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RESEARCH ARTICLE

COMPARATIVE EVALUATION OF MULTIWALL CARBON NANOFILLERS AND GRAPHENE ON THE IMPACT STRENGTH AND FLEXURAL STRENGTH OF AUTO POLYMERIZED ACRYLIC RESIN

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ABSTRACT

Context: Polymethylmethacrylate (PMMA) is widely used for fabrication of removable dental prosthesis, but it is still insufficient to fulfil the mechanical requirements for dental applications.
Aims: Comparative evaluation of addition of carbon nano fillers and graphene and its effect on impact strength and flexural strength of Autopolymerized Acrylic resin. - an in vitro study
Methods and Materials: Characterisation of carbon nanofillers (MWCNTs) and graphene were done to check the purity, diameter and size using FT RAMAN & FESEM. Monomer without microaddition acted as control group. Nanofillers by weight were added in monomer in different percentages 0.50% by weight for group b and group c and combination of 0.25% by weight of graphene and 0.25% by weight of carbon for group d. Nanofillers were subjected to probe sonification apparatus for uniform dispersion. Polymer and monomer containing nanofillers were mixed according to the groups and packed in the mold and processed according to conventional method.80 specimens were fabricated and divided into four groups according to the test.The retrieved specimens were kept in artificial saliva before testing. Universal maching and izod impact testing machine was used to check impact and flexural strength.
Statistical analysis used: Kruskall Wallis ANOVA and Mann Whitney test.
Results: 0.5% by weight of carbon nanofillers showed highest flexural and impact strength of PMMA resin followed by graphene and by combination of 0.25% by weight of carbon and0.25% by weight of graphene nanofillers.

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INTRODUCTION

Polymethyl methacrylate (PMMA) is widely used as denture base material.PMMA has mechanical properties such as hardness, rigidity, biological properties, aesthetic properties (Peyton, 1975). One of the most common complications of denture base prosthesis is fracture. In earlier studies, fracture rate was reported to be 68 % (Hargreaves, 1969). These fractures may occur inside or outside the mouth due to expelling the denture from the mouth while coughing, or simply dropping it. Other reasons could be excessive bite force, improper occlusal plane, high frenal attachment, lack of balanced occlusion, poor fit and poor quality of the denture base material, long-term fatigue failure caused by repeated masticatory force or from extra-oral high impact force resulting from accidental dropping of the prosthesis (Eick, 1977). Risk of fracture due to accidental fall of dentures is more, so high impact strength is a desirable property.

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Flexural and impact strength are important properties which are essential for strength and to increase the longevity of prosthesis (Anusavice *et al.,* 2013). Flexural fatigue occurs after repeated flexing of a material (Diaz-Arnold *et al.,* 2008). The midline fracture in dentures is often the result of flexural fatigue (Diaz-Arnold *et al.,* 2008). When the patient exhibits parafunctional habits such as bruxism and clenching, the flexural strength is an essential property. Autopolymerizing resin has gained more popularity due to its easy handling, saving chairside time, and no laboratory processing; moreover, the patient spends less time without denture during the repair process. Since fabrication of a new denture is time-consuming and costly for patients, denture repair is considered an alternative (Stipho and Stipho, 1987). Repaired dentures should have adequate strength, dimensional stability and color match; moreover, the repair should be easily and quickly performed and must be affordable (Rached *et al.,* 2004). Amongst various methods proposed for repairing fractured denture bases, use of auto-polymerized acrylic resins, which generally allows a simple and quick repair, is considered the most popular method but dentures repaired with cold cure

acrylic resin broke at the repaired site, which may be due to the lower strength of cold cure acrylic resin (Stipho and Stipho, 1987). In order to overcome these problems and increase the longevity and durability of the prosthesis, several attempts were made to modify and improve the strength of the PMMA like zirconia, glass fibre, alumina, tin, and copper or addition of whisker to resin like tin oxide(TiO2),zinc oxide (ZnO2), aluminium oxide(AlO2) (Ellakwa *et al.,* 2008; Kim *et al.,* 2004). Recently, much attention has been directed toward the incorporation of filler particles into cold cure resin to improve its properties. MWCNTs (Multiwall Carbon Nanotubes)can successfully reinforce the fracture lines by strengthening the fibrils and bridging voids to enhance the fatigue performance of the polymer (Xia *et al.,* 2008; Ormsby *et al.,* 2010). Graphene has attracted great interest due to its exceptional physical, chemical, thermal and electrical properties (Huang *et al.,* 2011). Combination of graphene and MWCNTs has never been used in literature. Hence, aim of this in vitro study was comparitive evaluation of effect of MWCNTs (Multiwall Carbon Nanotubes) and graphene on flexural strength and impact strength of cold cure resin.

MATERIALS AND METHODS

A total 80 specimens were fabricated for testing two parameters i.e. impact strength & flexural strength and were divided into 4 groups (Table.1).10 samples for each group (n= 10) for flexural strength. 10 samples for each group (n= 10) for impact strength

Table 1. Grouping Of Specimens

Group A (Control group)	Heat cure denture base resin without incorporation of nano fillers
GROUP B	Incorporation of 0.5% by weight of carbon Nano fillers in autopolymerizing denture base resin.
GROUP C	Incorporation of 0.5% by weight of graphene in autopolymerizing denture base resin.
GROUP D	Incorporation of 0.25% of carbon Nano fillers and 0.25% of graphene in autopolymerizing denture base resin.

Fig. 1a. Fabricated molds for flexural strength

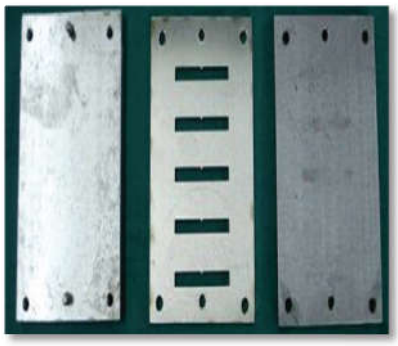


Fig.1b.Impact strength

Preparation of stainless steel mold

The molds were fabricated for flexural strength consisted of stainless steel plates of 2mm thickness with ten slots of dimension 60x40x2 mm (length x width x thickness) according to (ISO20795) (International Organization for Standardization) (Fig.1 a). Mold for fabrication of samples for impact strength consisting of 5 slots were made up of dimension 80 X 10 X 4mm (length x width x thickness) (ISO20795) (International Organization for Standardization) (Fig.1.b). Graphene and carbon nanofiller (MWCNTs) solution (Fig 2) used in this study were in ethanol dispersed form. MWCNT and graphene nanoparticles were characterized with the help of Fourier Transform Raman (FT RAMAN) &Field Emission Scanning Electron Microscopy (FE-SEM)(fig3 &4).Graphs of MWCNTs (Graph.1)and Graphene (graph.2) were verified for purity of the nanofillers.FE-SEM image of MWCNTs (Fig.5) and graphene (Fig.6) verified the size of nanoparticles. FE-SEM aids in examining small area of contamination spots.FT-RAMAN provides a fingerprint by which the molecule can be identified and also the purity of the nanofillers.

Fig. 2. Graphene and Multiwall Carbon nanotubes solution- United Nanotech Innovations Pvt.Ltd Bangalore

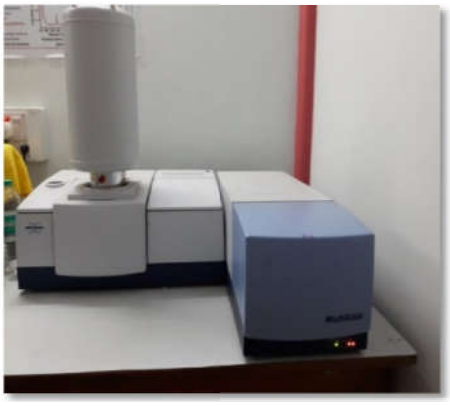


Fig.3 FT RAMAN (Bruker's MultiRAM)

Preparation of acrylic denture base resin specimens

Electronic precision balance (Fig.7) was used to weigh 50 gm of cold cure acrylic resin - DPI cold Cure- Dental Products of India Ltd Mumbai, India, (Fig.8) 20ml of monomer was measured using calibrated beaker. Monomer without micro additions of nanoparticles were mixed with polymer (PMMA) in standard 1:2 ratio by weight acted as group a (control group). For fabrication of samples of group B and C 0.5% by weight of MWCNTs (multiwall carbon nanotubes) i.e. 0.083 µl

Fig.4.FE-SEM (JEOL JSM 5800; JOEl Ltd, Tokyo, Japan)

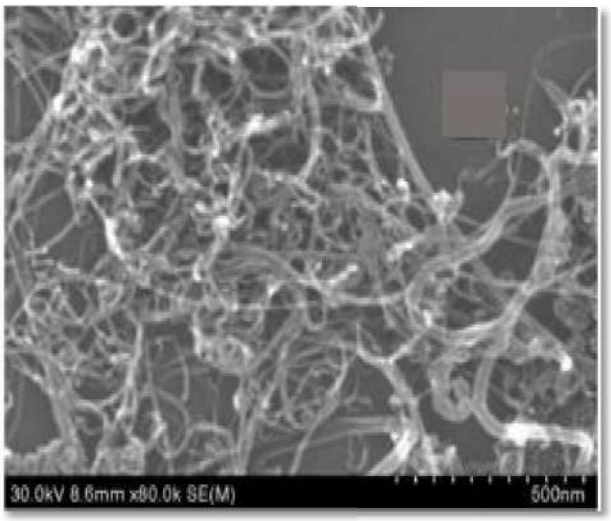


Fig. 5. FE-SEM images of MWCNT's after characterisation

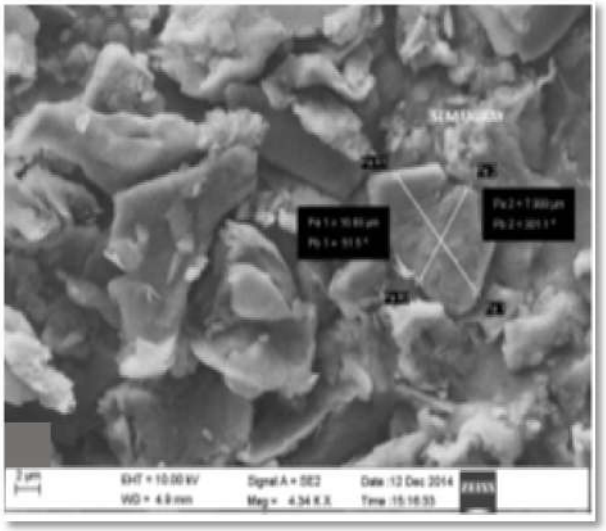
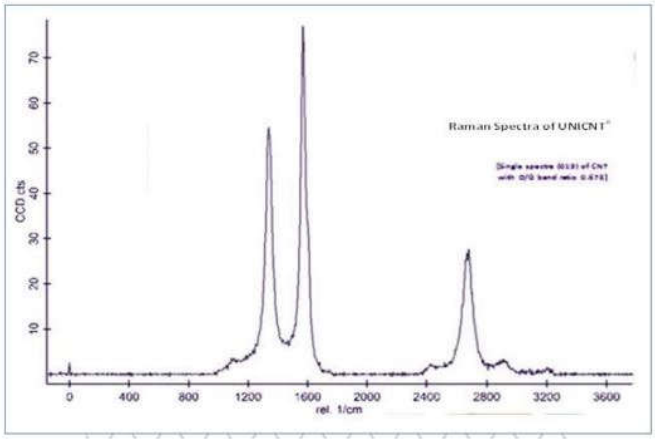
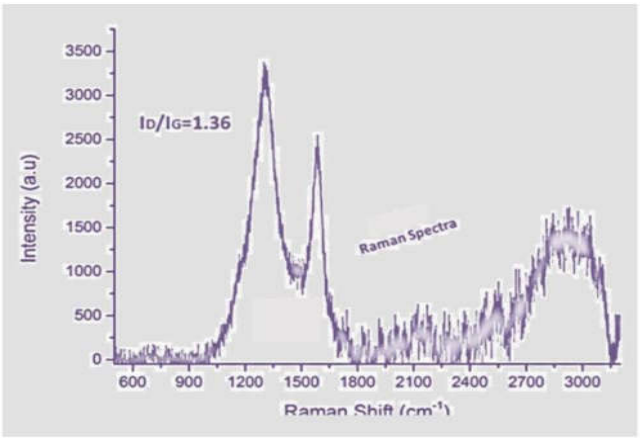


Fig. 6. FE-SEM images of graphene after characterisation



Graph1.FT RAMAN spectroscopy of MWCNT' safter characterization



Graph 2. FT RAMAN spectroscopy of graphene after characterization

Table 2. Percentages and amounts of polymer, monomer and carbon (MWCNTs) and graphene Nano filler used in the study

Percentage of Nano fillers	Amount of Nano fillersin µl	Amount of PMMA	Amount of monomer
0.5% Carbon Nano fillers	0.083 µl	5gm	2ml
0.5%Graphene Nano fillers	0.0108µl	5gm	2ml
0.25%Carbon Nano fillers	0.042 µl	5gm	2ml
0.25%Graphene Nano fillers	0.054µl	5gm	2ml

Fig.7.Electron precision balance(Hindustan Scientific Linkers)

Fig.8.Cold cure acrylic resin - DPI cold Cure- Dental Products of India Ltd,Mumbai, India

Fig.9.Nanofillers measured using Micropipette

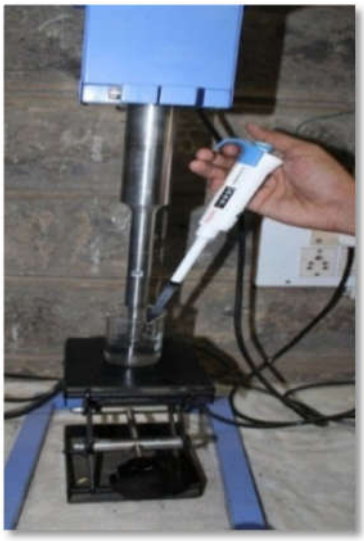


Fig.10.Nanofillers added to the beaker

Fig.11. Ultra probe sonication

Fig.12.Mixing of the mixture

and 0.5% by weight graphene i.e.0.0108 µl were measured using micropipette (Fig.9) and 0.25% by weight of MWCNTs (multiwall carbon nanotubes) i.e. 0.042 µl & 0.25% by weight of graphene i.e. 0.054 µl respectively for group D as mentioned in (Table.2). Measured nanofillers were added to a beaker containing monomer (Fig.10) Graphene particles and MWCNTs (multiwall carbon nanotubes) would be subjected to ultra probe sonication at 140W, 40 KHz for 3mins according to the groups (Fig.11) Probe sonication apparatus (Ultrasonicator Apparatus, *Pci Analytics* Pvt. Ltd)is used for breaking them into individual nano crystal and for uniform dispersion in the monomer (methyl methacrylate) to prevent agglomeration Monomer containing MWCNTs (multiwall carbon nanotubes) and graphene of different concentration by weight of respective groups were mixed with polymer (PMMA) in standard 1:2 ratio by weight (Fig.12). The jar was kept closed till the mix attained the dough stage. Petroleum jelly was applied to the porcelain mixing jar and molds. The resin dough was removed from the mixing jar and loaded in the mold. The mold was reassembled and bench pressed incrementally pressure was applied 1500 psi until metal to metal contact of the mold was achieved. The mold was clamped and kept for three hours for polymerisation. After polymerisationthe specimens were retrieved and any excess flash was trimmed off and finishing of samples was done (Fig.13).

Artificial saliva (Spinco Biotech Pvt. Ltd, Chennai) was used to remove any impurity from the samples and to standardize the procedure. Samples were kept in artificial saliva for 48hrs before testing (Fig.14). Before testing the samples for impact and flexural strength, the samples were evaluated for uniform dispersion of nano fillers in PMMA resin with the help of FE-SEM. (Fig.15). Four study groups, each contained about 20 specimens, with different concentration of nanoparticle in specimens and control groupfor flexural strength and impact strength (Fig.16, 17). For flexural strength, Universal Testing Machine, Star Testing System, India. Model No. STS 248(Fig.19) was used. The distance between the specimen supports was 40 mm and the loading force was applied to the specimens at a crosshead speed of 5 mm/min until the specimens fractured. The maximum load exerted on the specimens was recorded. Impact strength test was conducted following the procedure given by the (ISO20795) (International Organization for Standardization) with Izod impact testing machine (Fig.20). Data was subject to statistical analysis using Statistical package for social sciences (SPSS v 22.0, IBM).Comparison of differences in means of flexural and impact strengths between the 4 groups was done using Kruskall Wallis ANOVA, followed by Mann Whitney test for pair-wise comparisons. For all the statistical tests, $p<0.05$ was considered to be statistically significant, keeping α error at 5% and β error at 20%, thus giving a power to the study as 80%.

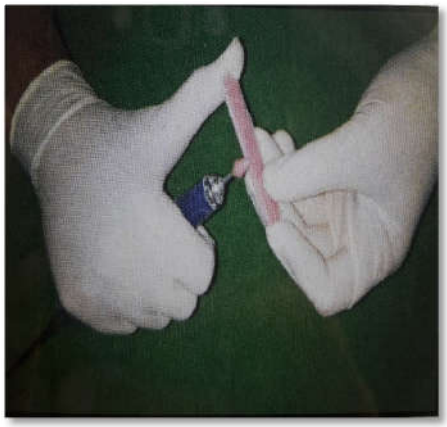


Fig. 13.Finishing of the specimens

Fig.14.Samples kept in artificial saliva

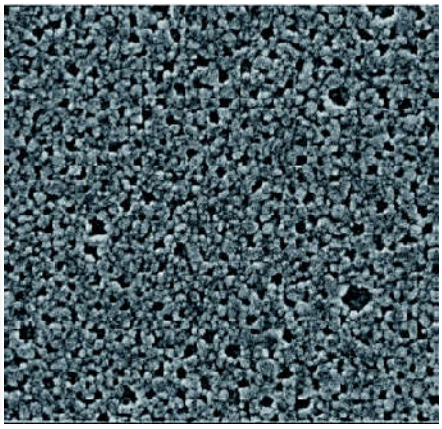


Fig.15.FESEM images of dispersed nanofillers in resin

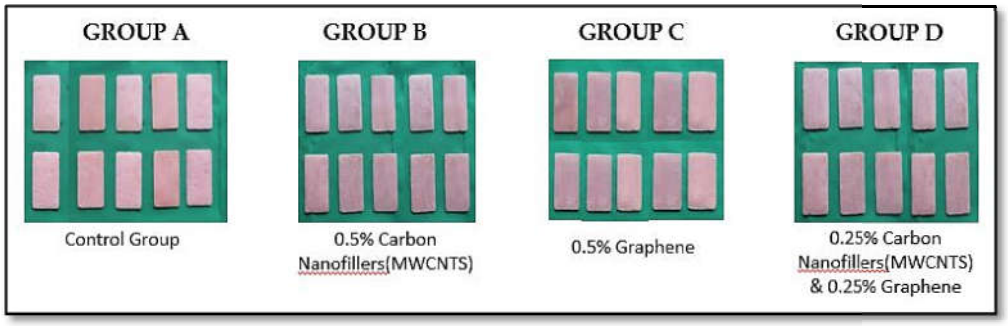


Fig.16.Samples Of Different Group for flexural strength

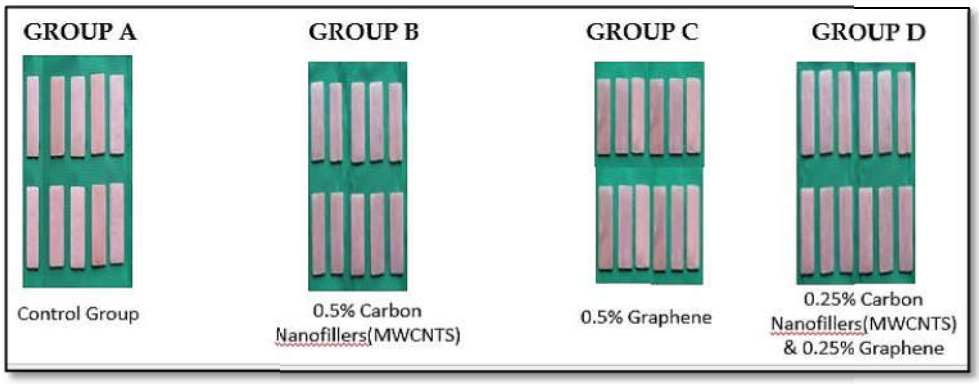


Fig.17.Samples Of Different Group for impact strength

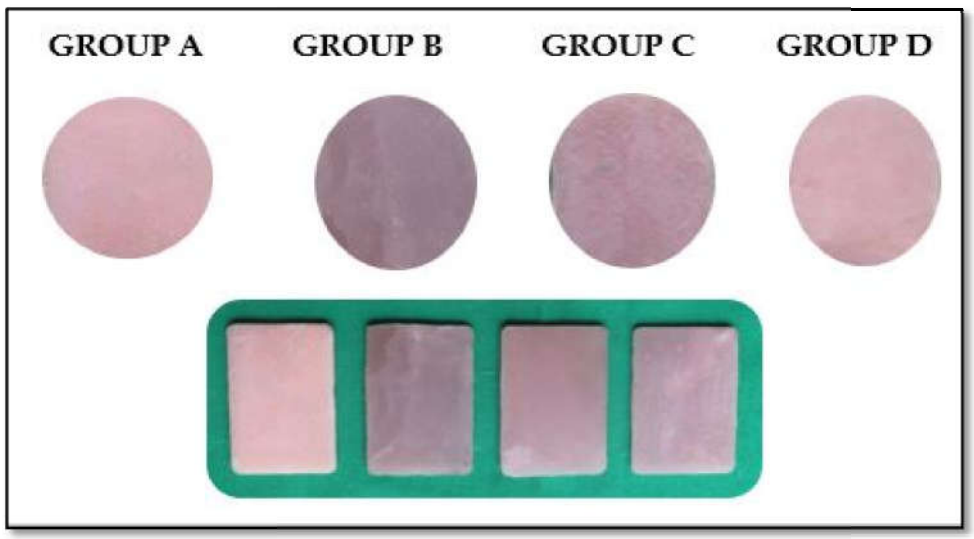


Fig.18.Samples Of Different Group showing color variation

RESULTS

Higher flexural strength were of samples of Group B (cold cure resin incorporated with 0.5% of carbon nanofillers (MWCNTs) by weight) 69.86 Mpa followed by Group C (cold cure resin incorporated with 0.5% of graphene by weight) 63.66Mpa, Group D (cold cureresin incorporated with0.25 % of carbon nanofillers (MWCNTs) by weight and 0.25% of graphene by weight) 60.95 MpaGroup A control group 58.29 Mpa. There is a highly significant difference between the means of different groups ($p<0.001$).

The mean impact strength of all group specimens as tabulated in (Table 4)demonstrate higher impact strength for Group B (cold curebase resin incorporated with 0.5% of carbon. nanofillers (MWCNTs) by weight) 11.62 kJ/m^2 followed by Group D (cold cureresin incorporated with0.25 % of carbon nanofillers (MWCNTs)by weight and 0.25% of graphene by weight)9.90 kJ/m^2, Group C (cold cureresin incorporated with 0.5% of graphene by weight)8.92 kJ/m^2 and Group A, control group 7.95 kJ/m^2. There is a highly significant difference between the means of different groups ($p<0.001$).

Fig. 19. Universal testing machine for Three-point flexural strength test

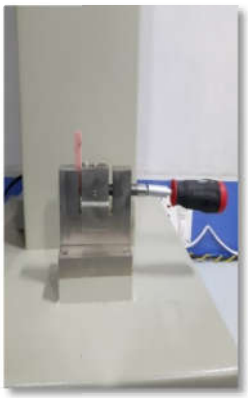


Fig 20.Izod Impact Testing Machine for Izod impact strength test

Table 3. Comparison of mean flexural strengths of four groups evaluated by Kruskal-Wallis test

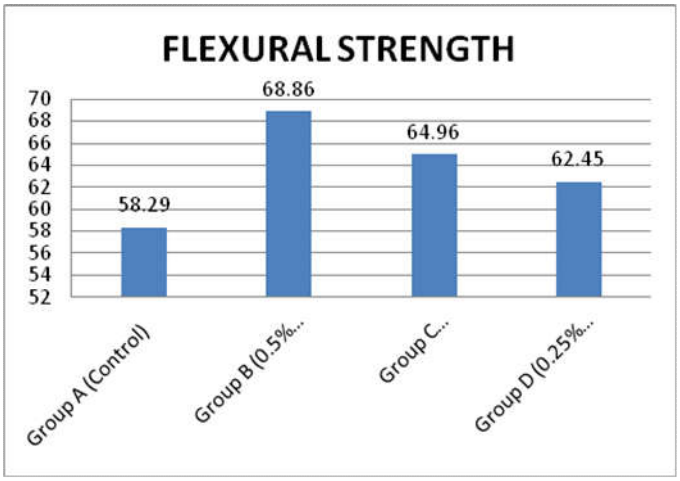
Flexural strength	Groups	N	Mean (Mpa)	Std. Deviation	p value by Kruskal-Wallis ANOVA
	Group A(Control)	10	57.29	1.862	
	Group B (0.5% MWCNTs)	10	69.86	.889	
	Group C (0.5%Graphene)	10	63.66	.950	‹ 0.001**
	Group D (0.25%MWCNTs + 0.25%Graphene)	10	60.95	.935	HS

Table 4.Comparison of impact strength of four groups evaluated by Kruskal-Wallis test

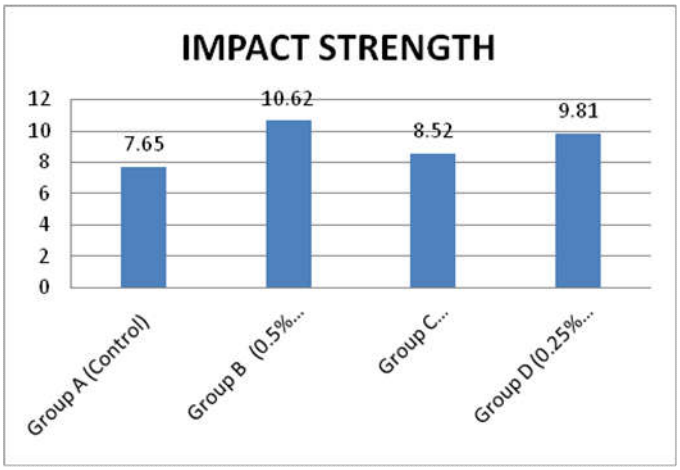
Impact Strength	Groups	N	Mean (kJ/m$^{2)}$	Std. Deviation	p value by Kruskal-Wallis ANOVA
	Group A (Control)	10	7.95	.487	
	Group B (0.5% MWCNTs)	10	11.62	.878	
	Group C (0.5%Graphene)	10	8.92	.456	‹ 0.001**
	Group D (0.25%MWCNTs + 0.25%Graphene)	10	9.90	.643	HS

Table 5. Comparison of differences in means of flexural and impact strengths between the four groups by Mann Whitney test for pair-wise comparisons

	Groups	N	Mean	Std. Deviation	Std. Error	p value of Mann Whitney test
Flexural Strength	Group A (Control)	10	58.29	.854	Group A Group B	0.000**
	Group B (0.5% MWCNTs)	10	67.86	1.092	Group A Group C	0.000**
	Group C (0.5%Graphene)	10	63.96	.533	Group A Group D	0.028*
	Group D (0.25%MWCNTs+ 0.25%Graphene)	10	60.45	.889	Group B Group C	0.000**
					Group B Group D	0.000**
					Group C Group D	0.033*
Impact Strength	Group A (Control)	10	7.95	.299	Group A Group B	0.000**
	Group B (0.5% MWCNTs)	10	11.62	.178	Group A Group C	0.024*
	Group C (0.5%Graphene)	10	8.92	.194	Group A Group D	0.000**
	Group D (0.25%MWCNTs+ 0.25%Graphene)	10	9.90	.224	Group B Group C	0.000**
					Group B Group D	0.001**
					Group C Group D	0.000**



Graph 3. Mean Flexural Strength among groups (Mpa)



Graph 4. Mean Impact Strength among groups (kJ/m2)

DISCUSSION

PMMA has been established as principal material in denture base construction. Nevertheless it is generally recognized that despite fulfilling aesthetic requirements, the fracture strength of PMMA are not entirely satisfactory and this is reflected by the expenditure on a large number of denture repairs annually (Jagger *et al.,* 2013). Most fractures of dentures occur during function, primarily from denture resin fatigue. Because of the risk of fracture, if patients drop their dentures, high impact strength is a desirable property. Recently, much attention has been directed toward the incorporation of inorganic nanoparticles into PMMA to improve its properties. Nanomaterials have been developed promptly and some researches of nanomaterials have been carried out in prosthodontics. Many of the current dental materials are available through nano crystallization to improve their original performance and play a key role in oral applications.

Nano composite denture base has higher interfacial shear bond strength between the resin matrix and nanomaterial's, compared to the conventional resin matrix. It creates thick interface, which enhances the bond between the resin molecules and creates higher molecular weight polymers (Zhang *et al.,* 2008). Because of the interface and cross linking polymerisation there is less chances of leaching of residual monomer from the resin. CNTs are strong, resilient, lightweight, and usually form stable cylindrical structures. CNTs have high mechanical properties with reported strengths 10 to100 times higher than steel at a fraction of the weight (Wang *et al.,* 2014). CNTs that have a flawless structure are classified into 2 main types, namely single walled and multiwall CNTs. Single-walled CNTs (SWCNTs) consist of a single graphite sheet seamlessly wrapped into a cylindrical tube, and multiwall CNTs (MWCNTs) have an array of such nanotubes concentrically nested like the rings of a tree trunk. Single-walled CNTs (SWCNTs) are costly and less available. The addition of carbon fibres to a matrix not only gives strength and elasticity to the material but also improves toughness. The stress transfer efficiency can be 10 times higher than that of traditional additives.[15]Multi-walled carbon nanotubes (MWCNTs) have unique atomic structure, and extra ordinary mechanical properties, making ideal reinforcing materials.[16]An efficient exploitation of the CNT properties in polymers is related to their homogenous dispersions in the matrix or an exfoliation of the agglomeration and a good wetting with the polymer. Various dispersions methods (stirring, extrusion, sonication, etc.) for the distributions of CNTs (carbon nanotubes) in polymers have been used. A common technique used in order to disperse CNTs is the sonication's technique (Huang *et al.,* 2011; Wang *et al.,* 2014; Mars *et al.,* 2007). A pulsed ultrasound exfoliates agglomerates and disperses CNTs in the matrix effectively. To take full advantage of these unique mechanical properties, optimization of nanotube-polymer interface properties such as wet ability and adhesion is required. In pure PMMA fibres, polymer necking occurs under increasing tension, which results in failure at relatively small strains. However, adding CNTs to a polymer may dramatically improve the resistance of the polymer to mechanical failure. Incorporating MWCNTs to polymer matrices may effectively bridge cracks and reduce the extent of plastic deformation by a PMMA matrix (Wang *et al.,* 2014). MWCNTs can successfully reinforce the fracture lines by strengthening the fibrils and bridging voids to enhance the fatigue performance of the polymer. In the last years, graphene has attracted great interest due to its exceptional physical, chemical, thermal and electrical properties. This material can be described as a single layer of pure carbon only one atom thick.

Graphene is flexible, practically transparent, very strong and biocompatible. Graphene shows superior mechanical properties including high fracture resistance, excellent mechanical strength, high Young's Modulus (1TPa) (Chen *et al.,* 2011). Furthermore, it is biocompatible, and very light, it has a large surface area, and the reinforcement of denture base material has been a subject of interest to the dental material community. The effects of CNT and graphene reinforcement on some mechanical properties of denture base materials have not been explored (Sodagar *et al.,* 2012). This investigation studied the effect of MWCNT reinforcement on the mechanical properties of a commonly used PMMA denture base material. The null hypothesis that the addition of multiwall carbon nanotubes (MWCNTs) by weight and graphene by weight would not improve the flexural strength and impact strength of the prosthesis was rejected. Multi wall carbon nanotube and graphene was chosen because limited studies are there in literature and also it is known for its high mechanical properties, like flexural strength impact strength, hardness. Multiwall carbon nanotube and graphene was incorporating with concentrations 0.5% by weight of MWCNTs and graphene by weight and combination of 0.25%MNCT and graphene. 0.5% was selected because studies have shown that concentration higher than 0.5% leads to more discoloration of PMMA resin and also it leads to decrease in

strength of the resin.25 Higher concentrations of nano fillers will lead to flexural and impact strength deterioration of the resin material. This is attributed to higher filler content above saturation point at which the resin cannot incorporate further filler particles. Any attempt to add filler particles after reaching saturation of matrix leads to interruption in the resin matrix continuity and thus causing a decrease in the strength of reinforced specimens. These findings are consistent with those reported by (Mars and Pienkowski, 2007; Sodagar *et al.,* 2012).

The increase in impact strength and flexural is due to the interfacial shear strength between nano-filler and matrix is high due to formation of cross-links or supra molecular bonding which cover or shield the Nano fillers that in turn prevent propagation of crack. Mars and Pienkowski (2007) studied that CNT effectively bridge the cracks, also CNT is strong and stable because, carbon in nanotubes are arranged in hexagonal ring. This lead to a reduction in segmental motion thus increasing the impact strength and flexural strength. Nano fillers have tendency to agglomerate when incorporated with heat cure resin directly to prevent this ultrasonification prevent agglomeration of nanoparticles and leads to uniform dispersion of nanoparticles in monomer by using probe ultrasonification apparatus. Agglomeration causes cluster formation of nanoparticles in PMMA resin leading to decrease in strength of the PMMA resin.

The samples were stored in artificial saliva for 48hrs to simulate oral environment and to remove any impurity before testing (Alhareb and Ahmad, 2011). Incorporation of nano fillers increases flexural and impact strength of cold cure resin. Highest impact and flexural strength was shown by incorporation of 0.5% by weight of carbon nano fillers (MWCNTs), but there was more colour change in the specimen compared to other group i.e. 0.5% graphene incorporated in cold cure resin and combination of 0.25% of carbon nanofillers (MWCNTs) and 0.25% of graphene by weight incorporated in cold cure resin (Fig.18). The colour change of group B specimen i.e cold cure resin incorporated with 0.5% by weight of carbon nano fillers (MWCNTs) was more compared to other groups can be used in patients with darker complexion for characterisation. Cold cure resin incorporated with 0.5% of graphene or combination of 0.25% of carbon nanofillers (MWCNTs) and 0.25% of graphene by weight improved flexural strength and impact strength as well as less colour change in the specimen as compared to control group, so they can be used in patients with fair complexion. The results of the present study, especially with respect to impact strength and flexural strength, suggest that the repair strength of auto polymerized resin repairs can be improved significantly through the addition of carbon nanotube and graphene nanofillers.

Conclusion

Within the limitations of this study, following conclusions can be drawn:

- 0.5% by weight of nano fillers increases flexural and impact strength of cold cure resin
- Highest impact and flexural strength was shown by incorporation of 0.5% by weight of carbon nano fillers (MWCNTs), but there was more colour change in specimen compared to other group I.e. 0.5% graphene and combination of 0.25% of carbon nano fillers(MWCNTs) and 0.25% of graphene by weight. but it can be used in patients with darker complexion for characterisation.
- 0.5% of Graphene or combination of 0.25% of carbon nano fillers(MWCNTs) and 0.25% of graphene by weight can be used to increase the flexural and impact strength of cold cure resin as well as in patients with fair complexion.

Further in-vivo studies using these materials are recommended to substantiate these results and for the characterisation of the prosthesis in patient with darker complexion so that ideal and best material can be determined for clinical success.

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