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Investigations on mechanical properties of selected restorative dental materials upon fiber reinforcement

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Abstract

The purpose of this in vitro experimental study was to evaluate the mechanical properties such as flexural strength, fracture toughness and abrasive resistance of restorative acrylic resins namely, PMMA (Travelon) and Bis-Acryl resin (Protemp™4) reinforced with glass, polyethylene and carbon fibers. A total of 240 rectangular specimens (30 each from 8 groups) having dimension (65 x 10 x 3.3 mm^3) were fabricated and tested for flexural strength, fracture toughness and abrasive resistance. The unreinforced group served as the control. The data pertaining to flexural strengths, fracture toughness and abrasive resistance were compared by one way ANOVA test, followed by Post Hoc Tests (Bonferroni Multiple Comparisons) analysis, using a significance level of 0.05. The flexural strength of specimens of PMMA and Bis-Acryl resin (Protemp™4) reinforced with carbon, polyethylene and glass fibers were significantly higher than their respective control groups. Incorporation of carbon fiber reflected significantly higher flexural strength, followed by polyethylene fiber and glass fiber. The mean value of fracture resistance was significantly increased in specimens of reinforced with carbon fiber. However, the mean values on abrasive resistance in specimens of PMMA (Travelon) and Bis-Acryl resin (Protemp™4) reinforced with carbon fiber were significantly lowered when compared to the respective control group.

Key words: Polymethyl methacrylate (PMMA), carbon fiber, glass fiber, polyethylene fiber, flexural strength, fracture toughness, abrasive resistance.

Introduction

Synthetic resins have been widely used in medical and dental applications such as intraocular lens, bone cements in orthopedics, filler in bone cavities and skull defects, cavity filling, maxillofacial reconstructive materials and prosthetic appliances etc.[1] Amongst these, polymethyl methacrylate (PMMA) is regarded as the most suitable biomaterial due to its favorable characteristics such as biocompatibility, acceptable aesthetics, chemical inertness, dimensional stability and ease of use and repair.[2] However, the inadequate mechanical characteristics of PMMA, e.g. poor thermal conductivity, low flexural and fatigue strength, low impact strength has resulted lowered clinical life of the prostheses coupled with increased patient's dental visits and the cost factor.[3-5]

Dentures are known to undergo various failures such as fractures and de-bonding of the teeth, and other types of failures in complete or partial dentures.[6] It was observed that 29% of all repairs to dentures were related to the midline fractures of complete dentures.[7] Therefore, it is logical to understand the reasons to which the fracture occurs and to explore the methods to prevent such failures. Different polymerization and reinforcement techniques have been examined to enhance the mechanical characteristics of denture base acrylic resins. Flexural strength is an important mechanical characteristic that signifies the long-term prognosis of interim restorations. Therefore, various types of fibers such as carbon, aramid, woven polyethylene, and glass fibers have been reinforced with the polymer materials to improve their mechanical characteristics.[8-10]

However, the studies are lacking regarding the effect of silanation of the various types of fibres impregnation on mechanical properties of commercially available synthetic acrylic resins. Therefore, the present in-vitro study was designed to evaluate the flexural strength, fracture toughness and abrasive resistance of two commercially available acrylic resins namely, i) PMMA (Travelon), ii) Bis-Acryl resin (Protemp^{TM}4) utilizing reinforcement with glass fibre, polyethylene fibre and carbon fibre.

Materials & Methods

Chemicals: The acrylic resins namely PMMA (Travelon) and Bis-acryl resin (Protemp^{TM}4) were procured from M/s Densply India Private Limited, India and M/s 3M ESPE Deutschland, GmbH, Germany, respectively. Silano (Silane coupling agent) was obtained from M/s Angelus, Londrina, PR, Brazil.

Sample size: A total of 240 specimens (30 from each 8 groups) were fabricated from PMMA (Travelon) and Bis-Acryl resin (Protemp^{TM}4) as defined in Table 1.

Preparation of specimens for flexural strength and fracture toughness: The required quantity of acrylic resin powder & liquid of PMMA (Travelon) was taken in the ratio 2:1 by weight. These were mixed in a porcelain mixing jar with a lid. When the mixture reached the dough stage, it was packed in the molds. As per ISO standards, Master die was fabricated in Aluminum die with dimensions (65 x 10 x 3.3 mm^3). The two parts of the metal die were finally closed ensuring metal-to-metal contact. The Bis-Acryl resin (Protemp^{TM}4) which was supplied in auto-mixing cartridge, was loaded onto the dispensing gun and through the nozzle, the material was dispensed into the aluminum die. The specimens were removed from the die and finishing was done to remove the flash and any roughness present. Thus, the specimen (n=30) each of PMMA (Travelon) and Bis-Acryl resin

(Protemp^{TM}4) were prepared and designated used as control group

Incorporation of fibers: The fibers of glass, polyethylene and carbon were wrapped in aluminum foil and cut into size (Length = 50 mm and Width = 8 mm) with the help of a sharp BP blade. To ensure uniform weight of the fibers, the control group specimens were weighed in a digital weighing machine and 5% of the total weight of specimen was calculated as 0.125 gm. Each type of fiber was weighed to be equal to 0.125 gm so that all specimens have the same weight of fibers. The required quantity of acrylic resin powder & liquid of PMMA (Travelon) were taken in the ratio 2:1 by weight, mixed in a porcelain mixing jar with a lid, the fibers were wet with part of this mixture of polymer and monomer for PMMA and bonding agent (Silano) for Bis-Acryl resin (Protemp^{TM}4). These were preserved in tinfoil till the mixture reached the dough stage, for packing into the molds and a space was created for the fibers by pressing it with a glass slab and the fibers were placed lengthwise in the molds approximately in the middle of the thickness of acrylic dough. Then the remaining dough was packed over the fibers and closure was done. The flash was removed and the metal die was kept under constant pressure (pressure to be noted). Thus, the reinforced samples (n=30) each of PMMA (Travelon) and Bis-Acryl resin (Protemp^{TM}4) were prepared.

Preparation of specimens for abrasive resistance: To have all the specimens of similar dimension, a round aluminum die was prepared having the dimension (Diameter = 100 mm; Thickness=3.3 mm). The metal die had two detachable parts which could be assembled together. The specimens of control group and reinforced samples, each of PMMA (Travelon) and Bis-Acryl resin (Protemp^{TM}4) were prepared as mentioned above.

All the specimens were tested for flexural strength, fracture toughness and abrasive resistance at M/s CiMEC Infralabs Private Limited (Formerly Apex Geotechnical Services; A Unit of CiMEC Group NABL Accredited Lab at Ahmedabad), Mohan Nagar, Ghaziabad – 201 003 (UP), India. These specimens were tested in the standard laboratory atmosphere.

Testing of specimen for flexural strength: Specimen (n=10) of each group were tested for flexural strength by Lloyd's Universal Testing Machine LR 100K, at a crosshead speed of 5 mm/min. These specimens were placed at the centre until fracture occurred. The maximum load required to fracture the specimens in each group was recorded. The load (F), deflection data was recorded to calculate the flexural strength (**σ**) in unit (MPa or N/mm^2) by using the equation:

$\boldsymbol{\sigma = (3FL)/2bh^2}$,

where **F** is the maximum load applied in Newton,

L is the supporting width in mm,

b is the width of specimen in mm, and **h** is the thickness of test specimen in mm.

Testing of specimen for fracture toughness: Specimen (n=10) of each group were tested in tension for fracture toughness by Lloyd's Universal Testing Machine LR 100K with the direction of force perpendicular to the plane of pre formed crack. A pre crack was placed in the test specimen by placing a sharp scalpel at the end of slot and by applying hand pressure. Each specimen was held in a specialized tension device in the machine and tension force was applied at a crosshead speed of 5 mm/min. The peak force (F) in Newton which caused fracture in specimen, was recorded and used to calculate the fracture toughness ($\mathbf{K_{1C}}$) in unit (MPa.m$^{1/2}$) by using the equation: $\mathbf{K_{1C} = pc / bw^{1/2}}$. $\mathbf{F(a/w)}$, where **pc** is the maximum load before crack advance, **b** is the average specimen thickness (cm), **w** is the width of the specimen (cm), **F** is the peak force in Newton and **a** is crack length (cm).

Testing of specimen for abrasive resistance: Specimen (n=10) of each group were tested in tension for abrasive resistance by Taber Abraser Tester Model 5130. The round flat specimens were mounted to a turntable platform that rotated on a vertical axis at affixed speed and the depth of the wear (mm) was calculated.

Statistical analysis: The data were tabulated and analyzed by IBM SPSS version 23, for one-way analysis of Variance (ANOVA) and Post Hoc tests - Bonferroni multiple comparisons to determine whether there was any significant statistical difference between each control and reinforced experimental groups. As the intent of this study was to make comparisons between the different materials tested, independent samples t-test was used for analysis.

Results & Discussion

The data pertaining to flexural strength of control group of PMMA (Travelon) and Bis-acryl resin (Protemp™4) and experimental groups reinforced with 5% each of glass fiber, polyethylene fiber and carbon fiber were mentioned in Table 2. There was significant increase in flexural strength of specimens of PMMA (Travelon) reinforced with glass (A1), polyethylene (A2) and carbon fibers (A3) when compared to control group (A) [Table 2(B)]. On comparative evaluation, the maximum mean value (80.99 N/mm^2) of flexural strength was observed in specimens reinforced with carbon fiber followed by polyethylene fiber (76.52 N/mm^2) and glass fiber (73.80 N/mm^2). Intergroup comparison on flexural strength indicated significant rise in flexural strength in specimens of group A3, in comparison to group A1 [Table 2(B)]. Data given in Table 2 indicate that the flexural strength of specimens of Bis-acryl resin (Protemp™4) reinforced with glass (B1), polyethylene (B2) and carbon fiber (B3) were significantly higher than specimens of control group (B). It was maximum (91.82 N/mm^2) for specimens reinforced with carbon (B3), followed by polyethylene fiber (84.24 N/mm^2) and glass fiber (79.34 N/mm^2). The flexural strength of specimens of group B3 were significantly higher than the specimens of group B1 and B2 [Table 2(D].

The data related to fracture toughness of control group of PMMA (Travelon) and Bis-acryl resin (Protemp™4) and experimental groups reinforced with 5% each of glass fiber, polyethylene fiber and carbon fiber were mentioned in Table 3. The mean value of fracture toughness in specimens of PMMA (Travelon) reinforced with carbon fiber (A3) was significantly higher than its control group (A) and experimental group reinforced with glass fiber (A1). The mean value of fracture resistance was highest in group A3 followed by polyethylene and glass fiber [Table 3(B)]. The data on mean value of fracture toughness in specimens of Bis-acryl resin (Protemp™4) reinforced with carbon fiber (B3) was significantly increased than the control group (B) [Table 3(D)].

Table 4 defines the data on abrasive resistance in control group of PMMA (Travelon) and Bis-acryl resin (Protemp™4) and experimental groups reinforced with 5% each of glass fiber, polyethylene fiber and carbon fiber. Mean values of abrasive resistance in experimental groups of PMMA (Travelon) reinforced with glass (A1) and carbon fiber (A3) was significantly lower when compared to the control group (A) [Table 4B)].

The abrasive resistance in experimental groups of Bis-acryl resin (Protemp™4) reinforced with glass, polyethylene and carbon fibers (B1, B2 and B3) were significantly decreased when compared with the control group (B). The decrease in abrasive resistance was maximum in experimental group reinforced with carbon fiber followed by glass and polyethylene fiber [Table 4(D)].

Provisional restorations, or interim restorations, have been widely used in dentistry today as it provides a protective coverage to the teeth during fabrication of permanent fabrication. The fabrication of an ideal provisional restoration is important for a successful outcome and a happy patient life.[11] However, the function of an ideal provisional restoration must satisfy many requirements for an optimal interim restoration such as biological and esthetics considerations coupled with the mechanical characteristics. Biologically, a provisional fixed restoration must provide pulpal protection by preventing the conduction of temperatures through the outer surface of the enamel into inter-pulpal tissues.[12]

Consequent upon consideration of mechanical characteristics, the fracturing of the restoration is due to the excessive occlusal forces, parafunctional habits, clenching/grinding and bruxing which leads to failure of the provisional restoration and thus demands extra visits of patients and requisite more expenses.

The type of material used for fabricating a provisional restoration also plays an important role in the strength and the resistance to fracture. PMMA resin has stood the test of time due to its durability and inexpensive cost. Currently, the newer provisional materials made from bis-acryl composite (BAC) resin submit improved physical properties over PMMA resins, including the ease of handling due to auto mixing cartridges, less polymerization shrinkage and a decrease in heat released during curing. However, the problem we usually encounter with composite provisional material is that it's brittle. Some evidences have shown that the more complex the case, especially in multi tooth replacement situations with the requirement of long-term durability, the PMMA resin has been the material of choice, but there is paucity of data on this aspect.

Studies indicated that experiments pertaining to reinforcement with different types of fibers as a method of improving the fracture strength of provisional restorations.[13-15] Fiber materials are classified by the fiber type and orientation. The most commonly used fibers in today's dental application are glass, polyethylene and carbon fibers. These materials increased the mechanical properties of provisional restorative resins which may be explained due to the transfer of stress from weak polymer matrix to the fibers that have high tensile strength. The stronger the adhesion between the fiber and the matrix, greater the strengthening effect. Experimental evidences indicate that the position, quantity, direction and degree of adhesion between the fibers and polymer matrix affect the degree of reinforcement.[16-18]

Flexural strength is defined as materials ability to resist deformation under load. It is measured as the highest stress expressed within the material at its moment of rupture. Strength is given as a general mechanical term, but what we are really measuring are stress within the resin. The findings of the present study are in accordance with the studies by other investigators.[19,20] Fracture toughness is a quantitative way of expressing a material's resistance to crack propagation, the standard values for a given material are generally available and may more accurately determine the likelihood of fracture of a provisional restoration in practice. It was inferred that reinforcement of specimens especially with carbon fiber resulted an increase in fracture toughness as compared to unfilled PMMA (Travelon) and Bis-acryl resins. From the clinical standpoint, improvements in the flexural properties to meet the requirements of clinical use would be desirable because they reflect the stiffness of the denture base resin and resistance a force that may develop in oral environment. Nevertheless, the procedures described in this study reflect promise of potentially practical techniques for strengthening provisional fixed partial dentures.

Conclusion

Within the restrictions of this study, the following conclusions were achieved:

1. PMMA (Travelon) and Bis-acryl resin (Protemp^{TM}4) reinforced with carbon, polyethylene and glass fiber had significantly higher values of flexural strength. This elevation was more marked in specimens reinforced with carbon, followed by polyethylene and glass fiber.
2. Fracture toughness was significantly higher in specimens of PMMA (Travelon) and Bis-acryl resin (Protemp^{TM}4) reinforced with carbon fiber.
3. Specimens of PMMA (Travelon) reinforced with carbon and glass fiber had significantly lower value of abrasive resistance. However, reinforcement of specimens of Bis-acryl resin (Protemp^{TM}4) with carbon, polyethylene and glass fiber resulted significantly decreased value of abrasive resistance.
4. From the result of this study, it can be concluded that reinforcement of PMMA (Travelon) and Bis-acryl resin (Protemp^{TM}4) resins by non metallic fibers like glass, polyethylene, carbon fibers results in significant increase in flexural strength, fracture toughness and decrease in abrasive resistance of provisional restorative resin materials.

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Legend Tables

Table 1: Data related to various groups and respective sample size.

S. No.	Synthetic Resin	Group	Number of Specimen
1	PMMA (Travelon)	Control group (A)	30
		Reinforced with glass fiber (A1)	30
		Reinforced with polyethylene fiber (A2)	30
		Reinforced with carbon fiber (A3)	30
2	Bis-Acryl resin (Protemp™4)	Control group (B)	30
		Reinforced with glass fiber (B1)	30
		Reinforced with polyethylene fiber (B2)	30
		Reinforced with carbon fiber (B3)	30

Table 2: Data on flexural strength (N/mm^2) of PMMA (Travelon) and Bis-acryl resin (Protemp^{TM}4) in control group and experimental groups reinforced with 5% each of glass fiber, polyethylene fiber and carbon fiber.

Resin	Control Group	Reinforced with glass fiber	Reinforced with polyethylene fiber	Reinforced with carbon fiber
PMMA (Travelon)	A	A1	A2	A3
	67.86 ± 1.026 (n=10)	73.80 ± 1.475 (n=10)	76.52 ± 2.662 (n=10)	80.99 ± 1.642 (n=10)
Bis-acryl resin (Protemp^{TM}4)	B	B1	B2	B3
	67.81 ± 1.787 (n=10)	79.34 ± 2.054 (n=10)	84.24 ± 1.471 (n=10)	91.82 ± 1.850 (n=10)

Data represented as Mean ± SE; n = Number of samples

Table 2(A): One-way ANOVA on flexural strength for PMMA (Travelon)

	Sum of Squares	df	Mean Square	F	Significance
Between Groups	903.534	3	301.178	9.253	.000
Within Groups	1171.710	36	32.547		
Total	2075.244	39			

Table 2(B): Post Hoc Tests – Games-Howell Comparisons on flexural strength for PMMA (Travelon)

Group	Sub-group	Mean Difference	Standard Error	Significance
A	A1	-5.93800*	1.79787	.021
	A2	-8.62700*	2.85351	.047
	A3	-13.13200*	1.93739	.000
A1	A	5.93800*	1.79787	.021
	A2	-2.68900	3.04393	.813
	A3	-7.19400*	2.20828	.021
A2	A	8.62700*	2.85351	.047
	A1	2.68900	3.04393	.813
	A3	-4.50500	3.12836	.495
A3	A	13.13200*	1.93739	.000
	A1	7.19400*	2.20828	.021
	A2	4.50500	3.12836	.495
*Mean difference is significant at 0.05 level.				

Table 2(C): One-way ANOVA on flexural strength for Bis-acryl resin (Protemp™4)

	Sum of Squares	df	Mean Square	F	Significance
Between Groups	2997.576	3	999.192	30.737	.000
Within Groups	1170.266	36	32.507		
Total	4167.841	39			

Table 2(D): Post Hoc Tests – Bonferroni Multiple Comparisons on flexural strength for Bis-acryl resin (Protemp™4)

Group	Subgroup	Mean Difference	Standard Error	Significance
B	B1	-11.52900*	2.54980	.000
	B2	-16.42800*	2.54980	.000
	B3	-23.81000*	2.54980	.000
B1	B	11.52900*	2.54980	.000
	B2	-4.89900	2.54980	.376
	B3	-12.28100*	2.54980	.000
B2	B	16.42800*	2.54980	.000
	B1	4.89900	2.54980	.376
	B3	-7.38200*	2.54980	.038
B3	B	23.81000*	2.54980	.000
	B1	12.28100*	2.54980	.000
	B2	7.38200*	2.54980	.038
*Mean difference is significant at 0.05 level.				

Table 3: Data on fracture toughness ($MPa.m^{1/2}$) of PMMA (Travelon) and Bis-acryl resin (Protemp™4) in Control group and experimental groups reinforced with 5% each of glass fiber, polyethylene fiber and carbon fiber.

Resin	Control Group	Reinforced with glass fiber	Reinforced with polyethylene fiber	Reinforced with carbon fiber
PMMA (Travelon)	A	A1	A2	A3
	1.32 ± 0.088 (n=10)	1.33 ± 0.113(n=10)	1.69 ± 0.111 (n=10)	1.82 ± 0.081 (n=10)
Bis-acryl resin (Protemp™4)	B	B1	B2	B3
	1.23 ± 0.073 (n=10)	1.53 ± 0.093 (n=10)	1.59 ± 0.078 (n=10)	1.77 ± 0.134 (n=10)

Data represented as Mean ± SE; n = Number of samples

Table 3(A): One-way ANOVA on fracture toughness for PMMA (Travelon)

	Sum of Squares	df	Mean Square	F	Significance
Between Groups	1.933	3	.644	6.507	.001
Within Groups	3.565	36	.099		
Total	5.499	39			

Table 3(B): Post Hoc Tests – Bonferroni Multiple Comparisons on fracture toughness for PMMA (Travelon)

Group	Subgroup	Mean Difference	Standard Error	Significance
A	A1	-.00800	.14074	1.000
	A2	-.36700	.14074	.079
	A3	-.50000*	.14074	.007
A1	A	.00800	.14074	1.000
	A2	-.35900	.14074	.091
	A3	-.49200*	.14074	.008
A2	A	.36700	.14074	.079
	A1	.35900	.14074	.091
	A3	-.13300	.14074	1.000
A3	A	.50000*	.14074	.007
	A1	.49200*	.14074	.008
	A2	.13300	.14074	1.000
*Mean difference is significant at 0.05 level.				

Table 3(C): One-way ANOVA on fracture toughness for Bis-acryl resin (Protemp^{TM}4)

	Sum of Squares	df	Mean Square	F	Significance
Between Groups	1.516	3	.505	5.262	.004
Within Groups	3.459	36	.096		
Total	4.975	39			

Table 3(D): Post Hoc Tests – Bonferroni Multiple Comparisons on fracture toughness for Bis-acryl resin (Protemp^{TM}4)

Group	Subgroup	Mean Difference	Standard Error	Significance
B	B1	-.29400	.13862	.245
	B2	-.36700	.13862	.072
	B3	-.53900*	.13862	.003
B1	B	.29400	.13862	.245
	B2	-.07300	.13862	1.000
	B3	-.24500	.13862	.514
B2	B	.36700	.13862	.072

	B1	.07300	.13862	1.000
	B3	-.17200	.13862	1.000
B3	B	.53900*	.13862	.003
	B1	.24500	.13862	.514
	B2	.17200	.13862	1.000
*Mean difference is significant at 0.05 level.				

Table 4: Data on abrasive resistance (mm) of PMMA (Travelon) and Bis-acryl resin (Protemp™4) in control group and experimental groups reinforced with 5% each of glass fiber, polyethylene fiber and carbon fiber.

Resin	Control Group	Reinforced with glass fiber	Reinforced with polyethylene fiber	Reinforced with carbon fiber
PMMA (Travelon)	A	A1	A2	A3
	1.10 ± 0.0155(n=10)	0.98 ± 0.015 (n=10)	1.05 ± 0.016 (n=10)	0.92 ± 0.016 (n=10)
Bis-acryl resin (Protemp™4)	B	B1	B2	B3
	1.54 ± 0.022 (n=10)	1.20 ± 0.017 (n=10)	1.46 ± 0.009 (n=10)	1.12 ± 0.012(n=10)

Data represented as Mean ± SE; n = Number of samples

Table 4(A): One-way ANOVA on abrasive resistance for PMMA (Travelon)

	Sum of Squares	df	Mean Square	F	Significance
Between Groups	.197	3	.066	25.963	.000
Within Groups	.091	36	.003		
Total	.287	39			

Table 4(B): Post Hoc Tests – Bonferroni Multiple Comparisons on abrasive resistance for PMMA (Travelon)

Group	Subgroup	Mean Difference	Standard Error	Significance
A	A1	.12300*	.02247	.000
	A2	.05200	.02247	.159
	A3	.18500*	.02247	.000
A1	A	-.12300*	.02247	.000
	A2	-.07100*	.02247	.019
	A3	.06200	.02247	.054
A2	A	-.05200	.02247	.159
	A1	.07100*	.02247	.019
	A3	.13300*	.02247	.000
A3	A	-.18500*	.02247	.000
	A1	-.06200	.02247	.054
	A2	-.13300*	.02247	.000
*Mean difference is significant at 0.05 level.				

Table 4(C): One-way ANOVA on abrasive resistance for Bis-acryl resin (Protemp^{TM}4)

	Sum of Squares	df	Mean Square	F	Significance
Between Groups	1.211	3	.404	152.203	.000
Within Groups	.095	36	.003		
Total	1.306	39			

Table 4(D): Post Hoc Tests – Bonferroni Multiple Comparisons on abrasive resistance for Bis-acryl resin (Protemp^{TM}4)

Group	Subgroup	Mean Difference	Standard Error	Significance
B	B1	.33700*	.02303	.000
	B2	.07900*	.02303	.009
	B3	.41900*	.02303	.000
B1	B	-.33700*	.02303	.000
	B2	-.25800*	.02303	.000
	B3	.08200*	.02303	.006
B2	B	-.07900*	.02303	.009
	B1	.25800*	.02303	.000
	B3	.34000*	.02303	.000
B3	B	-.41900*	.02303	.000
	B1	-.08200*	.02303	.006
	B2	-.34000*	.02303	.000
*Mean difference is significant at 0.05 level.				