ORIGINAL ARTICLE

The Effectiveness of Chitosan Nano-Particles Addition into Soft Denture Lining Material on Tensile strength and Peel bond Strength of Soft Denture Lining Material

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ABSTRACT

Background: the soft liners are designed to have low compliance. Therefore, they appear to absorb a lot of occlusal forces (cushioning effect) by distortion (elastic deformation). Such material adapts very well to the mucosa in the unloaded situation and can provide appropriate retention and resistance for prostheses.
Methods: This study include Incorporation of Chitosan nano-particles into soft liner monomer in concentration (0% control, 1.5%, 2% by wt.) to determine the effect of chitosan addition into the soft lining material on tensile strength and peel bond strength. for each group , 10 specimens were prepared. For tensile strength test 30 specimens with central cross section area (33mm length, 3mm width, 6mm depth) were prepared according to (ASTM D412). For peel bond strength test 30 specimens were prepared according to (ASTM D903-93) specification.
Results: An Improvement in the tensile strength and a reduction to limited degree of Peel bond strength of soft lining material after adding different percentage of chitosan nano-particles (1.5%, and 2% by wt. incorporation).
Conclusion: The tensile strength of soft liner was increased after 1.5% and 2% by wt. chitosan incorporation, which makes the soft liner more resistant to forces attempting to create plastic deformation, while the Peel bond strength was reduced to a limited degree after adding different percentage of chitosan nano-particles.
Keywords: Chitosan, Nano-Particles, Soft Denture Lining Material.

INTRODUCTION

While the average life span of a complete denture could be 4–5 years, the exact life depends on the degree of alveolar bone resorption. If a denture's intaglio surface needs to be replaced to enhance the fitting of the denture, a relining material can be applied[1]. Poorly fitting dentures need a new resin coating on the base of the denture to enhance the fit of the dentures to the residual ridges and to improve occlusion, retention and stability[2].

Specifically; the soft liners are designed to have low compliance. Therefore, they appear to absorb a lot of occlusal forces (cushioning effect) by distortion (elastic deformation). Such material adapts very well to the mucosa in the unloaded situation and can provide appropriate retention and resistance for prostheses [(3)]. The soft liners have the ability to improve comfort for denture patients with residual ridge atrophy, uneven ridge , bony undercut , non-resilient thin mucosa, jaw clenching and also xerostomia patients[4].

Chitin is the main structural component of crustacean and arthropod shells. The partial deacetylation of chitin results in chitosan which is a polysaccharide composed of glucosamine and N-acetyl glucosamine units linked by glycosidic bonds β(1–4)[5].

Due to its biocompatibility, biodegradability, nontoxic properties and antimicrobial activity, there has been growing interest in the modification and application of chitosan in biomedical fields. [(6)]

MATERIALS AND METHODS

This study include Incorporation of Chitosan nano-particles (India) into soft liner monomer (Netherland) in concentration (0% control, 1.5%, 2% by wt.) to determine the effect of chitosan addition into the soft lining material on tensile strength and peel bond strength for each group, 10 specimens were prepared **.**

Specimens' preparation

Tensile strength test specimens' preparation: A specimen with central cross section area (33mm length, 3mm width, 6mm depth) were prepared according to (ASTM D412)[7]**.** These plastic patterns were invested into dental stone of the lower part of the flask, and after complete stone setting was occur, the surface of stone was coated with a thin layer of separating medium, then the upper part of the flask was placed on the lower one and filled with dental stone mixture. One hour later, the flask was opened and the plastic patterns were removed**.** The proportioning and mixing of soft liner were done according to the manufacturer's instructions (P / L ratio 1.2 g:1ml). Chitosan nano-powder was incorporated into the soft liner monomer with probe sonication apparatus and the weight of the added chitosan powder should be subtracted from the soft liner powder weight to keep the same manufacturer's P/L ratio[8,9]. Then the packing and curing of soft liner were done.

Peel bond strength test specimens' preparation: Because the specimens size to be prepared according to ASTM D903-93 specification is too long, so half of the length and width was considered satisfactory for testing[10]. Two rectangular stainless steel plates were fabricated, one for PMMA contains cavities with dimensions of 100x10x2 mm (length, width, height respectively), and the other for soft liner contains cavities with dimensions of 150 x 10 x 2mm (length, width, height respectively). By Auto CAD2015 computer software, a special flask was designed with four plates, two of them were used as a cover (5 mm thickness), and the others (2 mm thickness) contain holes inside them, one for acrylic and the second for soft liner as mentioned above. The first step in specimens preparation was packing of heat cured acrylic resin (the mixing according to the manufacturer's instructions P/L ratio: 2.3g/1ml) into the plate holes prepared for acrylic[11]. The flask was then closed completely by placing it under hydraulic press with slow pressure application to permit even flow of the material until reach 100 MPa and left for five minutes. After packing was completed, the flask was placed in the water bath for 20 min at 100℃ according to the manufacturer's instructions.

After polymerization was completed, the flasks were removed from the water bath and allowed for bench cooling (for 30 min.), then it placed under running tab water for 15 min. for cooling. The strips of acrylic were removed from the flask and trimming of excess material was done. The acrylic specimens surfaces to be bond with soft liner were smoothed (with 240 - grit silicon carbide paper) and cleaned with distilled water and allowed to dry, then returned to flask place[11]. Acrylic resin part of 30 cm must be covered with tinfoil before soft liner packing to ensure that just 70mm length of the soft liner was bonded and the remaining length of soft lining material was free of bonding to acrylic[10]. The proportioning and mixing of acrylic based soft liner powder and liquid was done according to the manufacturer's instructions (P/L ratio 1.2g:1ml) , when the mixture was reached to dough stage, it was inserted into the plate hollow spaces designed for soft lining material**.** This plate was closed by another plate of 5 mm thickness, then the screws were tightened, and the flask was placed under hydraulic press until pressure of 100MPa was reached and left for 5 min., then the excess materials were removed[12]. The ready flask was placed in water path with the temperature of (70°C) for (90 min.), then it was raised to reach (100°C) for (30 min.). After that period, the flask was removed from water path for bench cooling, the specimens were removed from the flask and any excess material was trimmed away by using a sharp blade.

Evaluating the effect of aluminum potassium sulfate on tensile strength and peel bond strength of the soft-liner

Tensile strength testing procedure: A total of 30 specimens, 10 specimens for each group were prepared and stored in distilled water for 24 hours . [(13)] The cross sectional area of the narrow portion of the specimen was calculated by measuring the specimens width and thickness at the center of the narrow portion by using a vernier caliper with digital readout. The specimen was mounted in universal testing machine in which the upper member kept constant, while the lower member moved at a steady rate (500 mm / min) [(14)] , the maximum force when each specimen was broke by stretching was recorded by the computer software**.** The ultimate tensile strength (T) was calculated according to ISO 37: 2011 from the maximum stretching force at break (F) divided by the cross sectional area (width×thickness) of the narrow portion of the specimen (A):

$$T = \frac{F\ (N)}{A(mm2)}$$

Peel bond strength testing procedure: The specimens analysis of peel bond strength test was made according to ASTM D903-93 by using universal testing machine at 180º angle and speed of 152 mm/min. the heat - cured acrylic resin free portion was clamped on the UTM upper clutch, while the portion of the soft liner that is free was folded and fixed on the lower clamp of the UTM (gripping of 25 mm from the soft liner material). To keep the specimens in the plane of clamps during testing, the peel specimen was held alongside an alignment plate. After specimens testing was completed, by naked eyes the nature of the failure at bonded area was evaluated and named as cohesive, adhesive or mixed failures. Cohesive means the tearing happen in soft liner itself. Adhesive means there was a total separation in the bonded area between the soft liner and acrylic resin while, mixed failure refers to both failure types[10].

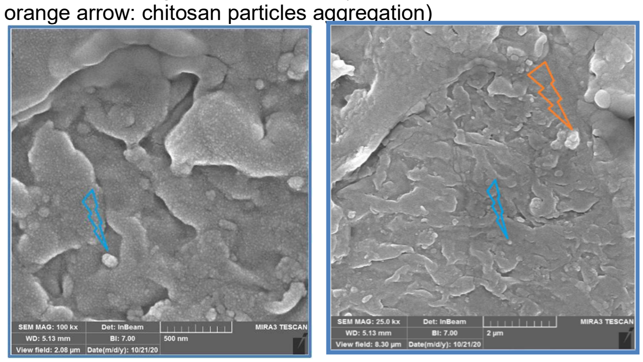
Peel bond strength was calculated through using the following equation in which the angle of peeling is considered 180°[15].

$$peel\ bond\ strength = \frac{average\ load}{wid\quad of\ the\ sample}$$

RESULTS

Scanning electron microscope (SEM): SEM results of soft liner before and after the addition of chitosan nano-particles powder are shown in **Figure 1**. The results show well distributed nano-particles with some degree of filler aggregation into the soft lining material matrix.

Figure 1: SEM images of soft liner with 1.5% of chitosan at A-25 kx, B-100kx magnification power. (Blue arrow: chitosan particle, orange arrow: chitosan particles aggregation)



A B

Tensile strength test: Descriptive statistics of tensile strength test results are presented in Table 1 with maximum value (1.73 MPa) in 2% by wt. chitosan experimental incorporation group, and the mean of this group (1.4670 MPa), while the minimum value (0.34 MPa) in control with mean of (0.4070 MPa).

One-way ANOVA for tensile strength test results showed a high significant difference (0.000) between the tested groups . Between groups , sum of squares = 5.780 , degree of freedom = 2 , mean square = 2.890 , F-statistic = 40.981. Within groups , sum of squares = 1.904 , degree of freedom = 27 , mean square = 0.071. Total sum of squares = 7.684, degree of freedom = 29.

Test of Homogeneity of Variance and multiple comparison test for tensile strength test: In accordance with the results of data homogeneity of Levene's test

(Levene's Statistic = 2.645 , df1=2 , df2=27 , P value = 0.089) , which show that there is no significant differences between the groups variance , Bonferroni test has been chosen for tensile strength test multiple comparisons, there was a high significant difference between all tested groups (Table 2).

Peel bond strength test: The higher mean value of control group (4.54 N/mm) while the lowest mean value is (3.188 N/mm) for experimental group (2% by wt. of chitosan nano-particles incorporation)**.**

By examining the failure mode of the specimens, it appeared that all samples of control group failed cohesively.

In 1.5% by wt. incorporation group all the samples failed cohesively except three of them show adhesive failure, while in 2% by wt. incorporation group, 2 specimens show cohesive failure, 6 specimens show adhesive failure and 2 specimens show both failures. Descriptive statistics of peel bond strength test results are presented in (Table 3) with maximum value (5.51 N/mm) in control group and the mean of control group (4.54 N/mm), while the minimum value (2.39N/mm) in 2% by wt. incorporation group with mean of (3.188 N/mm).

One - way ANOVA for peel bond strength test results showed a high significant difference (0.001) between all studied groups Between groups , sum of squares = 10.244, degree of freedom = 2 , mean square = 5.122 , F-statistic = 10.195 . Within groups, sum of squares = 13.564 , degree of freedom = 27 , mean square = 0.502 . Total sum of squares = 23.808 , degree of freedom = 29.

Test of Homogeneity of Variance and multiple comparison test for Peel bond strength test: In accordance with the results of data homogeneity of Levene's test (Levene's Statistic =1.921 , df1=2 , df2=27 , P value = 0.166), which show that there is no significant differences between the groups variance, Bonferroni test has been chosen for Peel strength multiple comparisons, there was a high significant difference between all tested groups except the difference between 1.5% by wt. incorporation group and 2% by wt. incorporation group was non-significant (table 4)

Table 1: Descriptive statistics of tensile strength test for the studied groups

	N	Mean (MPa)	Std. Deviation	95% Confidence Interval for Mean		Min.	Max.
				Lower Bound	Upper Bound		
Control	10	0.407	0.1011	0.3347	0.4793	0.34	0.69
1.5%	10	0.781	0.3449	0.5342	1.0278	0.63	1.76
2%	10	1.467	0.2869	1.2617	1.6723	0.94	1.73

Table 2: Boneferroni multiple comparisons test of tensile strength test

Tested Groups		Mean Difference (I-J) (MPa)	Sig.
Control	1.5%	-.3740*	0.012 (HS)
	2%	-1.060*	0.000 (HS)
1.5%	2%	-.6860*	0.000 (HS)

** The mean difference is significant when P value < 0.05.*

Table 3: Descriptive statistics of peel strength test for the studied groups.

	N	Mean (N/mm)	Std. Deviation	95% Confidence Interval for Mean		Min.	Max.
				Lower Bound	Upper Bound		
Control	10	4.54	0.62216	4.0949	4.9851	3.54	5.51
1.5%	10	3.457	0.48169	3.1124	3.8016	3.07	4.64
2%	10	3.188	0.94236	2.5139	3.8621	2.39	4.93

Table 4: Boneferroni multiple comparisons test of Peel bond strength test

Tested Groups		Mean Difference (I-J) (N/mm)	Sig.
Control	**1.5%**	**1.08300***	**0.006**
	2%	1.35200*	0.001
1.5%	2%	0.26900	1.000

** The mean difference is significant when P value < 0.05.*

DISCUSSION

Due to the viscoelastic properties of soft denture liners that minimize and redistribute the functional load over the denture bearing area, soft lining materials play a major role in prosthetic dentistry [16]. Due to the biocompatibility, biodegradability, non-toxic properties and antimicrobial activity of Chitosan, there has been growing interest in the application of Chitosan in biomedical field[6].

Tensile strength test: It is the maximum stress that a material can withstand before being deformed locally [17]. It is one of the desirable mechanical properties of soft lining material, it was evaluated by applying a tension to the specimens [18]. The tensile strength is directly influenced by the number of polymer chains parallel to the force direction , degree of cross linking and filler -polymer bonding[19].

The result of the present study showed an elevated tensile strength in experimental groups with 1.5% and 2% by wt. addition of chitosan nano-particles to soft liner, with the maximum increase was noticed in 2% by wt. concentration, this may be due to a reduction of cross-linking degree as the nano-particles was infiltrated in to inter macro molecular polymer chains , so the mobility of segments of polymer chains was increased and the tensile strength was improved[14]. The bond strength of the chitosan / polymer may have an effect on tensile property. Stronger chitosan / polymer bonding increasing the tensile strength values[14].

Peel bond strength test: A satisfactory bond between the soft liner and the acrylic resin denture base is one of the important requirements for these materials. Unfortunately, a common clinical failure found in dentures that are relined was peeling of the soft lining materials from the denture bases. With the inclusion of antimicrobial agents into the soft lining products, this problem can be aggravated because it has been shown that these drugs can affect their physical properties[20].

A 180° peeling is a common method used to evaluate the bond strength and this test has been identified as a more meaningful test to predict a material's ability to bond in a clinical setting since debonding typically begins at the exposed edge of the lining through an apparent peeling process [(15)]. The interface between the denture base and the soft liner was visually examined and the failure modes were classified as adhesive, cohesive, or mixed failure. Depending on whether, the tear region was only in the interface area, soft liner only or in both locations, respectively[21-23].

The results of present study showed a decrease in peeling strength of groups of experimental specimens of different concentrations (1.5%, 2% by wt. incorporation of chitosan nano-powder) compared to the results of the control group, with the highest mean value (4.54N/mm) for the control group, while the lowest mean value was (3.188 N/mm) for 2% by wt. experimental group. The physical presence of chitosan nano-powder within the polymeric matrix has been suggested to affect the polymerized material structure, and since the samples are stored in distilled water, the chitosan may absorbed the water due to its hydrophilic properties, affecting the particles volume in the sample and influencing the soft lining material's bond strength to the denture base. In addition, the presence of the chitosan in soft lining material can affect the ability of plasticizers to penetrate the polymer chains and form a softened gel that will lead to a decreased plasticizer content that can minimize polymer bead disengagement, resulting in poor cohesion between the polymeric chains [(11, 20)]. The reduced peel strength of experimental groups (1.5%, 2% by wt.) also may be attributed to slight aggregation of chitosan nano-particles (as noticed by the SEM results) because of high surface energy, which may cause micro fracture that weaken the polymer structure and affecting soft liner / acrylic bond strength.

This test also involves each sample's failure mode, cohesive failure was predominant in the group of control specimens due to its decreased tear resistance, while adhesive failure was noticed in (3) samples of the experimental group (1.5% by wt.) and (8) samples of the experimental group (2 % by wt.) due to the water absorption by the soft lining material (because of the hydrophilic nature of chitosan), which may lead to swelling of the interfacial bonded surface between soft lining material and denture resin which lead to increase stress and the resultant adhesive rather than cohesive failure. An effort may be use to improve the bond with resin bases involve the painting of bonding agents by using special primers applied to the acrylic base to solve the problem of peel bond strength reduction[24].

The mixed failures (both adhesive and cohesive) may be due to bonding surface swelling and / or decreasing in tear resistance of soft liner after chitosan nano-particles incorporation.

CONCLUSIONS

Depending on the study results, the following conclusions were obtained:

1. An Improvement in the tensile strength (increased), which makes the soft liner more resistant to forces attempting to create plastic deformation.
2. Peel bond strength of soft lining material was reduced to a limited degree after adding different percentage of chitosan nano-particles (1.5%, and 2% by wt. incorporation).

Ethical clearance: The researchers already have ethical approval from the University of Baghdad's College of Dentistry.
Source of Funding: Self funding
Conflict of Interest: No conflict of interest

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