Original Article

Comparison of the flexural strength of polymethyl methacrylate resin reinforced with multiwalled carbon nanotubes and processed by conventional water bath technique and microwave polymerization

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Abstract

Purpose: This *in vitro* study was done to compare the flexural strength of polymethyl methacrylate resin reinforced with multiwalled carbon nanotubes (MWCNTs) and processed by conventional water bath technique and using microwave energy.

Materials and Methods: A total of 180 acrylic resin specimens measuring 65 mm × 10 mm × 2.5 mm were fabricated, with conventional water bath groups and microwave group having ninety specimens each. Ninety specimens were divided into thirty specimens as control and subgroups containing 0.025% MWCNTs and 0.050% MWCNTs with thirty specimens each. The specimens were tested for flexural strength by three-point bending test on universal testing machine. The statistical analysis was done using Student's *t*-test and one-way analysis of variance, and the intercomparison between each group was done using Tukey's *post hoc* analysis.

Results: The mean flexural strength of specimens cured by water bath technique was 95.563 MPa and microwave technique was 118.416 MPa. Control Group B possesses highly significant increase in flexural strength than Control Group A with $P < 0.01$. Unpaired Student's *t*-test showed that Subgroup B1 and Subgroup B2 possess highly significant increase in flexural strength than Subgroup A1and Subgroup A2.

Conclusion: Heat polymerized denture base resin with and without reinforcement of MWCNTs and polymerized by microwave technique possess higher flexural strength than heat polymerized fiber reinforced denture resin polymerized by water bath technique. MWCNTs could be used as an effective reinforcement material for denture base resin polymerized by either water bath technique or microwave energy.

Keywords: Flexural strength, heat polymerized resin, microwave polymerization, multi-walled carbon nanotubes, polymethyl methacrylate

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INTRODUCTION

Currently, polymethyl methacrylate (PMMA) is the material of choice for denture base fabrication. Despite the excellent properties of the material, there is a need for improvement in the fracture resistance of PMMA.[1] Primarily, due to resin fatigue during function, the dentures most often undergo fracture inside the mouth. Flexural fatigue often leads to the midline fracture of the denture.[2] Various types of materials such as beads or fibers, carbon, glass, aramid, polyethylene, PMMA, metal inserts, plates, and meshes had been incorporated into acrylic resin to improve its mechanical properties.[3-8] Although the chemical modification of acrylic resin was successful with the incorporation of rubber in the form of butadiene, the incorporation of rubber had its effect on the modulus of elasticity and hence the rigidity of the denture base was affected. Various materials are marketed as high strength resins, but these are expensive in comparison to conventional heat-cured denture base resin.[9,10]

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. They are artificial novel nanomaterial belongs to the third allotropic form of carbon, i.e., fullerenes family. 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).[11,12] SWCNTs consist of a single graphene cylinder and usually occur as hexagonal close-packed bundles, with diameter varying between 0.4 and 2 nm. Multi-walled carbon nanotubes (MWCNTs) were made of two to many coaxial cylinders, and each cylinder is made of a single graphene sheet surrounding a hollow core. The inner diameter of MWCNTs ranges in between 1 and 3 nm and outer diameter ranges in between 2 and 100 nm and their length varies in between 0.2 to several micrometers.[12,13] CNTs had tensile strengths 4000 times stronger than steel and almost 200 times stronger than carbon fibers. The carbon matrix formed by CNTs and PMMA was very large, with a greater bond and thus the compressive strength and mechanical fatigue strength were enhanced. Incorporation of CNTs with PMMA resins improves the strength of the prostheses and they can withstand the masticatory forces in a better way.[14,15] CNTs incorporated in PMMA resin prevent polymerization shrinkage and dimensional changes in the resin and help in better adaptation of the denture bases. Hence, the augmentation of nanotubes in acrylic resins will improve the mechanical properties of the acrylic, eliminating the need for metal reinforcement in stress-bearing areas.[16] The effect of CNTs on living cells is still being studied, and till now, no known adverse effects have been reported.[17]

For polymerization of acrylic resins, various curing techniques and denture base materials containing modifications of PMMA have been developed.[18] The conventional method of processing denture base polymers is done in a water bath by polymerizing the dough molding of monomer and polymer. Other processing techniques used are microwave energy or light curing. Although the conventional method has been considered as the best means of processing heat-cured denture resins, it takes relatively long time to cure the material, and also it is not a very clean procedure. The processing of denture with microwave is quite cleaner, less time taking, homogeneous mix of the material with excellent adaptation, and less residual monomer.[19,20]

This *in vitro* study was done to compare the flexural strength of PMMA resin reinforced with MWCNTs and processed by two techniques - conventional water bath technique and using microwave energy. The null hypothesis of the study was (a) there was no difference in the flexural strength of PMMA denture base resin reinforced with MWCNTs; (b) there was no difference in the flexural strength of denture base materials polymerized by conventional curing method and polymerized by microwave technique with and without reinforcement with MWCNTs; and (c) there was no difference in the flexural strength of denture base material reinforced with different percentages of MWCNTs polymerized by two different techniques.

MATERIALS AND METHODS

The study was undertaken in the Department of Prosthodontics with supporting technical assistance from Central Institute of Plastics Engineering and Technology and Centre for Scientific Research and Development. Ethical clearance for the study was obtained from Ethical Committee of the Institute. Heat-cured acrylic resin specimens measuring 65 mm × 10 mm × 2.5 mm were fabricated according to American Dental Association Specification No. 12.[21] The experimental design included two main groups, with each group having three subgroups [Figure 1]. A total of 180 specimens were fabricated with each groups containing 90 specimens, further divided into thirty specimens in each subgroup.

Specimen fabrication

A specially designed mold was used for the fabrication of the specimens [Figure 2].[22] The mold was lubricated with petroleum jelly to facilitate easy removal of the

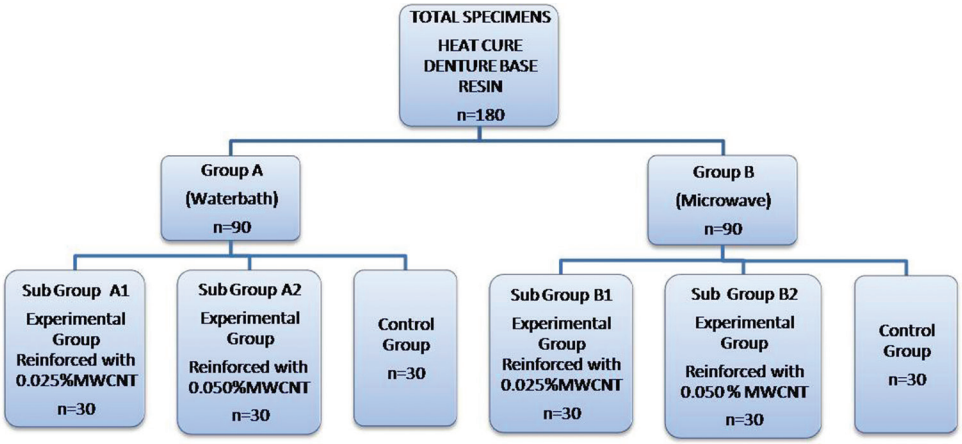

Figure 1: Flow chart showing specimen distribution among groups

wax specimens. The lower and the middle plates of the mold were assembled together, and the modeling wax (DPI, Mumbai, India) was melted and poured into the mold. The upper cover plate was placed and screws were tightened to remove excess wax and the mold was allowed to chill. The upper plate was unscrewed, and excess wax was removed using a sharp Bard Parker blade. The solidified wax specimens were removed from the mold and prepared for flasking. The final specimens were uniform in all dimensions; distorted, damaged, or broken patterns were discarded.

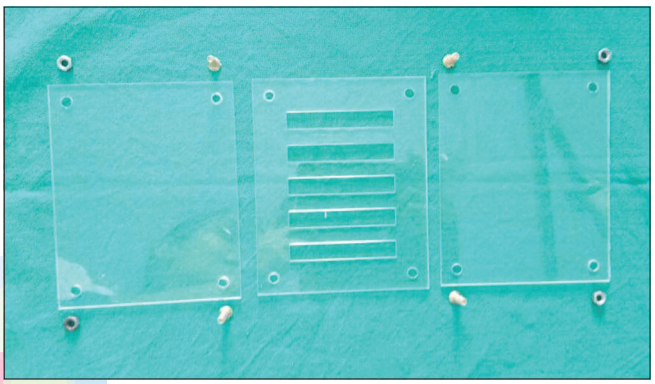

Figure 2: The three piece standardized mold

Specimens processed using conventional heat polymerization

The wax pattern specimens for conventional water bath polymerization were invested with dental stone (Type III, Kalrock, Kalabhai, Mumbai, India) in brass flasks and were allowed to set for 1 h. The flask assemblies were placed in the dewaxing unit for 8 min and then they were separated and thoroughly flushed with hot water to remove any residual wax. The molds were cleaned with soap water and dried in open air. A thin, single uniform layer of separating medium (Coe-Sep, GC Acro-Sep, Europe) was applied to stone on both the parts of the flask while the mold was still warm. For control group, specimen's heat-cure acrylic resin (Trevalon HI, Dentsply, Mumbai, India) was mixed in a porcelain jar in the ratio of 21 g polymer: 10 ml monomer as per manufacturer's instructions. For experimental group, the MWCNTs (Nano Green Technologies LLP Gwalior, India) [Figure 3 and Table 1] were added to the measured acrylic monomer at 0.025% wt/wt (Subgroup A1) and 0.050% wt/wt (Sub Group A2), in a glass beaker. Liquid monomer was then stirred with magnetic stirrer for around 15 min. The prepared monomer was added to heat cure acrylic resin in a ratio of 10 ml monomer: 21 g polymer. The mixes were allowed to reach the dough stage and were then kneaded and packed in the mold. The flasks were

Figure 3: Multiwalled carbon nanotubes

reassembled and placed into a bench press and trial closure was done at 1500 psi with uniform pressure application and excess flash was removed. The final closure was done at 2750 psi[23] and the flasks were bench cured for 1 h. The flasks were immersed in an acrylizer and curing was done at 74°C for 2 h followed by 100°C for 1 h. The flasks were removed from the water bath and before deflasking they were kept overnight at room temperature for bench cooling.

Table 1: Multi-walled carbon nanotubes

MWCNT	Description	Characterization method
Production method	Chemical vapor deposition	Proprietary method
Available form	Black powder	Visual
Diameter	Average outer diameter 20 nm	TEM, SEM
Length	Average 20 um	SEM, TEM
Nanotubes purity	>98%	TGA, Raman
Metal particles	<1%	TGA
Amorphous carbon	<1%	TGA, XRD
Specific surface area	330 m^2/g	BET
Bulk density	0.020-0.035	Pycnometer

SEM: Scanning electron microscopy, XRD: X-ray powder diffraction, TEM: Transmission electron microscopy, TGA: Thermogravimetric analysis, BET: Brunauer-Emmett-Teller, MWCNT: Multi-walled carbon nanotube

Specimens processed using microwave polymerization

The wax pattern specimens for microwave polymerization were invested in specially fabricated fiber-reinforced plastic flasks (Acron MC, GC, USA) [Figure 4]. The removable plates of the flasks were inserted into the bottom of flasks. A coating of Vaseline was applied to the inside of the flask and the escape holes. The flasks were invested with dental stone and were allowed to set for 1 h. The dewaxing was done by keeping the flasks in the microwave oven (Whirlpool India Pvt. Ltd.) at 500 W for 1 min.[2,9] The flasks were opened and the softened wax was flushed with hot water. The molds were cleaned with soap water and dried in open air. While the mold was still warm, a layer of separating medium was applied. For control group, specimen's heat-cure acrylic resin was mixed in a porcelain jar in the ratio of 21 g polymer: 10 ml monomer as per manufacturer's instructions. For experimental group, the MWCNTs were added to the measured acrylic monomer at 0.025% wt/wt (Subgroup B1) and 0.050% wt/wt (Subgroup B2), in a glass beaker. Liquid monomer was then stirred with magnetic stirrer for around 15 min. The prepared monomer was added to heat cure acrylic resin in a ratio of 10 ml monomer: 21 g polymer. The mixes were allowed to reach the dough stage and were then kneaded and packed in the mold. The flasks were reassembled and placed into a bench press and trial closure was done at 1500 psi with uniform pressure application and excess flash was removed. The final closure was done at 2750 psi[23] and the flasks were bench cured for 1 h. The flasks were kept in the microwave oven and cured for 3 min at 500 W.

After polymerization, the flasks were allowed to cool slowly at room temperature for 30 min. Subsequently, the flasks were immersed in cool tap water for 15 min. After complete cooling, the specimens were carefully retrieved and finished. The specimens were sequentially polished with silicon carbide paper (1000, 800, and 600 grit) to achieve smooth edges [Figure 5] and stored in distilled water at 37°C ± 1°C in an incubator for 48 h. The accuracy of the dimensions

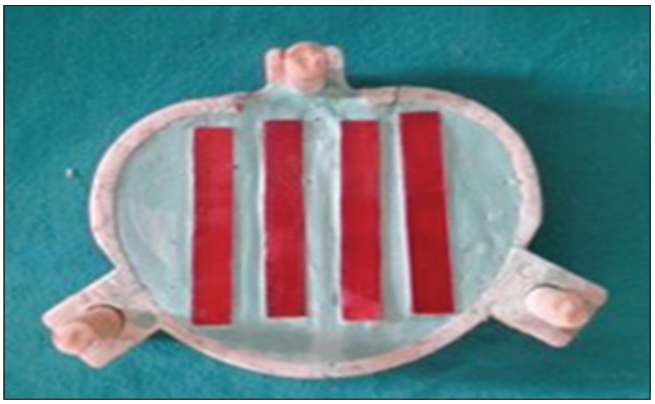

Figure 4: Microwave specimens invested in a specially fabricated fibre reinforced plastic flask

of the specimens was verified with digital Vernier caliper, at three locations of each dimension to within 0.2 mm tolerance.

The specimens were tested for flexural strength by 3-point bending test on universal testing machine ((Instron Corporation, Canton, MA, USA)) [Figure 6] at a crosshead speed of 2 mm/min. The peak load (fracture load) was recorded in chart recorder. The peak load is converted to flexural strength by the formula: $S = 3PL/2bd$

Where S = flexural strength (N/mm^2); P = load at fracture; L = distance between jig supports; b = specimen width; d = specimen thickness.

The data obtained were subjected to statistical analysis using Statistical Package for the Social Sciences (SPSS Version 20; Chicago Inc., IL, USA). Data comparison was done by applying specific statistical tests to find out the statistical significance of the comparisons. Quantitative variables were compared using mean values and qualitative variables using proportions. The mean for different readings between the two groups and subgroups was compared using Student's *t*-test and one-way analysis of variance (ANOVA), and the intercomparison between each group was done using Tukey's *post hoc* analysis.

RESULTS

The mean flexural strength among Control Group A, Subgroup A1, and Subgroup A2 was presented in Table 2. Mean flexural strength of Control Group A was 84.601 MPa, Subgroup A1 was 94.651 MPa, and Subgroup A2 was 107.507 MPa. One-way ANOVA showed that progressive reinforcement of MWCNTs significantly improves the flexural strength with $P < 0.01$. Unpaired Student's *t*-test showed that, with an increase in the concentration of MWCNTs from 0.025% to 0.05%, there

was a highly significant increase in flexural strength with $P < 0.01$ [Table 3]. Tukey's *post hoc* analysis showed that both the subgroups possess higher flexural strength than control group and Subgroup A2 possess statistically significantly higher flexural strength than Subgroup A1 [Table 4].

The mean flexural strength among Control Group B, Subgroup B1, and Subgroup B2 was presented in Table 5. Mean flexural strength of Control Group B was 92.622 MPa, Subgroup B1 was 130.881 MPa, and Subgroup B2 was 131.742 MPa. One way ANOVA showed that progressive reinforcement of MWCNTs significantly improves the flexural strength with $P < 0.01$. Unpaired Student's *t*-test showed that, with an increase in the concentration of MWCNTs from 0.025% to 0.05%, there was no significant increase in flexural strength with $P > 0.05$ [Table 3]. The Tukey's *post hoc* analysis showed that both the microwave subgroups possess higher flexural strength than Control Group B, but there was no statistically significant rise in flexural strength in between Subgroup B2 and Subgroup B1 [Table 4].

The mean flexural strength of specimens cured by water bath technique was 95.563 MPa and microwave technique was 118.416 MPa. Unpaired Student's *t*-test showed that specimens cured by microwave energy possess highly significant increase in flexural strength than conventional water bath technique with $P < 0.01$ [Table 6]. Control Group B possesses highly significant increase in flexural strength than Control Group A with $P < 0.01$. Unpaired Student's *t*-test showed that Subgroup B1 and Subgroup B2 possess highly significant increase in flexural strength than Subgroup A1 and Subgroup A2. Specimens cured by microwave energy with MWCNTs possess higher strength than specimens cured by water bath technique with $P < 0.01$ [Table 7].

Table 2: Mean flexural strength among Control Group A, Subgroup A1, and Subgroup A2

Groups	*n*	Flexural strength (Mpa), mean±SD	ANOVA *F*	*P*
Control Group A	30	84.601±3.048	1615.902	0.001
Subgroup A1	30	94.651±2.374		
Subgroup A2	30	107.507±2.022		
Total	90	88.025±15.657		

$P > 0.05$ - nonsignificant, $P < 0.05$ - significant, $P < 0.01$ - highly significant, $P < 0.001$ - very highly significant. SD: Standard deviation, ANOVA: Analysis of variance

DISCUSSION

The PMMA denture base material becomes popular due to easy processing of the material, less cost, light in weight, less water absorption and solubility, easy to repair, and excellent esthetic properties. There are certain disadvantages of the material which make it prone to failure such as they had low thermal conductivity, reduced mechanical strength; brittle in nature, coefficient of thermal expansion is high, and having low modulus of elasticity.[24] There was usually two types of failures, first is caused due to high stresses outside the mouth and second is due to repeated low stresses inside the mouth.[1,25] Factors such as a frenum notch, reproduction of rugae, and scratches on the denture base alter the stress distribution in the denture base and predispose the denture to fracture.[26]

Several conventional methods have been proposed for the polymerization of heat-cured PMMA to simplify the technique and reduction in time, but similar advantages were obtained using microwave energy for denture processing.[27] Kimura *et al.* discovered the technique of curing acrylic resins by microwave energy, and it was reported that it curing of acrylic resin can be done in a very short time with this technique.[28]

Combination of compressive, tensile, and shear strengths denotes the flexural strength of a material. The force required to fracture the material increases as the tensile and compressive strengths increase. Three-point bending test simulates the type of stress applied to the denture during mastication, so it is used to find the flexural strength of a material. There are two methods for preventing the fracture of the denture, first is to strengthen the denture base material and the second is to reduction of stresses at the midline.[29] Various denture designs have been made to reduce stress at the midline such as increasing the thickness of the denture base, changes in teeth arrangement, and strengthener can be used. There were certain limitations such as if the thickness of denture base increased, it reduces the tongue space and affects speech. The artificial posterior teeth must be arranged with consideration of not only the shape of the maxillary and mandibular residual ridge but also the relationship between them.[30]

Table 3: Mean flexural strength among Subgroup A1 and A2 and among Subgroup B1 and B2

Groups	*n*	Flexural strength (mean±SD)	Mean difference	Unpaired Student's *t*-test value	*P*
Subgroup A1	30	94.651±2.374	12.856	22.578	0.001
Subgroup A2	30	107.507±2.022			
Subgroup B1	30	130.881±3.080	0.883	1.247	0.433
Subgroup B2	30	131.742±2.197			

$P > 0.05$ - nonsignificant, $P < 0.05$ - significant, $P < 0.01$ - highly significant, $P < 0.001$ - very highly significant. SD: Standard deviation

The present study was undertaken to evaluate the flexural strength of heat polymerized denture base material reinforced with MWCNTs and processed by two different polymerization techniques that is conventional water bath technique and microwave energy. Shlosberg *et al*.[31] studied the conventional and microwave methods of polymerization and found that both methods produced similar dimensional accuracy in complete denture bases. There were no differences in transverse strength, Knoop hardness, density, and residual monomer content of test resin strips. Alkhatib *et al*.[32] compared two microwave and one water bath polymerized resins and found no significant differences in flexural strengths or hardness between the materials regardless of the polymerization method used.[33] Other researchers found higher flexural strength for microwave-processed acrylic resins.[27,34,35]

Table 4: Tukey's *post hoc* analysis for intragroup comparison for conventional group and microwave group

Groups	Mean difference in flexure strength	*P*
Subgroup A1 versus Subgroup A2	12.856	0.001
Subgroup A1 versus Control Group A	10.049	0.001
Subgroup A2 versus Control Group A	22.905	0.001
Subgroup B1 versus Subgroup B2	0.861333	0.424
Subgroup B1 versus Control Group B	38.259333	0.001
Subgroup B2 versus Control Group B	39.120667	0.001

P>0.05 - nonsignificant, *P*<0.05 - significant, *P*<0.01 - highly significant, *P*<0.001 - very highly significant

Table 5: Mean flexural strength among Control Group B, Subgroup B1, and Subgroup B2

Groups	*n*	Flexural strength (Mpa), mean±SD	ANOVA *F*	*P*
Control Group B	30	92.622±2.615	2123.100	0.001
Subgroup B1	30	130.881±3.080		
Subgroup B2	30	131.742±2.197		
Total	90	118.415±18.531		

P>0.05 - nonsignificant, *P*<0.05 - significant, *P*<0.01 - highly significant, *P*<0.001 - very highly significant. SD: Standard deviation, ANOVA: Analysis of variance

The present study also found that the specimens cured by microwave energy exhibit higher flexural strength than conventional heat polymerized specimens. With the conventional method, the temperature rises at the end of the curing cycle, and some free monomer is left in the resin. Microwaves act only on the monomer, which decreases in the same proportion as the polymerization degree increases. Therefore, the same amount of energy is absorbed by less and less monomer, making the molecules increasingly active. This is important because a form of self-regulation of the curing program takes place and leads to complete polymerization of the resin.[36]

The reason for the decrease in flexural strength of denture base resin with conventional methods may be in this technique the methyl methacrylate boils and creates porosities in the denture base resin and these porosities lead to the formation of stress and cause propagation of cracks within the acrylic. In microwave technique, the methyl methacrylate molecules orient themselves in the electromagnetic field of the microwave and the polymerization heat is dissipated more effectively and the polymerization has a lesser risk of porosity. Moreover, as the temperature increases, the number of monomer molecules decrease, and the residual monomer content is reduced to minimum and thus they had highest flexural fatigue strength.[35]

In the literature, it has already been proven that addition of various quantities of CNTs to acrylic resin improves various properties such as flexural strength, impact strength, and polymerization shrinkage.[16,36,37] There is no documented evidence on evaluation of flexural strength of heat cure denture base resin reinforced with MWCNTs processed through microwave energy. Hence, the present study was done to compare the effect of reinforcement of MWCNT's on flexural strength of heat cure denture

Table 6: Mean flexural strength comparison between water bath and microwave technique among all groups

Groups	*n*	Flexural strength (Mpa), mean±SD	Mean difference	Unpaired Student's *t*-test value	*P*
Water bath technique	90	95.563±9.719	22.853	10.360	0.001
Microwave technique	90	118.416±18.532			

P>0.05 - nonsignificant, *P*<0.05 - significant, *P*<0.01 - highly significant, *P*<0.001 - very highly significant. SD: Standard deviation

Table 7: Mean flexural strength comparison between water bath and microwave technique among Control Group A and B, among Sub Group A1 and B1 (0.025% multi-walled carbon nanotube) and among Subgroup A2 and B2 (0.05% multi-walled carbon nanotube)

Groups	*n*	Flexural strength (Mpa), mean±SD	Mean difference	Unpaired Student's *t*-test value	*P*
Control Group A	30	84.601667±3.0482613	8.02033	10.937	0.001
Control Group B	30	92.622000±2.6156800			
Subgroup A1	30	94.651000±2.3744609	36.2303	51.025	0.001
Subgroup B1	30	130.88133±3.0801508			
Subgroup A2	30	107.43866±1.9874463	24.3040	44.924	0.001
Subgroup B2	30	131.74266±2.1978625			

P>0.05 - nonsignificant, *P*<0.05 - significant, *P*<0.01 - highly significant, *P*<0.001 - very highly significant. SD: Standard deviation

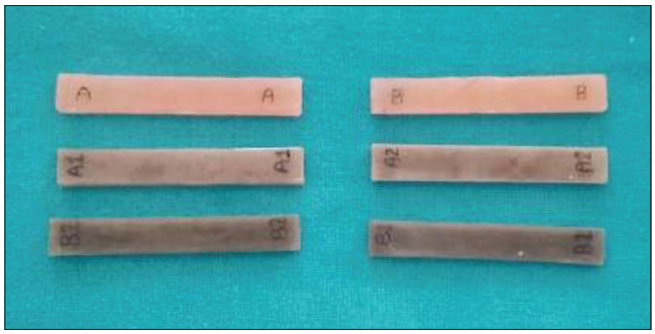

Figure 5: Fabricated specimens

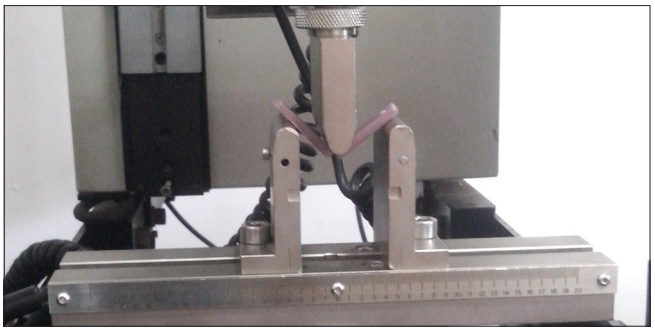

Figure 6: Specimen under 3-point bending test

base resin processed by conventional water bath technique and microwave technique. The results clearly showed the highly significant difference in flexural strength of specimens prepared by adding 0.025% and 0.05% MWCNTs processed through water bath technique and the result is similar to the study conducted by Wang *et al.*[38] and Mahmood.[39] Although flexural strength of denture base resin processed through microwave energy increases with addition of 0.025% and 0.05% MWCNTs, there are no statistically significant differences observed. However, there is highly significant difference in flexural strength of specimens cured through microwave energy and water bath technique and the specimens cured through microwave energy possess higher flexural strength than water bath technique. Overall result showed that flexural strength of MWCNTs reinforced denture base material has higher flexural strength than fiber reinforced denture base material and strength increases with increase in percentage of MWCNTs processed through either water bath technique or microwave energy.

Nanoscaled particles exhibit an enormous surface area and larger in magnitude than the surface of conventional fillers. This surface area is responsible for CNTs to form agglomerates and also acts as interface for stress transfer.[4] The clinical implications of this study suggest that micro-additions of CNTs in PMMA resins can produce denture base resins with higher flexural strength based on percentage of CNTs added. This study has also shown that microwave energy can efficiently polymerize denture base polymer with and without addition of MWCNTs. Polymerization of denture base resin using microwave energy may have a positive effect on the strength and longevity of complete dentures. Furthermore, microwave energy has a potential for saving a great amount of time in processing dentures.[27]

The major disadvantage of adding carbon nanotubes to acrylic resin is, it makes the acrylic resin black and it will make the denture base unaesthetic. It is advised to use carbon nanotube 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,[40] 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 only one type of resin is used for polymerization of both microwave and water bath technique. Discoloration of specimens as the percentage of MWCNTs increases was not measured.

CONCLUSION

Within the limitations of this study, it could be concluded that heat polymerized denture base resin with and without reinforcement of MWCNTs and polymerized by microwave technique possess higher flexural strength than heat polymerized fiber reinforced denture resin polymerized by water bath technique. MWCNTs could be used as an effective reinforcement material for denture base resin polymerized by either water bath technique or microwave energy.

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Nil.

Conflicts of interest
There are no conflicts of interest.

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