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An Evaluation of Tensile Bond Strength of Soft Liners to Conventional and Multi-Walled Carbon Nanotubes Reinforced Heat Cure Denture Bases – An Invitro Study

[1]Dr. Dilip Jayyarapu, Senior Lecturer, Department of Prosthodontics, Kamineni Institute of Dental Sciences and Kaloji Narayana Rao university, Narketpally, Telangana, India

[2]Dr. Ramu Reddy, Head of the Department , Department of Prosthodontics, Kamineni Institute of Dental Sciences and Kaloji Narayana Rao university, Narketpally, Telangana, India

[3]Dr N. Mamatha, Senior Lecturer, Department of Prosthodontics, Kamineni Institute of Dental Sciences and Kaloji Narayana Rao university, Narketpally, Telangana, India

[4]Dr Aditya sai Jagini, Reader, Department of Prosthodontics, Kamineni Institute of Dental Sciences and Kaloji Narayana Rao university, Narketpally, Telangana, India

[5]Dr Vidhyadhar.D, Senior Lecturer, Department of Prosthodontics, Kamineni Institute of Dental Sciences and Kaloji Narayana Rao university, Narketpally, Telangana, India

[6]Dr Vamshikiran.K, Senior Lecturer, Department of Prosthodontics, Kamineni Institute of Dental Sciences and Kaloji Narayana Rao university, Narketpally, Telangana, India

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Corresponding Author: Dr. Dilip Jayyarapu, Senior Lecturer, Department of Prosthodontics, Kamineni Institute of Dental Sciences and Kaloji Narayana Rao university, Narketpally, Telangana, India

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Abstract

Aim

The present in vitro study aimed to evaluate the tensile bond strength of soft liners to conventional and multi-walled carbon nanotubes reinforced heat cure denture base resins.

Materials and Methods

A total of 180 specimens were prepared to a size of 20mm×15mm×12mm with a 3mm spacer for soft liner, out of which 90 specimens were made with conventional lucitone denture base resin and the rest 90

were reinforced with multi-walled carbon nanotubes. Each group was divided into 3 subgroups based on the soft liner selected i.e COE – Soft, GC – Reline Soft, Molloplast – B that were polymerized against these denture base blocks.

Results

GC Reline soft with primer exhibited the highest mean tensile bond strength i.e 1.8Mpa with MWCNT's reinforced heat cure resins followed by 1.347MPa with conventional lucitone denture base resins.

Conclusion

This study concluded that the presence of MWCNT's in denture bases allowed the primer of the GC soft liner to functionalize them that ultimately led to highest bond strength compared to conventional denture bases, thus proving the importance of selecting an appropriate soft liner that contains a primer and also the significance of reinforcements in denture base materials.

Keywords

Soft liner, multi-wall carbon nanotubes, primer, functionalization, in vitro study.

Introduction

Meticulous attention and care during the construction of complete dentures will minimize adverse changes in the supporting tissues, but cannot preclude them, due to the fact that the residual ridge resorption, is inevitable and progressive that will be accompanied by some minimal to moderate clinical changes in the supporting tissues which includes loss of retention and stability, general soreness and inflammation, loss of vertical dimension of occlusion and loss of support for facial tissues. The severity of observed clinical changes in the patient, allows the dentist to make a decision regarding the servicing of a prosthesis which could be a laboratory reline, a rebase, or a remake.[1]

Considering the above clinical changes, if the dentist designs a treatment plan that includes relining, then the prognosis depends on selection of soft relining material and the existing denture base. Firstly, concentrating on the resilient liners, it's already been in the literature that these are being used for more than a century in the clinical management of prosthodontic patients. They are applied to the intaglio surface of a denture to achieve equal force distribution by reducing localized pressure and improves retention by engaging the undercuts. The liners are mainly of two types: plasticized acrylic resins and silicone elastomers, available in auto-polymerized and heat-polymerized forms.[2] While several problems have been surfaced with liners, like porosity, loss of softness and poor tear strength, the most important problem is bond failure between resilient denture liner and denture base especially for silicone liners.[1-2] The success of the bond strength depends on various factors like water absorption, surface pre-treatment, surface primer use or bonding agent, and denture base composition.

Clinically, in the process of application of a soft liner, the intaglio surface of a denture base is trimmed to accommodate the liner due to which the reduced thickness of the denture base would make it more prone to fracture. As a result, heat cure polymethyl methacrylate (PMMA) denture base resin are being reinforced with materials such as glass fibers, long carbon fibers, and metal wires so as to improve the mechanical properties.[3] Recently, according to various researches, carbon nanotubes (CNT),as a reinforcement material, is shown to have outstanding mechanical properties with reported strengths 10 to100 times higher than steel at a fraction of the weight and also they are strong, resilient and lightweight. They have a flawless structure that are classified into 2 main types, namely single walled and multi walled CNTs.[3-4]

With this, one can assess that the overall bond strength between the denture base and liner might increase through primer application, through sand blasting of acrylic denture base and changes in the prosthesis material like reinforcement with carbon nanotubes.[5]

In this regard, the present in vitro study aimed to evaluate the tensile bond strength of soft relining materials to conventional and multi walled carbon nanotubes reinforced denture base resins. Null hypothesis states that reinforcements in denture bases and primer application have no effect on the bond strength of liners with denture bases.

Materials and Methods

Preparation of Carbon Nanotubes Reinforced Lucitone Denture Base Material

Multi walled Carbon nanotubes (Monad nanotech, Mumbai) were added to monomer by weighing it with electronic precision balance. It was added as 0.5% by weight (i.e. 0.5 gm of carbon nanotubes in 94 grams of monomer) and mixed with monomer by ultrasonic agitation for uniform dispersion and disintegration. This monomer containing carbon nanotubes was mixed with polymer in the ratio of 10 ml: 21 g (32ml) for 20 seconds for specimen fabrication.

Specimen Preparation

A total of 180 specimens were prepared, out of which 90 specimens were made with conventional Lucitone denture base resin (Group-I) and the rest 90 with MWCNT's reinforced Lucitone denture base resin (Group-II). To start with the methodology, wax blocks of dimensions 20mm×15mm×12mm for denture base and 3mm thick stainless steel spacer were used for specimen fabrication and a putty index of wax blocks was made for further preparation of remaining specimens. Wax blocks with spacer were invested in denture flask (Figure 1) and after dewaxing, the mold space with stainless steel spacer was used as a matrix and packed with Conventional Lucitone (Dentsply International Inc., York, PA, USA) and MWCNT’s reinforced Lucitone denture base resins, for 30 specimens each.

Figure 1: wax blocks with stainless steel spacer

The specimens were cured and wet ground with SiC grinding paper (500 grit) to standardize the bonding surfaces. Each group was subdivided into 3 subgroups based on the soft liners that were polymerized against these denture base blocks (Table-1). Manufacturer's instructions for processing of these soft liners were shown in (Table-2).

Table 1: Groups and Sub groups

GROUP-I (n=90) Conventional Lucitone denture base resin			GROUP-II (n=90) Carbon nano tubes reinforced Lucitone denture base resin		
Sub group	SOFTLINER	N	Sub group	SOFTLINER	n
IA	COE-SOFT	30	IIA	COE-SOFT	30
IB	GC RELINE SOFT	30	IIB	GC RELINE SOFT	30
IC	Molloplast-B	30	IIC	Molloplast-B	30

Table-2: Soft liners Composition and Manufacturer's instructions for processing.

BRAND	COMPOSITION	PROCESSING METHOD
COE-SOFT (GC AMERICA INC)	Chair side acrylic soft liner consists of: POWDER: It consists mainly of Poly ethyl ethacrylate LIQUID: It consists mainly of Benzyl Salicylate and Ethyl alcohol.	Polymer and monomer were mixed in the ratio of 1.5 gm:8 ml .
GC- RELINE SOFT (GC AMERICA INC.) GC Reline soft primer- R	Chairside vinyl polysiloxane resilient denture liner consists of Silicone dioxide, Vinyl dimethyl polysiloxane, hydrogen polysiloxane. clear liquid mostly consisting of Ethyl Acetate	GC reline primer was applied to acrylic blocks and gently dried with air, then the soft liner was directly injected into mold space as it is auto polymerizing
MOLLOPLAST-B (Detax GmbH & CO.KG Germany) PRIMO ADHESIVE	Heat cured silicone soft liner consists of • Condensation silicone material, • Poly Methyl Methacrylate • Ymethacryloyoxypropyltrimethoxysilane • Methoxy and Ethoxy Silane Derivatives.	Primo adhesive was applied to denture base surfaces uniformly with a brush 1-2 times and allowed it to dry for 60-90 minutes, then the liner is applied directly as it was ready to use dough consistency and immersed in water bath for 2hours at 100^{0}c.

Then the flasks were placed in flask press for 30 minutes. After polymerization, the specimens were removed from the flask and trimmed with sharp blade. The obtained Specimens were stored in water (37^{0}c) for 24 hours before testing[1].

Each sample was attached to the Universal testing machine (Dak system UTB9251) and all the samples were tested in tensile bond mode at a cross head speed of 5mm/min until separation occurred (Figure 2).

Figure 2: The specimen loaded on Universal testing machine

The maximum force at which separation occurred was used to calculate the tensile bond strength (σ). The obtained value was divided by the surface area of sample to obtain the bond strength in MPa.

$$\sigma = F/A$$

Results

All the analysis was done using SPSS version 18 and results showed a p-value of <0.05 as statistically significant. Comparison between two groups was done using Two-way ANOVA test (Table-3) along with pair wise comparison of each group which was done using tukey HSD post hoc test (Table-4).

Table-3: Mean Bond strength of specimens between two groups

Group		N	Mean	SD	ANOVA	
					F-value	p-value
I	IA	30	0.098	0.027	293.39	<0.001*
	IB	30	1.347	0.133		
	IC	30	0.612	0.148		
II	IIA	30	0.118	0.037	848.38	<0.001*
	IIB	30	1.800	0.133		
	IIC	30	1.030	0.077		

Independent sample t test

*P<0.05 statistically significant

p>0.05 non significant, NS

Table-4: Pair wise comparison of the bond strength of each group using post hoc test

Group	Sub Groups		Mean Difference	Std. Error	P-value	95% Confidence Interval	
						Lower Bound	Upper Bound
1	1A	1B	-1.249	0.0518	<0.001*	-1.377	-1.120
		1C	-0.513	0.0518	<0.001*	-0.641	-0.384
	1B	1C	0.736	0.0518	<0.001*	0.607	0.864
2	2A	2B	-1.682	0.0408	<0.001*	-1.783	-1.580
		2C	-0.912	0.0408	<0.001*	-1.013	-0.811
	2B	2C	0.769	0.0408	<0.001*	0.668	0.872

Tukey post hoc test

*P<0.05 statistically significant

p>0.05 non significant, NS

Result for the intergroup comparison revealed that the highest mean bond strength for Group II subgroup B was 1.800Mpa (i.e, the bond strength between the MWCNT reinforced denture base resin and the GC Reline soft with primer). This is followed by Group I subgroup B's bond strength which was 1.347MPa (i.e, the bond strength between the Conventional Lucitone denture base resin and the GC Reline soft with primer). The intra group comparisons of two groups, Group I and Group II, had highlighted the fact that the same sub group B which had GC - Reline Soft Silicone as the liner, had shown to have the greatest bond strength. This proved the potency of GC-Reline soft silicone as a promising relining material in the clinical practice.

Discussion

The most serious problem associated with the use of resilient denture liners is bond failure between the resilient denture liner and denture base that will create a potential surface for bacterial growth, plaque and calculus formation. The other problems include colonization by Candida albicans, porosity, poor tear strength, and loss of softness. Various parameters like water absorption, surface primer use, and denture base composition affect the bond between the resilient lining material and the denture base[2].

The reinforcements of PMMA used in this study was in accordance with the studies done by , Russell Waig,[3] Peng Cheng Ma,[6] Shailaja,[7] Turagam,[8] where it was proved that multi walled carbon nano tubes reinforcements will improve the strength of the prosthesis to better withstand the masticatory forces. This can be achieved by addition of carbon nanotubes to the monomer and subjecting the mixture to ultra-sonication that results in insitu polymerization with the polymer resulting in a strong interfacial adhesion between the MWCNTs and the polymer matrix along with a good transfer from the matrix to the MWCNTs. These composites hold the promise of delivering exceptional mechanical properties and multi-functional characteristics.[6]

The concept of functionalization has been highlighted in this study due to the fact that the as-synthesized CNTs which were available in bundles tend to agglomerate due to strong vander Waals forces, that will reduce their interactions with other polymers/solvents systems. As they have relatively smooth surface, the interfacial bonding between the polymer matrix and the CNTs will be reduced. Thus chemical functionalization/ covalent modification of nanotube surfaces is an effective approach to overcome the tube– tube interactions along with creation of sharp edges that might improve the interactions by adhesion with the polymer matrix at nanotube-matrix interface.[4,7] Owing to the above facts, polymethylmethacryalate with and without multiwalled carbon nano tubes was selected so that their overall bond strength with the soft liners could be assessed.

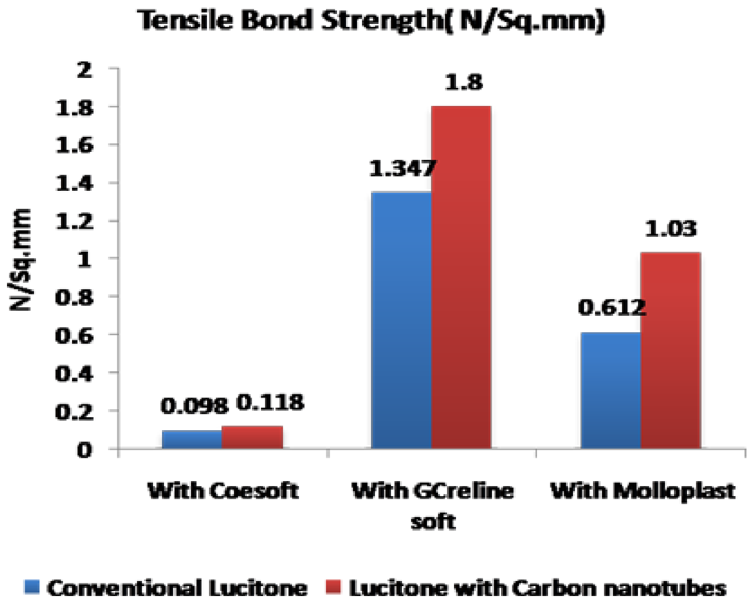
The critical limit of CNT content (that is upto 2%) was used in this study as the beyond limit effects the CNT on the strength of the reinforced matrix.[7] As CNT content increases, the strength decreases due to its high specific surface area that results in complete wetting of the CNT surface by the polymer and intercalation by the growing PMMA may not occur resulting in non-uniform dispersion, aggregation, and difficulty in load transfer.

The soft liners used in this study COE – Soft, GC – Reline Soft, Molloplast – B were in accordance with the studies done by Ayse Mese[2], Mustafa Murat Mutluay[9] in which the COE – Soft was used as a chairside acrylic soft liner, GC- Reline soft was also a chairside vinylpolysiloxane resilient denture liner with a primer that constitutes ethyl acetate as a main component and Molloplast – B was a heat cured condensation silicone material with a primo adhesive that constitutes methoxy and ethoxy silane derivatives. The primers role in this study was related to chemical functionalization of CNT for improving the dispersion and interfacial adhesion of CNTs with polymer resins.[6]

The present study evaluated and compared the tensile bond strengths of acrylic (coe-soft) and silicone soft liners (Molloplast-B, GC-RelineSoft) to Conventional Lucitone and the Carbon nanotubes reinforced lucitone denture base resins.

The results of present study within group 1 (intergroup comparison) was in accordance with the study done by Mustafa Murat Mutulay ,I. Eystein Ruyter [2,9] in which the tensile bond strengths of GC-Reline soft silicone liner with conventional lucitone denture base material (mean bond strength of 1.347MPa with a SD of 0.133) has greater bond strength when compared to Molloplast-B (mean bond strength of 0.612MPa with a SD of 0.147) and Coe-Soft (mean bond strength of 0.098MPa with a SD of 0.027) (Table:3, Graph-1) because GC-Reline soft has ethyl acetate based primer which has similar composition as PMMA, when compared to Mollopast-B that has organo silane and Coe soft which has no primer. This intra group comparison was done to assess the bond strength between the soft liners and conventional lucitone denture base materials which highlighted the importance of a primer for an adequate bond to establish between the liner and a denture base material.[2] The null hypothesis stands rejected as the difference in tensile bond strength was noticed with the MWCNTs reinforcements and the use of primers to establish a bond between the polymer resins and CNTs so that the overall bond strength would increase between the denture base and a liner.

Graph-1: Comparison of mean bond strength of different soft liners to conventional and reinforced denture base resin.



While testing the Group- II for tensile bond strengths between acrylic MWCNT reinforced lucitone denture base resin with soft liners, a well appreciated bond strength was recorded with GC-Reline soft silicone liner (mean bond strength of 1.800MPa with a SD of 0.133) when compared to Molloplast-B (mean bond strength of 1.039MPa with a SD of 0.077) and Coe-Soft (mean bond strength of 0.118MPa with a SD of 0.036) (Table 3, Graph-1). This is due to the fact that MWCNT'S may have chemically functionalized by acetate group primer of GC Reline Soft than silanol based primer's coupling agent's role of Molloplast-B. As acid treatment was among the methods of functionalization, it has shown promising results to provide functional groups (carboxyl groups) onto the CNTs surface that would covalently link to polymers. This method, however, if used in high concentrations not only creates defects on the CNT sidewalls, but also decreases the aspect ratio of CNTs because the strong acids cut the CNTs into short pieces during the oxidative process.[6] The silanol based primer's coupling agent has proved to provide a strong bond between the CNT and polymer matrix because of the adsorption of functional groups of coupling agent into the voids of CNT, thus increasing the compatibility between the matrix. [4]

The inter group comparison (Group IA & IIA) between acrylic soft liner (Coe-soft) with conventional lucitone denture base resin (mean bond strength of 0.098MPa with a SD of 0.027) and MWCNT reinforced lucitone denture base resin (mean bond strength of 0.118 MPa with a SD of 0.036) (Table 3, Graph-1) disclosed the insignificant difference for bond strength between them because of absence of a primer which would have a key role in acquiring the same. The slight increase in bond strength between soft liner with reinforced and conventional denture base resins was contributed by vanderwaal forces between carbon nanotubes and methacrylate groups which was in accordance with the study conducted by Shailaja Pande, R.B. Mathur, B.P. Singh, T.L. Dhami.[7]

The inter group comparison (Group IB & IIB) of bond strength between the GC Reline soft liners with conventional lucitone denture base resin and MWCNT reinforced lucitone denture base resin revealed that Group IIB (mean bond strength of 1.800MPa) has greater bond strength when compared to Group IB (mean bond strength of 1.347MPa) (Table:3, Graph-1). Similarly, the comparison of molloplast B groups (Group IC & IIC) indicated the same result as given by GC Reline soft thus proving the importance of carbon nano tubules reinforcements as they provide functional groups and sharp edges after functionalisation by primers of silicone soft liners that would enable the polymer molecules to wrap the CNTs resulting in a stronger interface and an effective stress transfer between matrix and CNTs.[7,10]

This study highlighted the importance of carbon nano tubes reinforcements in polymer matrix along with chemical functionalization of CNTs by primers that enhanced the tensile bond strength between the silicon soft liners and denture base resins. Recently, a new method is presented for the chemical functionalization of multi-walled carbon nanotubes (MWCNTs) through the combined process of UV light within ozone chamber and silanization reaction where the silane molecules are covalently bonded on the surface of the MWCNTs, that modifies the surface characteristics and improves the compatibility of MWCNTs with polymers.[6] In this view more studies have to be done in the future to enhance the chemical and mechanical properties for superior polymers that would result in increased tensile bond strengths between denture and a liner.

Conclusion

In conclusion, the highest tensile bond strength was between the multiwalled carbon nanotubules reinforced denture base resin and GC Reline soft, followed by Molloplast B and Coe Soft with MWCNTs. The lowest bond strength was recorded between the conventional denture base resin without reinforcements and Coe soft because of the absence of reinforcements and the primer. The presence of primer in silicone soft liners and nanotubes in denture base resins has attributed to the highest tensile bond strength between them supporting the studies done till now on MWCNT-PMMA composites.

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