) with 100 Kg load cell capacity and cross head speed of 0.5mm/min. Separation force was calculated and shear bond strength value of each sample was calculated by the following equation:

Shear Bond strength=F (N)/A (mm2).

F= failure force (N).

A= sample cross sectional area (mm2).

After shear bond test samples testing, failure mode at bonded area was then estimated visually. If tearing occurs in soft liner itself named as cohesive failure, while total detachment in the bonded interface between the soft lining and acrylic resin named as adhesive .Mixed failure refers to both failure types[21]**.**

Results

FTIR results showed that there was no chemical interaction between ethyl acetate and thermoplastic denture base material.

The morphological changes of untreated samples and samples with 180 sec ethyl acetate surface preparation determined by (SEM) are shown in figures (1, 2). SEM found that a dissolved topography with various pores in the thermoplastic acrylic surface was promoted following ethyl acetate treatment when compared with the control group in which many even parallel scratches have formed following abrasion with silicon carbide paper.

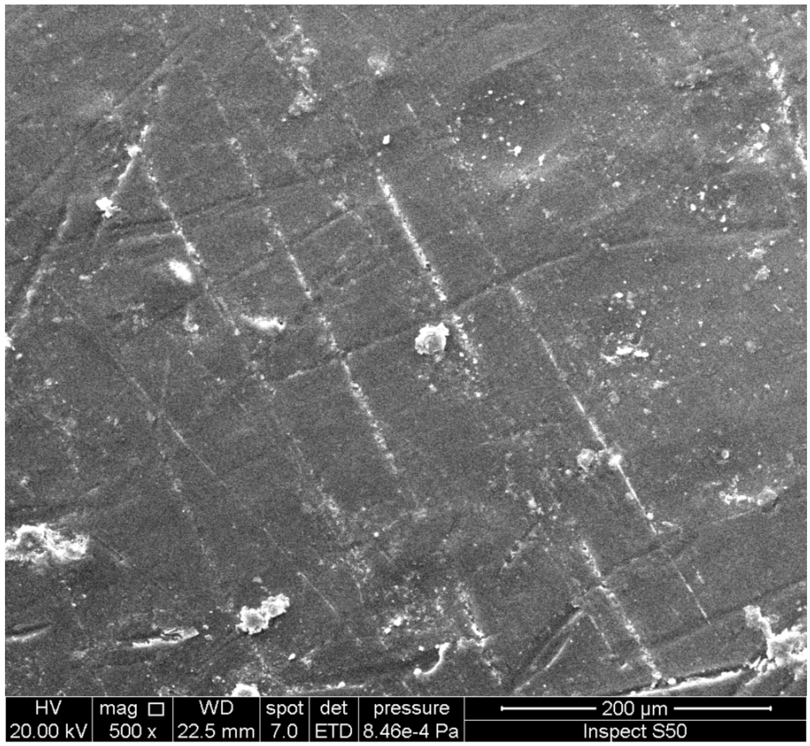

Figure (1): SEM of the untreated sample surface at a magnification ×500.

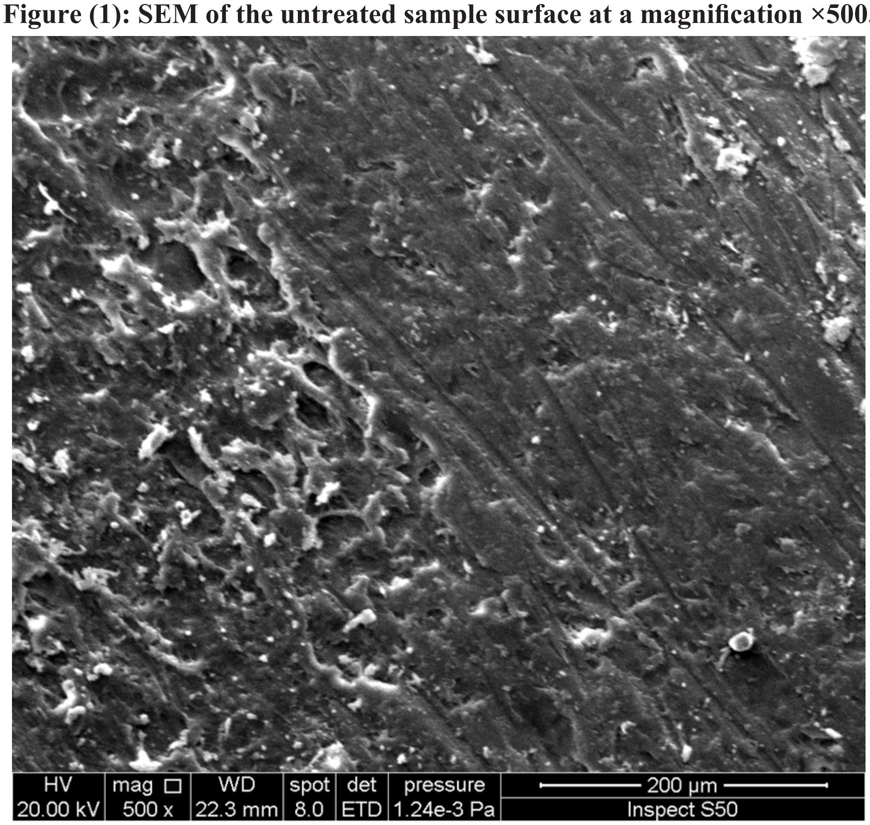

Figure (2): SEM of the 180 sec ethyl acetate surface treated surface at a magnification ×500.

The statistical analysis of the results gained, specified that surface treatment of thermoplastic acrylic with ethyl acetate caused increasing in surface roughness of thermoplastic acrylic material. As shown in table (1) surface roughness mean value of ethyl acetate surface treated groups was significantly higher than that of control group (no surface treatment).

Table (1): Comparison in surface roughness between study groups (values in µm).

Surface roughness	N	Study Group		Range	Student t-test	P - Value
		Surface treatment group Mean ± SD	Control group Mean ± SD			
	10	0.803 ± 0.06	0.351 ± 0.03	0.308 – 0.916	19.581	0.001

It has also been noticed that mean of shear bond strength test was significantly higher in surface treated group than in control group in cycles 0, 1500, and 3000. Highly significant difference was noted in shear bond strength of surface treated groups as compared to control group as shown in (table2).

Table (2): Comparisons in shear bond strength test between study groups in cycles 0, 1500, and 3000(values in N/mm$^{2)}$.

Shear bond strength test	N	Study Group		Range	Student t-test	P - Value
		Surface treatment group Mean ± SD	Control group Mean ± SD			
0 Cycle	10	0.845 ± 0.12	0.47 ± 0.01	0.36 – 1.037	7.761	0.001
1500 Cycle	10	0.794 ± 0.04	0.382 ± 0.06	0.288 – 0.831	17.015	0.001
3000 Cycle	10	0.71 ± 0.07	0.305 ± 0.05	0.18 – 0.812	14.009	0.001

Highly significant difference was also detected in comparison between cycle 0, 1500, and 3000 for each group. Highest significant difference was also detected in mean of shear bond strength in cycle (0) which was higher than that both cycles (3000, 1500) in study groups as shown in table (3).

Table (3): Comparison in shear bond strength test between cycles for each group. (One-way ANOVA) (Values in N/mm^2):

Study Group	N	Shear Bond Strength Test			F value	P - Value
		(0) Cycle Mean ± SD	1500 (Cycle) Mean ± SD	(3000) Cycle Mean ± SD		
Surface treated group	**10**	0.845 ± 0.12	0.794 ± 0.04	0.71 ± 0.07	6.532	**0.005**
Control	**10**	0.47 ± 0.01	0.382 ± 0.06	0.305 ± 0.05	12.708	**0.001**

Upon examining failure modes, it appeared that samples which were tested without thermocycling experienced cohesive failure in 80% of samples in control group and 20% had mixed failures with total cohesive failure 100% in samples treated with ethyl acetate.

Thermocycling turned failure mode to a mixed type. Samples that thermocycled 1500 times had mixed failure in which 60% of samples in the control groups failed adhesively while 40% of samples experienced mixed failure for the surface treated group 50% of samples failed cohesively with 50%had mixed failures.

Samples which thermocycled 3000 times had mixed failures total adhesive failure of the control group 100% with 20 % cohesive failure and 80% mixed failure for the surface treated group.

Discussion

Achievement of high bond strength between a denture base and soft denture liner is a challenging procedure. Several surface preparations have been suggested to improve that bond. Several researchers suggested surface wetting with organic solvents such as ethyl acetate. It was found that the bonding mechanism was based on monomer interdiffusion, swelling and the formation of IPN (interpenetrating network) during polymerization[14].

A significant increase that was noticed in surface roughness values considered that the chemical surface treatment with ethyl acetate had produced pits with a porous surface.

Noticeable improvement in shear bond strength following ethyl acetate surface preparation owed to the solvent ability for denture base surface swelling .Polymer chains infiltration by solvent took place and polymer depolymerizing enhances the formation of IPN and thus improving mechanical interlocking.

Although thermocycling decrease the bonding strength for all tested groups, surface treated groups showed high bonding strength than control groups. Statistical analysis showed that highest shear bonding strength values observed in samples tested without thermocycling while greatest post thermocycling values were observed in samples thermocycled 1500 times while samples which were tested after 3000 cycles had the lowest bonding strength. Decline in bonding strength related to the huge amount of absorbed water during thermocycling leading to swelling and concentration of stresses at the liner/ denture base interface or could be associated with viscoelastic properties changes of the liner [22, 4]. Water ageing, nature of the denture base material and temperature are the main factors affecting bonding between resilient lining and denture base [23, 22]. So declining in bonding strength was due to massive water amount absorbed by the hydrophilic acrylic liner leading to internal polymer damage by water droplets growth and irreversible polymer matrix breakdown and cracks formation by continual droplets growth[24, 4]. The material brittleness will increase and more external load is transferred to the interface. Significantly lower bond strength was detected in all thermocycled groups in comparison to non thermocycled groups. This outcome may be credited to thermal aging and water sorption of material at soft liner/denture base interface[22].Failure was predominantly cohesive in non thermocycled groups and mixed in thermocycled groups. Failure modes after testing suggested that exposing to thermal changes and multidirectional forces in mouth causes denture liner to undergo cohesive or mixed debonding.

Conclusions

1. Ethyl acetate surface treatment increased the surface roughness of thermoplastic acrylic denture base and improved the shear bond strength of thermoplastic acrylic with acrylic soft liner..

2. Thermocycling had a deleterious effect on bonding strength of lining materials to denture base materials.

Ethical Clearance: The Research Ethical Committee at scientific research by ethical approval of both MOH and MOHSER in Iraq

Conflict of Interest: None

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