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Comparative Evaluation of Chemically & Mechanically Modified Heat Polymerized Acrylic Resin for Transverse Strength: An Invitro Study

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ABSTRACT

Aim: The aim of the study was to evaluate and compare the chemically and mechanically modified heat polymerized acrylic resin for transverse strength.

Materials & Methodology: Seventy-five acrylic test specimens were divided into 5 groups (n=15/Group). Group A: Unmodified resin group, Group B: Stainless steel mesh reinforcement group, Group C: Glass fibre mesh reinforcement group, Group D: Silanized alumina nanoparticles reinforcement group and Group E: Multi-walled carbon nanotubes (MWCNTs) reinforcement group. Transverse strength was measured using Universal testing machine. The data were analyzed using 1 way ANOVA and subsequent multiple comparisons between the unmodified and modified resin groups were performed using Post hoc comparison with Bonferroni correction test.

Results: One way analysis of variance and multiple comparisons using post hoc comparison test revealed a significant difference among test groups ($p<0.01$), except Group A and Group B. The transverse strength was higher for MWCNTs reinforced group followed by Glass fibre mesh, Silanized alumina nanoparticles, Stainless steel mesh. The least transverse strength was shown by unmodified resin group.

Conclusion: Within the limitations of this invitro study it was concluded that all reinforcing materials enhance the transverse strength. Polymethylmethacrylate (PMMA) reinforced with MWCNTs showed the highest transverse strength value followed by the PMMA reinforced with glass fibre mesh, then silanized alumina nanoparticles and stainless-steel mesh. The least transverse strength was observed in the unmodified PMMA group.

Keywords: Transverse Strength Test, Multiwalled Carbon Nanotubes, Silanized Alumina Nanoparticles, Glass Fibre Mesh.

INTRODUCTION

Since the introduction of denture material in 1937, PMMA has become the most commonly used material for denture bases. It remains most popular of all the polymeric denture base materials. Despite its popularity in satisfying aesthetic demands it is still far from ideal in fulfilling the mechanical requirements of prosthesis. The fracture of dentures may be due to the mechanical properties

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of the acrylic resin or may be due to a multiplicity of factors leading to failure of the denture base material. Generally, there are three routes which have been investigated to improve the mechanical properties of PMMA [1,2].

A study of Johnston and colleagues[3] has shown that 68% of dentures will break within a few years of fabrication. Denture fracture occurs principally due to two different types of forces, flexural fatigue or impact. Flexural fatigue results from repeated application of masticatory force; it would have no detriment to the denture but may cause eventual structural failure, most likely due to the formation of microscopic cracks. So reinforcement of denture base material has been a subject of interest to the dental material aspect. One of the reinforcement step taken for improving mechanical properties were incorporation of metal mesh, but the primary problem with this technique was poor adhesion between resin and metal mesh, resulting denture base often aesthetically unsatisfactory.

Glass fibres have been considered as reinforcing materials for denture base resins since 20 yrs because of their excellent aesthetics, superior strength, and good biocompatibility[4].The mechanical properties of PMMA reinforced with glass fibres also depends on good adhesion between fibers and the resin matrix. In order to achieve better adhesion, glass fibers are treated with silane coupling agent before loading into the resin matrix.

The carbon nanotubes (CNTs) were discovered by Iijima in 1991 and since then they have been incorporated in experimental studies with PMMA to increase their mechanical properties. The nanotubes are thin and long cylinders of graphite with carbon atoms arranged in a hexagonal lattice. CNTs were found as single walled nanotubes (SWNTs) and multiwalled nanotubes (MWNTs)[5,6].

Aluminium oxide (Al2O3), commonly referred to as alumina, possesses strong ionic interatomic bonding, giving rise to its desirable material characteristics. It can exist in several crystalline phases, which all revert to the most stable hexagonal alpha phase at elevated temperatures. This is the phase of particular interest for structural applications. Alpha phase alumina is the strongest and stiffest of the oxide ceramics. Its high hardness, excellent dielectric properties, refractoriness, and good thermal properties make it the material of choice for a wide range of applications[8].

Therefore, present study compared transverse strength of unmodified heat polymerized acrylic resin and modified heat polymerized acrylic resin with four different types of reinforcing materials i.e. Stainless steel Mesh, Glass Fibre Mesh, Silanized Alumina Nanofillers and Multiwalled Carbon Nanotubes.

MATERIALS AND METHODS

The aim of the study was to evaluate and compare the chemically and mechanically modified heat polymerized acrylic resin for transverse strength. An invitro study was carried out in the Department of Prosthodontics, including Crown & Bridge and Implantology, Sharavathi Dental College and Hospital, Shivamogga, Karnataka. The fabrication of metallic bar specimen was done from Government Tool room and Training Centre, Bhadravathi, Karnataka. Carbon nanotubes integration was done at Department of Microbiology, Kuvempu University, Shankaraghatta, Shimoga. The Evaluation of Transverse strength was mechanically tested at Department of Textile Engineering, Bapuji Institute of Engineering and Technology, Davangere, Karnataka.

Experimental design consists of 5 groups; each group consists of 15 specimens with a total of 75 specimens. Group A: Heat cure acrylic without any modification; used as Control Group, Group B: Heat cure acrylic reinforced with Metal Mesh, Group C: Heat cure acrylic reinforced with Glass Fibre Mesh, Group D: Heat cure acrylic chemically modified with Silanized Alumina Nanoparticles, Group E: Heat cure acrylic chemically modified with Multiwalled Carbon Nanotubes. Mould preparation was done according to ADA specification no 12, acrylic strip specimen of dimension 65mm × 10mm × 2.5mm had to be prepared for the purpose of this study.

Preparation of specimens:

Group A: 15 Samples of group A served as a control group. Manufacturer's recommended amount of powder and liquid of DPI heat cure polymer (Polymer: Monomer ratio of 21g: 10ml), was taken and mixed on ceramic pot .The mixed resin was left in the mixing pot until it reached the dough stage, then the mix was kneaded thoroughly to make

homogenous dough. The dough was then packed into the mould. Care was taken to avoid porosities due to entrapment of air bubbles. Trial closure was performed and excess flash was removed and final closure was done under a bench press. The flask was left in the clamp for bench curing for 30 minutes at room temperature to allow proper penetration of the monomer into the polymer, for even flow of the material and outward flow of excess material. The flask was immersed in water in an acrylizer with automatic controls. Curing was done using a short curing cycle where the temperature was slowly raised to 73ºC and held for 90 min followed by boiling at 100ºC for 30 min. After the curing was completed, the flask was removed and left for bench cooling. Once the flask was cooled, the samples were retrieved from the flask and necessary finishing was done (Color plate 1)

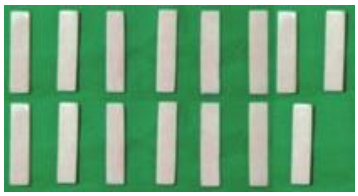


Fig 1: Color plate 1 - Control Group A

Group B: 15 Samples of group B were reinforced with Stainless steel mesh. Before reinforcing stainless steel mesh, it was sandblasted using 50-250 micrometer Al2O3 with a 5.5 bar pressure in a conventional sandblasting device. Sandblasted meshes were cut into15 pieces of length of 60mm ×6mm (Color plate 2). The flask which had mould space were kept ready for packing, the mixed resin which was reached in dough stage packed between the two halves of the mould, sandblasted mesh was kept on one half of the flask followed by trial closure was done, excess flash was removed and final closure was done under a bench press. Specimens were processed as explained for Group A. (Color plate 3)

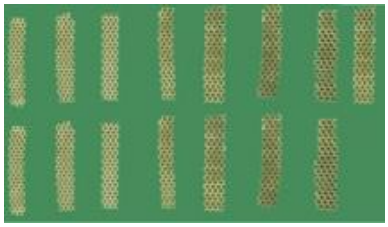


Fig 2: Color plate 2 - Sectioned Stainless-Steel mesh

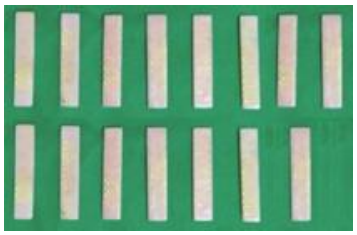


Fig 3: Color plate 3 - Group B Stainless Steel mesh

Group C: 15 Samples of group C were reinforced with Glass fibre meshes which were provided in pre impregnated resin form. It was cut into15 pieces of length 60mm×6mm (Color plate 4). The flask which had mould space were kept ready for packing, the mixed resin which was reached in dough stage packed between the two halves of the mould, glass fiber mesh has wetted with monomer and placed in between the two halves of mould followed by trial closure, excess flash was removed and final closure was done under a bench press. Specimens were processed as explained for Group A. (Color plate 5)

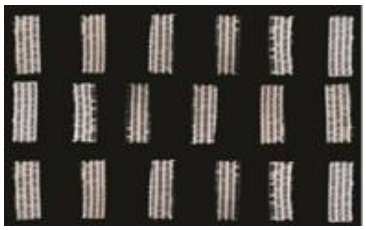


Fig 4: Color plate 4 - Sectioned Glass Fibre Mesh (60mm×6mm)

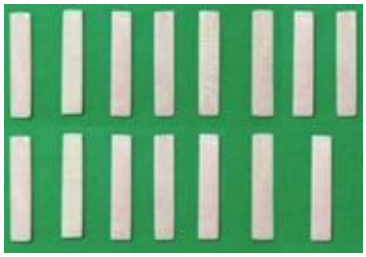


Fig 5: Color plate 5 – Group C Glass Fibre Reinforcement

Group D: 15 Samples of group D were reinforced with 1wt% of Silanized alumina nanoparticles. Silanized alumina nanoparticles were mixed by magnetic stirrer for 20 minutes with 10ml of monomer for dispersion of nanomaterial to the monomer. Then quick mixing was done with 21 g powder to prevent agglomeration. When it reached dough stage, taken into the mould space and packed, followed by trial closure, excess flash was

removed and final closure was done under a bench press. Specimens were processed as explained for group A. (Color plate 6)

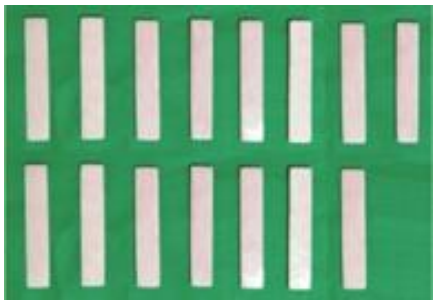


Fig 6: Color plate 6 – Group D Silanized Alumina Nanoparticles Reinforcement

Group E: 15 Samples of group E were reinforced with 0.5wt% of MWCNT. It was mixed by magnetic stirrer for 20 minutes with 10ml of monomer for uniform dispersion. Then quick mixing was done with 21 g powder to prevent agglomeration. When it reaches dough stage, taken into the mould space and packed followed by trial closure and excess flash was removed and final closure was done under a bench press. Specimens were processed as explained for group A. (Color plate 7)

Fig 7: Color plate 7 – Group E MWCNTs Reinforcement

Finishing and polishing of samples:

After curing, all the 75 samples from Group A to Group E were retrieved from the flask and necessary finishing was done with sandpaper of 120grit and polished with felt cone in slow speed. Minimal finishing was required and care was taken to maintain low heat during the procedure. All specimen thickness and width were cross examined with a digital caliper to maintain the standardization of specimen dimension advised by ADA specifications; 65x10x2.5mm. Specimens were stored in water at 370 C for 7 days before subjecting it to transverse strength.

Evaluation of Transverse Strength: (Color plate 8)

The specimens were tested for Transverse strength with a three point-bending test using universal testing machine at a crosshead speed of 5mm/min and span length of 50mm. A standard three point bending jig was attached to the machine and connected to specifically designed software installed in the machine. This software controlled the testing machine and recorded the breakage load and beam deflection. The specimens were then placed on the jig and the test was carried out using a crosshead speed of 5mm/min for transverse strength. The load was applied centrally on the bar specimen until fracture occurred. The maximum load at which fracture took place was noted for all the 75 specimens of group A to group B.

Color plate 8 -Test For Transverse strength under Progress

Sample size estimation:

By using G* Power Software version 3.1 on anticipating Kohen's effects size of (f) 0.5 from previous study with an α(type I) error 0.05 and probability of β type II error 0.20 and the power is 80% the minimum number of sample size was calculated as 11 per group. In order to minimize sampling error sample size was increased to 15 per group. Experimental design consists of 5 groups, so total of 75 specimens were fabricated.

RESULTS

Observations:

The present invitro study was conducted to evaluate and compare the transverse strength of unmodified resin and modified resin test groups, with Stainless steel mesh, Glass fiber mesh, Silanized alumina nanoparticles and multiwalled carbon nanotubes. The values of peak load that were obtained during the fracture of tested specimen from the universal testing machine were tabulated in master chart. Master chart for five groups containing 15 specimens in each group were prepared for statistical evaluation.

Statistical analysis:

All the data were translated in to computerized data base structure. Statistical analyses were done using SPSS version 16. The data were subjected to Shapiro-Wilk test for testing the Normality. Homogeneity of variance assumption was tested by using levene statistic homogeneity of variance. All the variables were following a normal distribution, (P>0.05); hence a parametric evaluation was adopted. Descriptive statistics is a summary statistic that quantitatively describes or summarizes features of a collection of information. Here descriptive statistics were carried out for the 5 test groups. The mean value, standard deviation, 95% confidence interval for mean with lower and upper bound level of the transverse strength between control group and reinforcement groups were calculated and summarized. **(Table 1)**

Table no 7 shows the standard deviation of the group A was 5.41, lower bound level 97.31MPa and upper bound level 103.30MPa. For the group B standard deviation was 5.98, lower bound level 104.08 MPa and upper bound level 110.71MPa. For the group C standard deviation was 8.14, lower bound level 131.44MPa and upper bound level 140.45MPa. For the group D Standard deviation was 5.94, lower bound level 121.35MPa and upper bound level 127.93MPa. For the group E standard deviation was 4.39, lower bound level 180.36MPa and upper bound level 185.22MPa.

Graph 1 shows mean and standard deviation of Group A- control group was 100.3MPa, Group B reinforced with stainless steel mesh was 107.39 MPa, Group C reinforced with glass fibre mesh was 135.95MPa, Group D reinforced with silanized alumina nanoparticles was 124.64MPa, Group E reinforced with multiwalled carbon nanotubes was 182.79MPa.

The data were analyzed by using a one–way analysis of variance (ANOVA) (**Table 2)** to identify statistically significance of group A to group E. ANOVA shows sum of squares between groups was 63650.139, Df was 4, Mean square was 15912.535, f value was 427.928 and p value <0.001. ANOVA shows sum of squares within groups was 2602.954, Df was 70, Mean square was 37.185. There was a significant difference between all the five groups at the $p < 0.001$ level [F (4, 70) = 427.93, p <0.001]. There was a significant influence of the reinforcement on the transverse strength of heat cure acrylic resin compared with the control group and reinforced groups. Highest mean value of transverse strength was shown by group E i.e; Carbon nanotubes reinforcement followed by Glass fibre mesh and then Silanized alumina nanoparticles and Stainless-steel mesh. The control group without any reinforcement exhibited the lowest strength value. The subsequent multiple comparisons between the different reinforcement groups were performed by Post-Hoc comparison using Bonferroni correction test kept in a significance level set at p<0.005, Not significant at p value >0.005 and Highly significant at p value <0.005.

Table 1: Descriptive statistics (n=15) shows mean, standard deviation, 95% confidence interval for mean, minimum and maximum value of group a to group e)

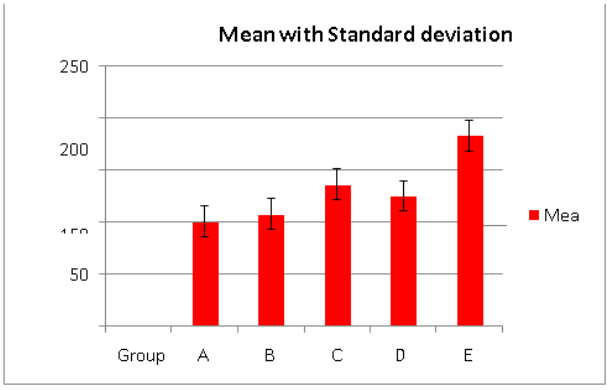
Group	Mean	Std. Deviation	95% Confidence Interval for Mean		Minimum	Maximum
			Lower Bound	Upper Bound		
A	100.30	5.41	97.31	103.30	89.46	107.00
B	107.39	5.98	104.08	110.71	98.17	117.95
C	135.95	8.14	131.44	140.45	120.07	147.62
D	124.64	5.94	121.35	127.93	114.54	135.84
E	182.79	4.39	180.36	185.22	175.99	188.70

Graph shows mean and standard deviation of Group A- control group was 100.3MPa, Group B reinforced with stainless steel mesh was 107.39 MP a, Group C reinforced with glass fibre mesh was 135.95MPa, Group D reinforced with silanized alumina nanoparticles was 124.64MPa, Group E reinforced

with multiwalled carbon nanotubes was 182.79MPa.

Table 2: Statistical comparison (one way anova) shows transverse strength between the groups and within groups.

ANOVA					
	Sum of Squares	Df	Mean Square	F	P value
Between Groups	63650.139	4	15912.535	427.928	<0.001
Within Groups	2602.954	70	37.185		
Total	66253.093	74			



Graph 1: Mean and standard deviation of Transverse strength (MPa) of control group and the reinforced groups.

DISCUSSION

One of the most common reinforcing techniques used in the laboratory was incorporation of metal mesh in the prosthesis. The primary problem of this technique was poor adhesion between the resin and mesh. Mechanical retention of PMMA to the surface of a metal caused by the penetration and interlocking of the resin into the macro or micro irregularities of the metal. The irregularities can be achieved by methods such as grinding, sandblasting, and electrolytic or chemical etching63,64,65. So a micro mechanical retention was available for resin matrix, however the study revealed that there was an increase of transverse strength but it is not much significant when compared with the unmodified resin group. Disadvantage of mesh reinforcement was aesthetically not acceptable, difficult to repair because of mesh protruding from the fracture site and lead to a stress concentrated area which act as a crack initiator66.

The other factors that relate to the strength of the fiber composite are quantity of fibers in polymer matrix and orientation of fibers. Vallitu et al19evaluated the effect of fibre concentration on fracture resistance of acrylic resin and observed better enhancement in fracture resistance of resin modified materials at higher concentration of glass fibres. Sang-Hui et al46 also mentioned that the concentration of the glass fibre mesh was more important than the structure. The orientation of glass fibres should be perpendicular to the loading force could enhance the mechanical properties.

In this study, preimpregenated silanized glass fibre mesh (dentapreg mesh) was used for the reinforcement (group C). The results and statistical analysis revealed a significant increase in the transverse strength of PMMA when compared with group A,B&D. Factors that mentioned above are almost followed for the preparation of specimen so the transverse strength was improved. Jaikumar et al67 found that acrylic resin specimens reinforced with glass fibers showed high flexural strength compared to high impact denture so the study agreement with present study, that improvement of transverse strength. Glass fibres show excellent esthetic properties and biocompatibility also so it can be a choice of material for reinforcement.

Alumina addition leads to both increased and decreased flexural strength depending upon the amount and size of the particles added. Different particle size has been used by various researchers which lead to variable results. However, it has been observed that range of particle size is more effective than uniform size of particles. Nanofillers are more effective at lower concentrations as compared to micro fillers50. From the previous studies43,44 they suggested 1wt% of silanized alumina for the better distribution and effectiveness. Improved flexural strength is attributed to uniform distribution of the filler particles within the matrix and transformation toughening. When sufficient stress develops and micro-cracks begin to propagate, the transformation phenomenon occurs, which depletes energy for crack propagation. Therefore, proper distribution of the filler within the matrix can stop or deflect cracks. Somkaur et al51 suggested to use the magnetic stirrer for proper mixing and dispersion of fillers to monomers.

Nanofillers treated with silane coupling agent leads to marked increase in properties of acrylic resin as compared to untreated particles. Various researchers such as Jasim BS et al (2014)43, Chaijareenont P et al (2012)68 have recommended the use of the silane coupling agent, MPS (3-methacryloxy propyl trimethoxysilane).

So the group D shows a significant improvement of transverse strength when compared with group A &B. Through Mean value comparison it was almost near to group C. Studies also reported**43,68** that the improvement of other properties like thermal conductivity, water solubility, surface hardness by the incorporation of silanized alumina nanoparticles. But the material was expensive.

Carbon nanotubes incorporation was a recent chemicomechanical reinforcement to improve the mechanical properties of denture base resin. In terms of high surface area, nanotubes consist of long cylinders with a hollow cavity at their centre they exhibit increase surface area compared with nanoparticles. Because of their high surface area to volume ratio, increases the interfacial interaction between nanomaterials with the resin matrix. The atomically smoothen surface of the nanotubes reduces interfacial adhesion with the resin matrix as well as the tendency for agglomeration which causes poor distribution of load throughout the matrix69. Hence good dispersion and enhancement of linking forces affect the reinforcing effect of CNTs. Sonication was one of the types of dispersion formula for CNTs, so in this study magnetic stirrer was used for proper dispersion.

Based on the previous studies45,48 the concentrations of CNTs recommended were 0.5% and 1%, if the concentration was more than that it shows adverse effect on the fatigue resistance and it should not improve the transverse strength. So in this study 0.5% concentration of MWCNTs were used for reinforcement. The results revealed that the higher transverse strength was shown by MWCNTs incorporated group specimen and it shows significant difference from other reinforcement groups.

Result of this study in an agreement with previous studies51,56,57 regarding the incorporation of MWCNTs. Naji et al56 reported that the open-ended tubular structure of the nanotubes may allow the MMA monomer to enter into the tubes by capillary action and undergoes polymerization. Thus the higher degree of cross linking leads to increased load transfer within the nanotubes resin composite. Crack bridging in the tubular structures of nanotubes by fiber pullout from the matrix has been the main reason for improvement of the mechanical properties by arresting the early phase of crack propagation.

The major disadvantage of adding carbon nanotubes to acrylic resin is, it makes the acrylic resin black and it will make the denture base unesthetic. It is advised to use carbon nanotubes incorporated resin in midpalatine region of the maxillary denture and lingual aspect of in mandibular denture, as these are the areas more prone to fracture, thus strength of the denture can be enhanced without compromising the esthetics of the patient.

The limitation of the present in vitro study was that specimens were not subjected to thermocycling. The invitro nature of this study makes it different from in vivo conditions. The release of stresses during finishing and polishing procedures were not considered. However, all test specimens of each group were prepared, cured, finished and polished following the standard and uniform protocol. The transverse strength carried out in this study utilizes static loading on the specimens however natures of forces in the mouth are dynamic. Discoloration of MWCNT specimens is also one of the limitations.

Nanomaterial incorporation and its release in oral environment, biocompatibility and Cytotoxicity level are still unknown. Well controlled clinical studies and further in–vitro studies are necessary to correlate the findings and examine those variables that influence the fatigue behavior of denture polymers. Fatigue testing of these materials under dynamic loading using the denture base configurations in simulated oral conditions, using saliva or its substitutes is an area for further research.

When the entire spectrum of this study is analyzed, it becomes evident that the denture reinforcement carried out with MWCNTs gives higher transverse strength than any other compared test specimens and same time due to the unesthetic appearance of MWCNTs specimens not preferable for entire

fabrication of complete denture. So fracture prone areas can be incorporated with MWCNTs.

Similarly, in this study shows a significant improvement in transverse strength by preimpregenated glass fibre mesh reinforcement, so it advisable option for the reinforcement. It does not alter the esthetics of the denture and provide good strength over other reinforcement. Though this method is slightly expensive in comparison to traditional methods but can prevent the frequent fracture of denture to a greater extent.

CONCLUSION

Within the limitation of this in- vitro study and the basis of results obtained; it may be concluded that the transverse strength values of heat polymerized PMMA were considerably enhanced by reinforcements. PMMA reinforced with MWCNTs showed the highest transverse strength value followed by the PMMA reinforced with glass fibre mesh, then silanized alumina nanoparticles and stainless-steel mesh. The least transverse strength was observed in the unmodified PMMA group. Transverse strength of stainless-steel mesh reinforcement group was not statistically significant compared with unreinforced group.

CONFLICTS OF INTEREST

The authors declare they have no potential conflict of interests regarding this article.

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