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Effect of Surface Treated Nano diamond Particles and Nano cellulose Fibre Reinforcement on the Flexural and Impact Strength of PMMA

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Abstract

Statement of Problem: Polymethyl methacrylate (PMMA) is a synthetic transparent polymer which is liable to be modified to improve its physical and mechanical properties. Due to poor mechanical properties of PMMA such as low flexural strength and impact strength several attempts have been made to improves the mechanical properties of acrylic resin by providing maximum bulk in the heavily stressed areas, addition of cross linking agents, through copolymerization and by various methods of reinforcement.

Aim: The aim of this study is to evaluate the effect of reinforcement of conventional PMMA with Nano diamond particles, Nanocellulose fibers, and their hybrid on its flexural strength and impact strength.

Methodology: Nanodiamond particles and nanocellulose fibres were surface treated. Nanodiamond was heat treated to get functionalized group on its surface. Nanocellulose was surface treated by TEMPO-mediated oxidation and eco-friendly modification. They were then added to polymethyl methacrylate in different proportions and their flexural and impact strength was evaluated.

Conclusion: The highest impact strength was observed with 1wt% nanocellulose fibre, followed closely by hybrid formulations combining both nanodiamond and

nanocellulose. Conventional PMMA exhibited the lowest impact strength, highlighting the need for reinforcement. Flexural strength improved effectively with the addition of 0.5wt% nanodiamond and hybrid combinations also showed superior performance.

Keywords: Distilled water, Furnace, Nanocellulose fibres, PMMA

Introduction

PMMA is extensively used for fabricating partial or complete edentulous denture bases due to its dimensional stability, aesthetics, biocompatibility and low manufacturing costs. However, the use of PMMA extends beyond fabrication of dentures to interim prostheses, removable orthodontic retainers, occlusal splints, craniofacial surgical splints, and biomimetic scaffolds. These applications are due to its low density, aesthetic qualities, ease of manipulation, and tailorable properties. [1,3,4]

Despite its widespread use, PMMA has several mechanical drawbacks, such as poor impact resistance, low transverse strength, limited flexural and fatigue strength, low surface hardness, and inherent brittleness, leading to fractures—most commonly midline fractures intraorally and impact fractures while it is out of the oral cavity. Its performance can further decline over time due to the absorption of fluids in the oral environment. [1,3,4]

Nano engineering is an attractive and promising way to enhance the performance and properties of polymer. In many studies, the drastic improvement of polymer properties as a result of secondary reinforcement by nanoscale particles (additionally to the usual micro scale fibres or particles) has been demonstrated. A very interesting and promising nanoscale material with the potential to enhance the polymer properties are the nanocellulose fibrils and nanodiamond particles. As differed from often used carbon nanotubes or graphene, the nanocellulose fibrils and nanodiamond particles are natural materials and are widely available.[2,6]

Incorporating nanodiamonds to PMMA improves flexural strength but reduces impact strength, whereas nanocellulose helps greatly in improving impact strength of PMMA. Thus, a hybrid of fibre structure of nanocellulose and particle structure of nanodiamond in different concentrations would reinforce the PMMA matrix for better flexural and impact strength. This study is aimed at analysing the flexural and impact strength of conventional PMMA reinforced with a hybrid of nanodiamonds and nanocellulose fibres.

Materials and Methods

Conventional Poly Methyl Methacrylate (PMMA), High Impact Poly Methyl Methacrylate, Nanodiamonds(ND) particles, Nanocellulose fibres(NCF), Furnace, Electric Mixer, Counter rotating internal mixer, Micro weighing scale [CAS], 2,2'-azobis(2-methylpropionitrile) (AIBN), Tetrahydrofuran (THF), Distilled water, Tempo oxidised cellulose nanofiber, Polyvinyl alcohol (PVA), TEMPO (2,2,6,6-tetramethylpiperidin-1-oxyl), Sodium chlorite, 2 M sodium hypochlorite solution, Tetra ethyloxysilan, 2,2′- Azobis(2-methylpropionitrile) (AIBN), Scanning electron microscope, IZOD impact testing machine .[7]

Specimens were milled with two different dimensions as shown in Fig.1. The set of first milled specimens, were used for testing the flexural strength in accordance with ANSI/ADA specification No.12 for denture base polymers (dimensions 65 × 10 × 2.5 mm3). The second set of milled specimens were used for the impact strength test in accordance with ASTM D- 256 standard and the British Standard Institute Specification (1984) no.771(B.S.I) (dimensions 55 × 10 × 10 mm3 and a standard notch of 2 mm depth at its mid-span).[7]

Surface Treatment of Nanodiamonds: ND particles were heat treated at 450°C for 2 hours in air to produce

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functional groups on their surfaces. Functionalized NDs were then weighed using an electronic balance at 0.25%wt, and 0.5%wt added separately to acrylic resin powder, first manually using conventional glass mortar and pestle with gentle hand pressure. The ND/PMMA composite powder was then stirred with an electric mixer at a rotating speed of 400 rpm at room temperature for 30 minutes to obtain more homogenous and equal distribution of the filler. The morphology of pure ND, PMMA, and dispersion of ND in the PMMA (ND/PMMA mixture) were examined before heat-polymerization by scanning electron microscopy (SEM).[7]

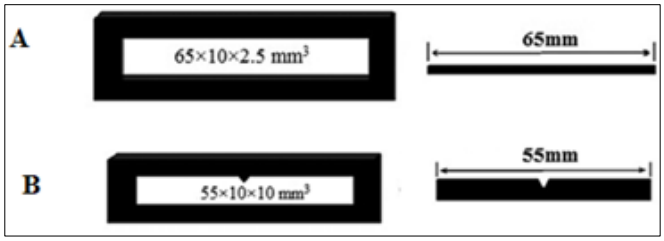

Figure 1: Schematic Diagram of Prepared Specimens.
(A) Specimen for Flexural Strength
(B) Specimen for Impact Strength

Figure 2: Grouping of Tested Specimens for Flexural and Impact Strength

	Group 1	Group 2	Group 3	Group 4	Group 5	Group 6	Group 7	Group 8
Conventional PMMA	100 wt%	0	99.5wt%	99 wt%	98.5 wt%	98.75 wt%	98.75 wt%	99 wt%
High impact PMMA	0	100 wt%	0	0	0	0	0	0
Nano diamond	0	0	0.5 wt%	0	0.5 wt%	0.5 wt%	0.25 wt%	0.25 wt%
Nano cellulose	0	0	0	1 wt%	1 wt%	0.75 wt%	1 wt%	0.75 wt%

Surface Treatment of Nanocellulose Fibres: Nanocellulose fibres was procured from Nano research lab and was surface treated.

TEMPO-mediated oxidation

Nanocellulose fibres (1 g) was suspended in a sodium phosphate buffer (90 mL, pH 6.8) containing TEMPO (0.016 g) and sodium chlorite (80%, 1.13 g) in an airtight flask. 0.5 mL of 2 M sodium hypochlorite solution was diluted to 0.1 M with the same buffer, and then, it was added as the oxidation medium. The TEMPO-mediated oxidation was started, and stirring continued at 60°C for 3.5 hr. After being washed with water, the TEMPO-mediated oxidation and purification were repeated for preparing TEMPO-oxidized NCF (OF). The size of TEMPO-oxidized fibres synthesized was about 55 nm.[8]

Eco-friendly modification of NCF

An Methyl methacrylate monomer, initiator (AIBN), and dispersant (PVA) were mixed according to the ratio of 1:0.05:0.03 in distilled water, and then, OF was added (The amount of Methyl methacrylate monomer relative to the TEMPO-oxidized NCF was 18 mmol Methyl methacrylate monomer /g OF). The solution was stirred continuously at 75 °C for 6 hr under a nitrogen atmosphere. At the end, Methyl methacrylate monomer modified fiber (MF) was obtained after filtration, washing with water, then washing with THF to remove the un-grafting part, and being freeze dried.[8]

Fabrication of nanocomposites (MF+PMMA)

The MF was dried in an oven at 55°C for 1 day and then, mixed in the ratios shown in Figure 2. The NCF, PMMA and PMMA with nanodiamonds were blended at 175°C in a counter-rotating internal mixer with a rotational speed of 50 rpm.

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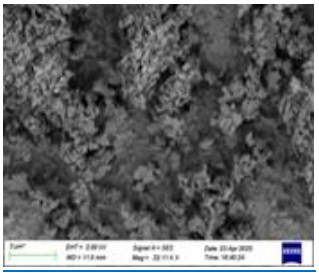

Figure 3: SEM View of Nd Particles.

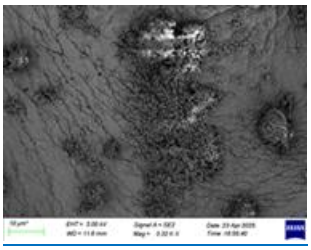

Figure 4: SEM View of Heat-Treated Nd Particles

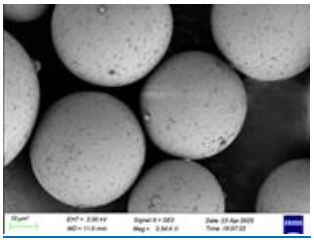

Figure 5: SEM View Of Pmma Mixed With Nd Particles

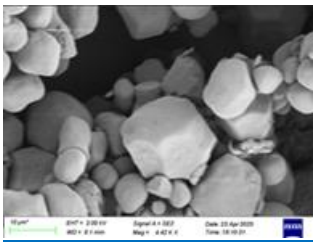

Figure 6: SEM View of Tempo Treated NCF

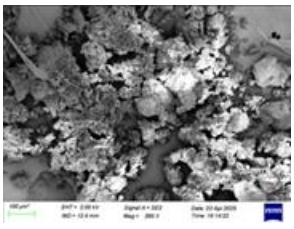

Figure 7: SEM View of Eco-Friendly Modification of NCF

Instrumental Analysis

A scanning electron microscope (SEM) was used to analyse the surface modification before and after surface treatment of nanodiamonds i.e., heat treatment of nanodiamonds and mixing nanodiamonds with PMMA. SEM observation demonstrates the uniform distribution of ND sheets within the PMMA matrix. The pure PMMA matrix shows the clean microspheres of PMMA with dimensions from a few micrometres to a few tens of micrometres. The dispersion of ND was obvious in the ND/PMMA mixture: the ND material was attached with PMMA microspheres.[7]

Tempo treated nanocellulose introduces carboxyl groups into cellulose, enhancing its dispersibility and reactivity. SEM analyses of TEMPO-oxidized cellulose nanofibers (TOCNs) reveal a more individualized and uniform fibril structure compared to native NCFs. The oxidation process disrupts hydrogen bonding, leading to thinner and more separated fibres.

Eco-friendly modification of nano-cellulose fibres as viewed under SEM shows homogeneous distribution and strong interfacial bonding between the components.[8]

Specimens' Fabrication

According to the ANSI/ADA Specification No.12 - 64 cuboidal shape specimens (n=8) with the dimensions of 65*10*2.5 mm3 was designed for testing Flexural strength.

According to ASTM D-256 standard and British Standard Institute specification (1984) no. 771 (B.S.I)- 64 cuboidal shape specimens (n=8) with the dimensions of 55*10*10 mm^3 with a V-shaped notch of 2 mm in the centre was designed for testing Impact strength.

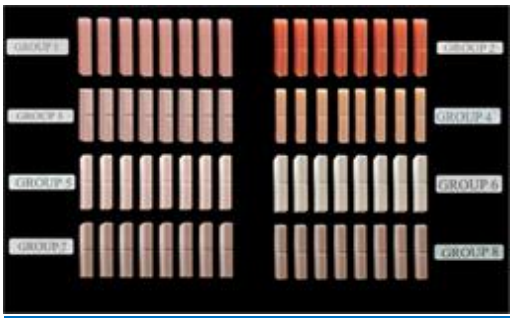

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Figure 8: 64 Nano Diamond And Nano Cellulose Reinforced Pmma Resin Specimens (55 X 10 X10 Mm^3) For Impact Strength Testing

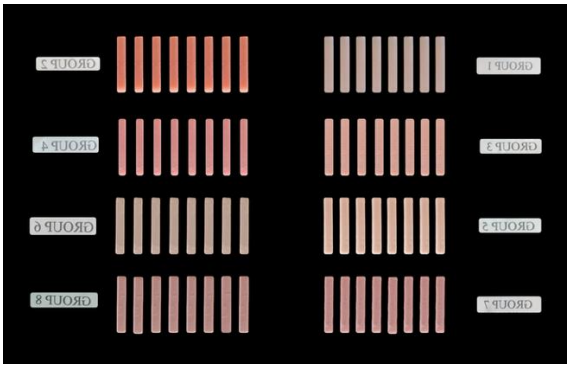

Figure 9: 64 Nano Diamond and Nano Cellulose Reinforced Pmma Resin Specimens (65 X 10 X 2.5 Mm3) For Flexural Strength Testing

Impact Strength Testing

It is determined using a pendulum IZOD-type impact-testing machine (Figure 11). Each specimen was firmly clamped in a vertical position within the testing machine. A pendulum hammer is raised to a specific height and released, striking the notch on the specimen. A drop weight of 0.5 J was applied at the mid-span of the specimen on the opposite side to the notch. The impact strength of each specimen (KJ/m2) of energy absorbed in breaking the specimen) was digitally recorded. The impact strength is measured using the formula:

Impact strength= Impact energy/Specimen thickness

Flexural Strength Testing

Fracture load was measured with a three-point bending test using a universal testing machine (Figure 10). A 50 kgf load cell was applied at the midpoint of the specimen with a 5 mm/min crosshead speed until the specimen fractured.

The flexural strength was calculated using the formula:

$S = 3WI/2bd^2$

(S = flexural strength, W = maximum load before fracture, I = distance between the supports [50 mm], b = width of the specimen, d = depth [thickness] of the specimen).

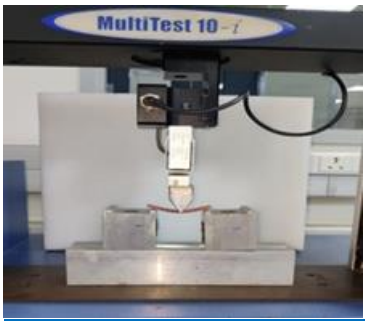

Figure 10: Universal Testing Machine (Mecmesin) For 3-Point Bending Test

Figure 11: IZOD Type Testing Machine (Kiyo) For Testing Impact Strength

Statistical Analysis

- **SPSS (Statistical Package for Social Sciences)** version 20. (IBM SPASS statistics [IBM corp. released 2011] was used to perform the statistical analysis. Descriptive statistics of the explanatory and outcome variables will be calculated by mean, standard deviation/median and IQR (based on normalcy test- Shapiro wilk test) for quantitative variables, frequency and proportion for qualitative variables.
- Inferential statistics like
  - Chi-square test was applied for qualitative variables to find the association.
  - ANOVA test / Kruskal-wallis test (based on data distribution) was applied to compare the mean

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quantitative variables among the groups with post-hoc Bonferroni/ Mann-whitney test (based on data distribution) for inter group comparison.

Comparison of mean Impact Strength (in KJ/m^2) b/w 8 groups using One-way ANOVA Test						
Groups	N	Mean	SD	Min	Max	p-value
Group 1	8	7.55	0.24	7.2	7.9	<0.001*
Group 2	8	8.73	0.26	8.4	9.1	
Group 3	8	7.05	0.24	6.7	7.4	
Group 4	8	9.95	0.24	9.6	10.3	
Group 5	8	9.15	0.24	8.8	9.5	
Group 6	8	7.95	0.24	7.6	8.3	
Group 7	8	9.55	0.24	9.2	9.9	
Group 8	8	8.35	0.24	8.0	8.7	

Comparison of mean Flexural Strength (in MPa) b/w 8 groups using One-way ANOVA Test						
Groups	N	Mean	SD	Min	Max	p-value
Group 1	8	86.50	2.45	83	90	<0.001*
Group 2	8	107.00	1.85	104	109	
Group 3	8	134.50	2.45	131	138	
Group 4	8	111.00	2.00	108	114	
Group 5	8	129.50	2.45	126	133	
Group 6	8	124.50	2.45	121	128	
Group 7	8	120.50	2.45	117	124	
Group 8	8	116.50	2.45	113	120	

Finally, Group 3 exhibited the lowest mean impact strength among all groups, differing significantly from every other group. The most substantial difference was recorded when compared with Group 4 (-2.90 KJ/m², 95% CI: -3.29 to -2. as the lowest-impact strength group in the analysis.51, p<0.001), confirming Group 3

In conclusion, the impact strength ranking followed a clear pattern:

Group 4 > Group 7 > Group 5 > Group 2 > Group 8 > Group 6 > Group 1 > Group 3

Null Hypothesis [H_0] – There is no difference in the mean Flexural Strength between 8 groups.

Alternative Hypothesis [H_A] – There is a difference in the mean Flexural Strength between 8 groups.

Finally, Group 1 exhibited the lowest mean flexural strength, differing significantly from all other groups. The most substantial difference was recorded when compared to Group 3 (-48.00 MPa, 95% CI: -51.67 to -44.33, p<0.001), confirming Group 1 as having the lowest flexural strength in the analysis.

In conclusion, the ranking of flexural strength follows a clear trend: Group 3 > Group 5 > Group 6 > Group 7 > Group 8 > Group 4 > Group 2 > Group 1.

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Discussion

The concept of modern nanotechnology was introduced by Feynman's famous talk entitled ''There's Plenty of Room at the Bottom'' refering to the extensive use of nanomaterials. Much progress in nanotechnology has been witnessed, and the area is continuously expanding into various fields.[9]

Nanodiamond surfaces can be homogenized with a single type of functional group according to the application requirements. The use of nanodiamond particles as a reinforcing material in polymer composites has attracted great attention for improving the performance of polymer composite materials. The superior mechanical properties and rich surface chemistry of nanodiamonds have made them a superior material for tuning and reinforcing polymer composites.[9]

The agglomeration of particles at the nanoscale level is an inherent issue that substantially damages performance in relevant fields. Most nanomaterials start to agglomerate when they encounter each other. This property of nano materials highly affect the mechanical properties of polymethylmethacrylate to which these nanomaterials are added. To overcome this inherent characteristic of nanoparticles and to ensure good dispersion of the reinforcement particles in any media they need proper surface modification or functionalization. [9,10]

Nanocellulose has a large amount of reactive hydroxyl group on its surface make it feasible to modify the surface properties by various chemical modifications such as esterification, etherification, silylation, tempo mediated oxidation, polymer grafting, click reaction, amidation, and sulfonation, etc.. The resulted nanocellulose after modification shows enhancement in properties for better compatibility between nanocellulose and hydrophobic host polymer. Physical treatment can also have employed on plant fibre to change the structural and surface properties without any chemical treatment and thereby affect the mechanical bonding with the matrix. [11]

The purpose of this study was to evaluate the effect of incorporating nanodiamond, nanocellulose and hybrid of nanodiamond-nanocellulose to conventional PMMA on both flexural and impact strengths of the resultant nanocomposite material. Based on the results of this study, the addition of NDs to conventional PMMA improved the flexural strength. While the impact strength results were different, the addition of NDs decreased the impact strength of PMMA nanocomposites. The addition of nanocellulose to the conventional PMMA. Among the hybrid formulations, the combination of with 0.5wt% nanodiamond and 1wt% nanocellulose fibres showed slightly lower impact strength but remained competitive and second highest flexural strength was recorded. The polymer reinforced 0.25wt% nanodiamond and 1wt% nanocellulose fibres displayed a good level of impact resistance and flexural strength. In contrast, the polymer containing only 0.5wt% nanodiamond exhibited the weakest impact strength, reinforcing the idea that nanodiamond reinforcement alone does not improve mechanical resilience as effectively as fibre-based formulations. The conventional polymer demonstrated slightly higher impact resistance than the nanodiamond-only variant but remained the least effective among fibre-reinforced groups.

Conclusion

Reinforcement of conventional PMMA with a combination of nanocellulose fibres and nanodiamond particles significantly enhances both impact and flexural strength, making them an effective modification. The highest impact strength was observed in the polymer reinforced with 1wt% nanocellulose fibre, closely

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followed by hybrid formulations incorporating nanodiamond and nanocellulose fibres in various concentrations. The conventional polymer exhibited the lowest impact strength, highlighting the importance of reinforcement for improved mechanical property.

Flexural strength results similarly indicated that nanodiamond reinforcement played a crucial role in enhancing structural durability. The polymer containing 0.5wt% nanodiamond demonstrated the highest flexural strength however the impact strength considerably reduced while hybrid formulations incorporating both nanodiamond and nanocellulose fibres maintained superior mechanical properties. The conventional polymer had the lowest flexural strength, reinforcing the need for structural modifications.

Overall, hybrid formulations of nanodiamond and nanocellulose fibres exhibited optimal performance across both mechanical parameters. The conventional polymer consistently ranked the lowest, emphasizing the necessity of material enhancements. These results demonstrate that tailored modifications significantly improve polymer strength, with hybrid reinforcements providing the best balance between impact resistance and flexural durability. The study validates the effectiveness of nanoparticle-based reinforcements in advancing polymer materials for enhanced mechanical reliability.

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