



Effect of Thermocycling on the Flexural Strength of Various PMMA Resins Reinforced with Different Fibres: An *In vitro* Study

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Authors' contributions

This work was carried out in collaboration between all authors. Author KN designed the study, wrote the protocol and wrote the first draft of the manuscript. Author LK managed the literature searches. Authors KN, MA and SC managed the experimental process. Authors AKV and LK managed the manuscript editing and review. All authors read and approved the final manuscript.

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ABSTRACT

Introduction: Fracture of an acrylic resin complete denture base after aging, poses problem for patients, dentists and dental laboratory technicians. This study was performed to determine the effect of thermocycling on the flexural strength of a commercially available, heat-polymerized acrylic denture base material reinforced with glass fibers, carbon fibers, aramid fibers and high (HI) impact resins.

Materials and Methods: Forty specimens were made of similar dimension from five groups of materials. Each group had eight specimens. A commercially available heat polymerized P.M.M.A

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denture base resin was selected as control and PMMA reinforced with glass, carbon, aramid fibres and HI impact resin were compared. All specimens were then subjected to thermocycling before testing. Flexural strength was evaluated with universal testing machine. The results were analyzed with Analysis of Variance (ANOVA) followed by Turkey HSD test.

Results: All reinforced specimens and HI impact resins showed better flexural strength than the conventional acrylic resin after thermocycling. Specimens reinforced with glass fibers showed the highest flexural strength, followed by HI impact resin, carbon fibers and aramid fibers. After artificial aging, a significant improvement in the flexural strength of conventional acrylic resin was observed when it was reinforced with glass fibers.

Conclusion: Within the limitations of this study, incorporation of 2% by weight of glass fibre in PMMA resin exhibited statistically significant maximum flexural strength followed by high impact resin, carbon, polyaramid reinforced P.M.M.A denture base resin. These can readily be used in the fabrication of dentures subjected to heavy masticatory loads.

Keywords: High impact resin; thermocycling; reinforcement; PMMA resin; glass fibers; flexural strength.

1. INTRODUCTON

Removable prosthodontics is that branch of prosthodontics that deals with the replacement of lost teeth and contiguous soft tissue structures for edentulous or partially edentulous patients by artificial substitutes that are removable from the mouth by the patient [1]. The prosthesis should be functionally effective and pleasing in appearance. The material used in the fabrication of the prosthesis should be biologically compatible, readily available, reasonably inexpensive, durable with good strength and simple to manipulate with a readily controlled technical procedure.

Various materials have been used in dentistry such as wood, bone, ivory and metal alloys from time to time. The methyl-methacrylate resin was introduced to the National Society of Denture Prosthesis at Atlantic city in July 1937 by Dr. Walter H. Wright [2]. It is presently the most widely used denture base material but, it has many inherent disadvantages as well. Fractures may occur in use, because of its unsatisfactory transverse strength, impact strength or fatigue resistance.

To overcome the shortcomings like fracture due to bone resorption and accidental fall, various approaches to strengthen the acrylic resin have been suggested. These include:

- Chemical modification by incorporation of butadiene styrene to produce graft copolymer high-impact resins.
- Mechanical reinforcement through the inclusion of: Carbon, glass, aramid and ultra high modulus poly ethylene fibres.

- Metals in the form of powder, whiskers and meshes.

Fibers, are easier to incorporate in the resin and exhibit good bonding if treated with a coupling agent. Effective fibers reinforcement is dependent on many variables, including the type of fibers; percentage in the matrix; modulus and distribution of fibers; length, orientation and form (chopped, continuous, unidirectional or bidirectional). Fibers can vary in diameter, length and form [3].

The length of the fiber is of importance because the end of the fiber does not carry loads and as the fiber length increases the ineffective portion of the fiber has smaller effect.

Many studies have evaluated the use of individual reinforcing fibers to improve the strength of the denture base but without thermocycling. Very few studies have compared the flexural strength of fibre reinforced specimens under wet thermocycling i.e. artificial ageing.

Dootz et al. in [4] had shown that material ageing can dramatically affect the physical and mechanical properties. During mastication, the oral cavity gets in contact with food at different temperatures and changes occur in the properties of the denture base materials. The present study was planned to measure and compare the flexural strengths of PMMA specimens reinforced with monomer treated glass fibres, nylon fibers and carbon fibres in random distribution with High – Impact resin and conventional P.M.M.A specimens after wet thermocycling.

2. MATERIALS AND METHODS

Forty specimens were made from five groups of materials with each group of eight specimens (Table 1). A commercially available heat polymerized P.M.M.A denture base resin (DPI, Bombay Burma Trading Cooperation Ltd, India) was selected as control and PMMA reinforced with glass (Ahlstrom Corp, Karhula, Finland), nylon (MRF Ltd, Chennai, India), polyaramid fibers (Kevlar; DuPont, Wilmington, Del) and HI impact resins (Pyrax Acryl HI, Pyrax polymars, India) were compared. During processing of specimens, the manufacturer's instructions were followed. For standardization of specimens, the brass metallic dies and specially fabricated flask was used.

The samples were prepared using brass metal dies (Fig. 1) of length, width and height (65 mm x 10.30 mm x 2.58 mm). This is in compliance to the International Standards Organization (ISO Standard, 1999:1567) [4] for fabricating specimens for testing flexural strength of denture base polymers. Customised Brass metal dies were invested in custom made flask of size 17 cm x 9.5 cm x 4.5 cm in die stone after lubricating them with a thin layer of petroleum

jelly. When the stone was set, the two halves of the flask were opened (Fig. 2).The metal dies were removed gently, thus making moulds for fabrication of test specimens.

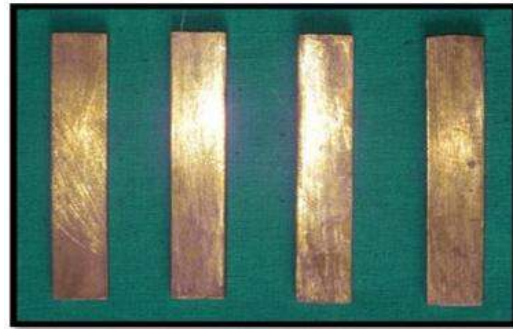


Fig. 1. Brass metal dies

For control group, the material used was conventional heat polymerized P.M.M.A denture base material. Separating media, cold mold seal (DPI) was applied on the dental stone mould with the help of a no.6 camel hair brush and was allowed to dry. A mixture of polymer and monomer in the ratio of (3:1 by volume) [5] was measured prior to mixing. When the mixture

Table 1. Five groups of materials

S. no.	Group	Treatments	No. of specimens
1.	I	PMMA without fibers(Control group)	08
2.	II	PMMA with glass fibers	08
3.	III	PMMA with Carbon fibers	08
4.	IV	PMMA with Polyaramid fibers	08
5.	V	PMMA High-Impact	08

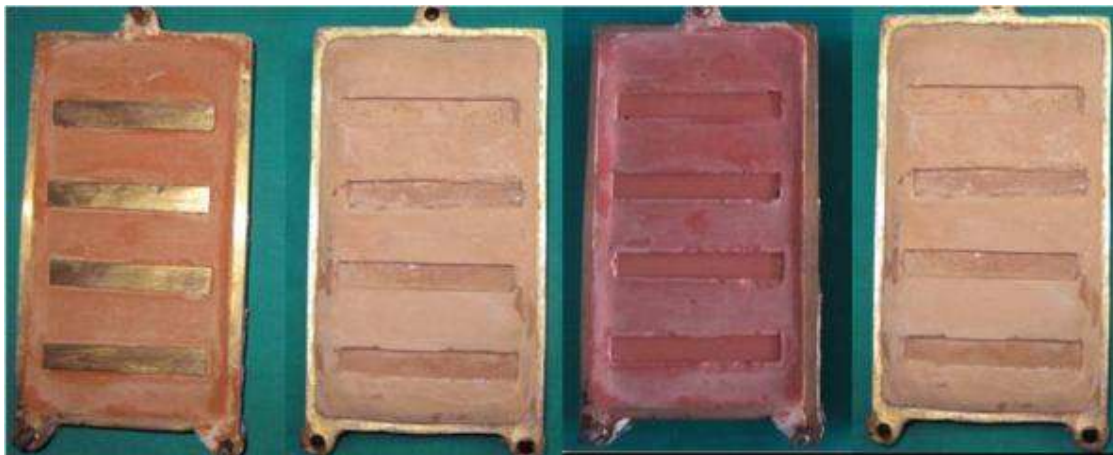


Fig. 2. Customised brass metal dies with two halves

reached the dough stage, it was kneaded and packed into the mould. Flask was closed and trial closure was carried out twice using a hydraulic press under 140 bar of pressure. Excess of the material was trimmed using a B.P blade No. 21. Finally, the flask was clamped and final closure was done and pressure was slowly raised to 140 00000 pascal of pressure for even flow of material. It was kept for bench curing to allow proper penetration of monomer into the polymer for 30 minutes.

Then, the flask was immersed in water in an acrylizer at room temperature and processing was done at 74°C, for eight hours, with no terminal boiling treatment(i.e long curing cycle) as suggested by Anusavice [6]. After the completion of the curing cycle, overnight bench cooling was done and the specimens were carefully removed from the moulds and the excess was trimmed and finished (Fig. 3).

For preparation of fibers reinforced PMMA test specimens, the amount of fibers incorporated into PMMA resin was 2% by weight in each group in random distribution [4]. The fibers were weighed using electronic balance. The three experimental groups consisted of PMMA resin specimens of the same dimensions reinforced with glass, polyaramid and nylon fibers. These fibers were cut to 6 mm length and had a thickness of 10 – 15 microns (Fig. 4). The cut fibers were soaked in monomer for 10 minutes for better bonding with the acrylic resin. After the fibers were removed from the monomer excess liquid was allowed to dry. The resin and fibers were mixed thoroughly to disperse the fibers. On reaching dough stage the mixture was kneaded

and packed into the prepared mold. The specimens were polymerized and recovered in the same manner as the control group. After deflasking, if the specimens revealed the exposed fibers at the peripheral borders trimming was performed with tungsten carbide burs to avoid delamination of the specimens.

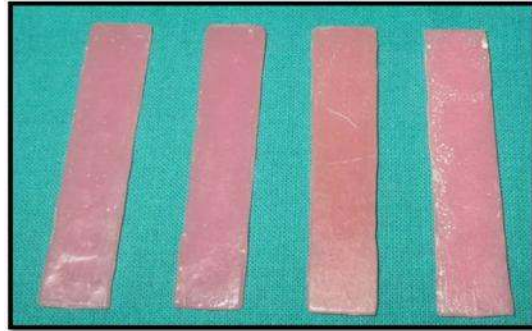


Fig. 3. Specimens incorporated with fibres

Specimens were labeled on each end before testing for easy identification (Fig. 5).

All specimens were then subjected to thermocycling before testing. This step was carried out in a thermocycling machine. It contains a specialized tray in which specimens were placed in the plastic tray to expose all the surfaces of each specimen and also for transferring the specimens to different water-baths maintained at different temperatures. The specimens of each group were thermocycled between the maximum and minimum temperatures that the mouth is subjected to: 65±1°C and 4±1°C respectively, and the mouth temperature, 37±1°C.



Fig. 4. Different types of fibers



Fig. 5. Group distribution of specimen (labeled)



Fig. 6. Universal testing machine with specimen

The frequency of the thermocycling was chosen 300 cycles which represent approximately one month of service for a P.M.M.A resins. This is based on the assumption that oral cavity may subjected to maximum of 10 times extreme hot and cold during eating of food per day. All specimens were tested for flexural strength with a three point bending test with Universal Testing Machine at a crosshead speed of 1.1mm/min (Fig. 6). A load was applied by centrally located rod until fracture occurred. The flexural strength was calculated with the following formula: $FS=3/2 \times PL/BD^2$.

All the datas were analysed by Analysis of Variance (ANOVA) followed by Tukey HSD test ($p<0.001$).

3. RESULTS

Table 1 shows distribution of samples in different groups according to flexural strength. In Group I (Commercially available PMMA resin), flexure strength values ranged from 632.13 to 660.27 kgf/cm² with a mean value of 645.97±10.07 kgf/cm². In Group II (reinforced with glass fibers), flexure strength values ranged from 735.72 to 766.92 kgf/cm² with a mean value of 754.06±9.79 kgf/cm². In Group III (reinforced with carbon fibers), flexure strength values ranged from 674.99 to 707.33 kgf/cm² with a mean value of 692.64±11.14 kgf/cm². In Group IV (reinforced with aramid fibers), flexure strength values ranged from 650.86 to 681.28 kgf/cm² with a mean value of 667.64±9.67 kgf/cm² and a median value of 669.13 kgf/cm². In Group V (HI impact resin), flexure strength values ranged from 704.15 to 736.01 kgf/cm² with a mean value of 720.67±10.61 kgf/cm². Group II (reinforced with glass fibers) specimens had the highest flexural strength, followed by groups V, III, IV and I (Table 2). The amount of force required to fracture the specimens is called fracture resistance.

Analysis of variance and box plot (Fig. 7) thereafter show a statistically significant intergroup difference in mean flexure strength of different groups. On the basis of above evaluation, the following order of flexure strength was observed in different groups:

Group I < Group IV < Group III < Group V < Group II

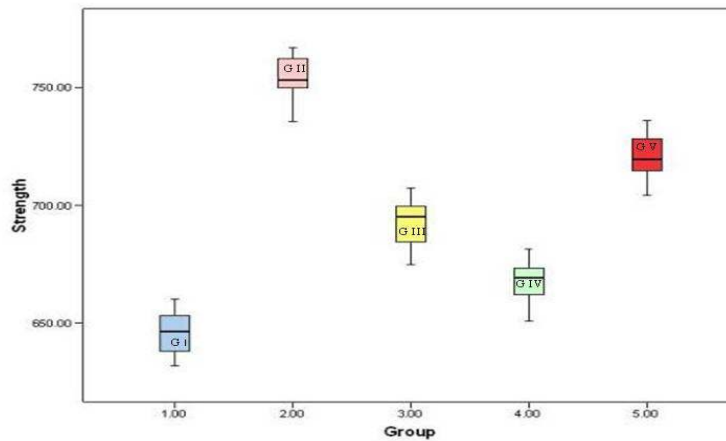


Fig. 7. Analysis of variance and box plot

Table 2. Distribution of samples in different groups according to flexural strength

S no.	Group I flexure strength (kgf/cm ²)	Group II flexure strength (kgf/cm ²)	Group III flexure strength (kgf/cm ²)	Group IV flexure strength (kgf/cm ²)	Group V flexure strength (kgf/cm ²)
1.	641.85	762.58	693.86	668.98	721.98
2.	645.15	749.91	696.53	671.47	719.97
3.	634.43	735.72	674.99	656.97	712.63
4.	632.13	766.92	677.87	650.86	716.78
5.	660.27	761.55	701.43	669.28	719.19
6.	647.98	754.25	697.89	681.28	704.15
7.	658.49	749.72	707.33	674.95	736.01
8.	647.43	751.85	691.18	667.33	734.64
Min	632.13	735.72	674.99	650.86	704.15
Max	660.27	766.92	707.33	681.28	736.01
Mean	645.97	754.06	692.64	667.64	720.67
SD	10.07	9.79	11.14	9.67	10.61
Median	646.29	753.05	695.195	669.13	719.58
Normality test	KS=0.171; p=0.200	KS=0.204; p=0.200	KS=0.198; p=0.200	KS=0.237; p=0.200	KS=0.201; p=0.200

4. DISCUSSION

Polymethyl methacrylate resin is the material of choice for denture base fabrication. Introduced in 1937 by Dr. Walter Wright [2], P.M.M.A resin continues to be used because of its favorable working characteristics, processing ease, accurate fit, its stability in the oral environment, superior esthetics and need of inexpensive equipments [4]. Despite these excellent properties, there is a need for improvement in fracture resistance of P.M.M.A resin. This is so because the P.M.M.A resin is subjected to various stresses during function; these include compressive, tensile and shear stresses and temperature changes of oral cavity. Most

fractures of the denture occur inside the mouth during function, primarily because of resin fatigue [7].

The study was conducted to compare the flexural strength of heat polymerized PMMA denture base resin when reinforced with E – glass fibres, polyaramid fibers and carbon fibers after thermocycling which is equivalent to simulating intra oral conditions in terms of temperature variations (65+/- 2°C, 37 +/- 2°C and 4 +/- 2°C) and presence of water. Brass metal dies and special brass flask of dimensions was used for polymerization of the specimens to ensure the ease of flasking the rectangular specimens in a rectangularly shaped flask and also to invest

more number of dies at one time. Glass fibers, Polyaramid fibers, Carbon fibers were used for reinforcement of PMMA resin. The specimens of control group and high – impact resin group were not reinforced with fibers.

The temperature during wet thermocycling was 65+- 2 degree C, 37 +- 2 degree C & 4 +- 2 degree C to simulate the oral conditions so that the specimens can be subjected to conditions caused due to temperature variations and water sorption [8,9].

The study showed that glass fibers exhibited maximum flexural strength of 766.92 kgf/cm². Glass is an inorganic substance that is cooled to a rigid condition without crystallization. Different types of glass fibres are produced commercially; these include E-glass, S-glass, R-glass, V-glass, and Cemfil. Of these, E-glass fibre, which has high alumina and low alkali and borosilicate, is claimed to be superior in flexural strength [7]. The modulus of elasticity of glass fibres is very high, and thus, they absorb more stresses without deformation. Moreover, glass fibres are esthetically pleasant and therefore, can be used to reinforce any part of the denture [10,11].

The values of flexural strength obtained in this study were less (735.72 to 766.92 kgf/cm²) than as compared to a study done by Jacob J in [4] which reported a strength value of 825.4 to 1084.5 kgf/cm² when glass fibres were added 2% by weight which was done at room temperature only without any temperature variations as the specimens were not subjected to thermocycling. The non fiber reinforced high – impact resin group exhibited flexural strength of 736.01 kgf/cm².

Although Manley in [12] reported that carbon reinforced P.M.M.A resin specimens exhibited better flexural strength than rubber modified P.M.M.A resin specimens. The greater flexural strength of High impact resin arises from the incorporation of a rubber phase into the beads during their suspension polymerization. High-impact resin polymers are similar to heat-accelerated methyl methacrylate materials but are reinforced with butadiene-styrene rubber. The rubber particles are grafted to methyl methacrylate to bond to the acrylic matrix [2,11].

Carbon fibres which are chemically polyamide fibres are based primarily on aliphatic chains. The chief advantage of carbon lies in its resistance to shock and repeated stressing.

Carbon fibers have dark color which is undesirable in a prosthesis as also discussed by Renu Tandon et al. in [2]. Carbon-reinforced specimens demonstrated higher fracture resistance than the unreinforced control group which was 692.64 kgf/cm² but lower than the glass reinforced specimens (754.06 kgf/cm²). Aramid is a generic term for wholly aromatic fibers. These fibers are resistant to chemicals, are thermally stable, and have a high mechanical stability, melting point, and glass transitional temperature. They also have pleated structure (molecules are radially arranged in the form of sheets) that makes aramid weak as far as flexural, compression, and abrasion behavior are concerned [4]. Aramid fibres are unaesthetic because after reinforcement the resultant prosthesis is yellowish in appearance which is difficult to mask and therefore its use is limited to certain intraoral applications.

Future research in this field of study includes comparison of flexural strength of the fibers reinforced specimens after fabrication through different polymerization cycles and also after thermocycling. Because the modulus of elasticity of the glass fibers is very high, most of the stresses could not produce deformation [13-15]. Thus, in this study, glass fibers reinforced resin specimen exhibited the best flexural strength than the other specimen groups. Glass fibers, high impact resin specimens appear to be suitable for long term use and are also esthetically pleasing [16].

5. CONCLUSION

Incorporation of 2% by weight of glass fiber in PMMA resin exhibited statistically significant maximum flexural strength followed by high impact resin, carbon, polyaramid reinforced P.M.M.A denture base resin. High - Impact denture base resin exhibited better flexural strength than other groups; with 10.2% increase in flexural strength as compared to 14.% of glass fiber reinforced P.M.M.A denture base resin. Glass fibers reinforced resins can readily be used in the fabrication of dentures subjected to heavy masticatory loads e.g. single complete dentures opposing natural dentition, distal extension partial denture bases, maxillofacial prosthesis, periodontal splints etc. which are considered to be prone to fracture [4].

CONSENT

It is not applicable.

ETHICAL APPROVAL

All authors hereby declare that all experiments have been examined and approved by the appropriate ethics committee and have therefore been performed in accordance with the ethical standards laid down in the 1964.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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