
Evaluation of The Effect of Thermocycling on the Fracture Toughness and Impact Strength of Bio High Performance Polymer and Conventional Denture Base Resin Material : An In Vitro Study

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Abstract: Aim: To carry out a comparative evaluation of the effect of thermocycling on the fracture toughness and impact strength of Bio High Performance Polymer and conventional heat cure resin.

Materials and methods: The study was carried out in four groups based on the manufacturing process of the sample: Group I (n = 8) control group containing specimens of BioHPP and PMMA before thermocycling; Group II (n = 8) test group of BioHPP and PMMA after thermocycling; The values for fracture toughness of each specimen were measured using a universal testing machine by a three-point bending test. Impact strength testing of the samples was done on the Izod impact testing machine. The mean values of the fracture toughness and impact strength were calculated by Mann-Whitney U test and Wilcoxon signed rank test are used for analysing the data.

Results: The statistical analysis shows that there was no significant difference in the fracture toughness and impact strength of BioHPP and PMMA before and after thermocycling. BioHPP has better mechanical properties than PMMA.

Conclusion: There is no significant effect on the mechanical properties of BioHPP and PMMA before and after thermocycling. Thus it concludes that both materials has clinical success and better longevity.

Keywords: Thermocycling, Fracture toughness, Impact strength, BioHPP, PMMA

1. Introduction

Over the last few decades, new dental materials have been introduced in dentistry with better longevity and efficiency. Long term success of dental materials depends on the durability of the material in the oral environment. Oral environment varies from person to person and the factors like oral inhabitants, occlusal forces, parafunctional habits, humidity, dietary factors and temperature fluctuations all contribute to uncontrollable factors that can affect longevity of the restoration.

Despite great efforts made in research, it is still not possible to say that there is an excellent material that can meet all the demands of physician. Therefore, studies are still ongoing in respect of the most suitable material. Several materials have been introduced over the last century for dentures such as cellulose product, vinyl resin and vulcanite. The poor performance of these materials has led PMMA as replacement. 1

The material used in the fabrication should be biologically compatible, readily available, reasonably inexpensive, durable with good strength. 2 The PMMA was introduced by the National Society of Denture Prosthesis at Atlantic city in July 1937 by Dr. Walter.H. Wright. It is presently the most widely used denture base material but it has many inherent disadvantages as well. Fractures may occur during function, because of its unsatisfactory transverse strength, impact strength or fatigue resistance. 2 80% of fractures are caused by impact and involves very high repair cost. 3

PMMA is a thermoplastic material. However, its application is constrained at elevated temperature due to its relatively poor thermal stability. Denture base resin should have enough strength and toughness to stand up to forces generated during function while also being dimensionally stable for years under varying thermal conditions.

In 1992, PEEK was introduced in the field of dentistry. PEEK material usage has increased tremendously in prosthetic dentistry. It combines excellent mechanical properties with the high level of biological compatibility. PEEK is used as a removable partial denture framework, implant material, metal and ceramic free crown and bridges. Exceptional solvent resistance, low modulus of elasticity and biocompatibility with bone make this polymer a good candidate to replace the use of metal in the body. It has increasingly been employed in dentistry as a biomaterial that can be used for medical purposes.

A modified PEEK material containing 20% ceramic fillers is a high performance polymer (Bio HPP), which presents high biocompatibility, good mechanical properties, high temperature resistance and chemical resistance. Bio HPP is a part of the PEEK family with excellent stability, crack resistance, optimal polishing properties, and low plaque affinity. Bio HPP has a lot of advantages like preparation of restoration with the lowest specific weight, elasticity similar to that of bone, bone absorbing effect, metal-free restoration, low material fatigue, no viscoelastic features, high biocompatibility, low plaque accumulation, and no corrosion.

Thermal stresses can be pathogenic in two ways. Firstly, mechanical stresses induced by different temperature changes can directly induce crack propagation through bonded interfaces. Secondly, gap volume can change which pumps pathogenic oral fluids in and out of gaps. This cylindrical flow has been incorrectly termed 'percolation'. The number of chewing cycles is predicted to be 20-50 cycles per day that means corresponding to 1000 cycles per year. According to Gale and Darwell, artificial saliva is used to simulate oral environment as it is important in extrapolating the results according to clinical conditions. Even though thermocycling does not exactly simulate oral environment, it can be used to evaluate the behaviour of dental materials under thermal stresses.

There is limited literature regarding the BioHPP and its mechanical properties. Hence, the purpose of this study was to evaluate its mechanical properties of BioHPP and PMMA before and after thermocycling. The primary research question is if there is any difference between the fracture toughness and impact strength of BioHPP and PMMA. The null hypothesis for this research is that there is no difference in the fracture toughness and impact strength of the test samples.

2. Materials and Methods:

2.1 Fabrication of Specimen

The study was divided into two groups based on the manufacturing process of the dentures:

- Group I (n=8)—control group; containing specimens of PMMA fabricated by conventional compression molding technique and BioHPP fabricated from milled disc.
- Group II (n= 8)—test group ; containing specimens of PMMA fabricated by conventional compression molding technique and BioHPP fabricated from milled disc.

A metallic rectangular strip of 65 mm × 10 mm × 3 mm for fracture toughness and 80mm ×4mm×10mm dimensions for impact strength was milled and fabricated for the preparation of stone mold in a dental flask.

PMMA samples were prepared using conventional heat curing PMMA denture base resin [polymer and monomer—powder and liquid (DPI, Mumbai, India)] according to the manufacturer's instructions (4.4 mL of monomer, 10 g of powder). When the mixture reached the dough state (usually 5–10 minutes, depending on the room temperature), the dough was collected and packed well into the rectangular prepared mold by a routine procedure including the trial packing. Bench curing was done for 30 minutes. The PMMA heat cure resin was processed by a short curing cycle according to the manufacturer's recommendation. After bench cooling for 30 minutes, the flasks were immersed in the cold water until they were cooled to room temperature and deflasking was done.

CAD/CAM BioHPP discs of 98 mm diameter and 25 mm height were scanned in the DC5 milling system (Dental Concept Systems GmbH, Ulm, Germany). A layout of the strip of 65 mm × 10 mm × 3 mm dimensions for fracture toughness sample and 80mm ×4mm×10mm dimensions for impact strength sample was designed on the CAD file. The 5-axis milling system was used to mill these blocks.

Samples with frank defects and not fulfilling dimensional specifications were discarded and new samples were fabricated. Finishing of all the specimens was done with tungsten carbide bur and polished using the conventional laboratory polishing method: coarse pumice, water, lathe bristle brush, and soft leather polishing wheel. The finished and polished specimens were stored for wet conditions in distilled water at room temperature for 50 hours. Each specimen was measured with a digital Vernier caliper and the excess was trimmed. All specimens after finishing were stored at room temperature. A total of 16 specimens were prepared, which were further subdivided into two subgroups of 8 specimens each for testing for fracture toughness and impact strength.

Testing of Specimen

The values for fracture toughness of each specimen was measured in a three-point bending mode using a Universal testing machine (UNITEST-10, ACME Engineers, India) at a crosshead speed of 5 mm/ minute. The span of the two supports was 50 mm. The fracture toughness was calculated by a program in the universal testing machine. The fracture toughness was calculated using the following formula

$$K_{Ic} = \frac{fP_{max}}{B W^{3/2}}$$

where P_{max} is the maximum load, B is the specimen thickness, W is the specimen width, f is a geometrical factor depending on the ratio a/W .

Impact strength testing of the samples was done on the Izod impact testing machine with a pendulum of S2 scale in air at $24 \pm 2^\circ\text{C}$. Before testing, the pendulum was released to freely swing in the air to record the air resistance (AR) encountered by the free swinging pendulum. Air resistance of 0.7 Joules was recorded. The readings were taken on the S2 scale where the pointer was stabilized after swing. The specimen was clamped in position precisely. The pendulum was released and reading indicating energy absorbed (EA) to break the specimens on the S2 scale was recorded. All the specimens were tested in the same manner.

Impact strength of the specimen was calculated by using the formula:

$$IS = \frac{\text{ENERGY ABSORBED} \times 1000}{\text{EFFECTIVE WIDTH} \times \text{THICKNESS}}$$

IS is impact strength (KJ/m²)

Thermocycling of the test samples:

In the present in vitro study thermal cycling was done to simulate the intraoral conditions. All samples subjected to thermal cycling for 5000 cycles respectively in distilled water bath between 5 degree Celsius and 55-degree Celsius with a 30 sec dwell time in a thermocycler apparatus (Fig 1). Upon completion of thermocycling the samples were stored in distilled water in their respective container at room temperature, until they were subjected to fracture toughness and impact strength testing.

2.2 Statistical Analysis

In this study there is comparison of fracture toughness and impact strength of BioHPP and PMMA before and after thermocycling. Mean and standard deviation of the control and test groups are calculated using Mann-Whitney U test and Wilcoxon signed rank test.

3. Results:

The fracture toughness of control group of BioHPP were found to be 1756 and 1752 MPa. The fracture toughness of test group of BioHPP were found to be 1749 and 1750 MPa. The fracture toughness of control group of PMMA were found to be 97 and 108 MPa. The fracture toughness of test group of PMMA were found to be 94 and 90 MPa. The fracture toughness value were not significantly lower after thermocycling. (Graph 1 and Graph 3), (Table 1,3,5,7)

The impact strength of control group of BioHPP were found to be 24 and 21 KJ/m². The impact strength of test group of BioHPP were found to be 22 and 20 KJ/m². The impact strength of control group of PMMA were found to be 4.12 and 3.8 KJ/m². The impact strength of test group of PMMA were found to be 3.18 and 3.51 KJ/m². The impact strength value were not significantly lower after thermocycling.

Regarding fracture toughness test of both BioHPP and PMMA before and after thermocycling, statistical analysis by Wilcoxon signed rank test shows that there is no significant difference in ($p=0.374$ for BioHPP and $p=0.09$ for PMMA) fracture toughness between control and test groups of both materials.(Graph 2 and Graph 4),(Table 2,4,6,8)

Regarding impact strength test of both BioHPP and PMMA before and after thermocycling, statistical analysis by Wilcoxon signed rank test shows that there is no significant difference ($p= 1$ for BioHPP and $p=0.205$ for PMMA) of impact strength between control and test groups of both materials(Table 3).

4. Discussion

PMMA were introduced to dentistry in 1937 following which various materials have also been introduced in material science, none of them closely mimics the oral soft tissue as it does.¹⁰ Since its introduction, it is routinely and successfully being used for the fabrication of full and partial prosthesis owing to its outstanding properties.¹¹ But this material cannot be considered as ideal because of its poor mechanical and physical properties. From the patients perceptible denture fractures and denture shrinkage are two commonly faced problems. Several studies have investigated the incidence and types of fracture of dentures. Hargreaves et al (1969) reported that 63% of dentures had broken within 3 years of this provision.

Fractures in dentures results from two different types of forces namely flexural fatigue and impact. Flexural fatigue occurs after repeated flexing of the material whereby the structure eventually fails after being repeatedly subjected to loads. Microscopic cracks are developed in the areas of the stress concentration and with continued load these cracks fuse to a growing fissure which ultimately weakens the material. The midline fractures which occur more often in maxillary dentures are results of flexural fatigue. Another cause of fractures which is seen commonly is the impact fracture which usually occur outside the mouth as a result of sudden blow to the denture or accidental dropping while cleaning or coughing. ¹²

BioHPP has shown to be a boon to the field of prosthodontics as a result of its excellent biocompatibility as well as mechanical properties. It is thus proving to be a suitable material for a variety of applications in the field of prosthodontics may it be fixed or removable prostheses.¹³ BioHPP (High Performance Polymer) is a PEEK variant that has been specially optimised for the dental field. Thanks to strengthening with a special ceramic filler, optimised mechanical properties have been created for dental technical and/or dental medical use in the crown and bridge area. This ceramic filler has a grain size of 0.3 to 0.5 μm . Due to this very small grain size, constant homogeneity can be produced. This homogeneity is an important prerequisite for these outstanding material properties and forms the basis for consistent quality.

Fracture toughness is a quantitative way of expressing a materials resistance to brittle fracture when a crack is present. Fracture toughness can be measured by the critical stress intensity factor (KIC). The KIC gives valuable insights into the risk of crack propagation. There is a requirement for denture base materials to endure high masticatory loads or impacts.¹⁴As such, manufacturers are consistently seeking methods of developing stronger and more durable denture base materials.

Impact Strength is the capability of the material to withstand a suddenly applied load and is expressed in j/m or kg cm/cm of notch. The impact strength of material determines the toughness of material it means resistance to high speed loading. The factors affecting the impact strength are rate of loading, temperature, orientation, processing condition and types, degree of crystallinity, molecular weight.

The fracture toughness and impact strength value of the samples were not significantly lower after thermocycling.

The limitations of the current study were the in vitro nature of the study and the samples prepared do not replicate the shape of an actual denture.

5. Conclusion

Within the limitations of the study, it can be concluded that the CAD/CAM denture bases have the highest fracture toughness and impact strength as compared to all the test groups. Moreover, the BioHPP improves the fracture toughness and impact strength of denture bases compared to the conventional PMMA.

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Fig 1: Samples during Thermocycling

Table 1: Measurement of fracture toughness of control group (Group 1A) – BioHPP

SL.no:	Sample No:	L(mm)	W(mm)	B(mm)	Fracture Toughness (MPa)
1	1A(1)	65	10.30	2.5	1756
2	1A(2)	65	10.30	2.5	1752

Table 2: Measurement of impact strength of control group (Group 1A) – BioHPP

SL.no:	Sample	L(mm)	W(mm)	B(mm)	Impact Strength (KJ/m ²)
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	No:				
1	1A(1)	80	4	10	24
2	1A(2)	80	4	10	21

Table 3: Measurement of fracture toughness of control group (Group 1B) – PMMA

SL.no:	Sample No:	L(mm)	W(mm)	B(mm)	Fracture Toughness (MPa)
1	1B(1)	65	10.30	2.5	97
2	1B(2)	65	10.30	2.5	108

Table 4: Measurement of impact strength of control group (Group 1B) - PMMA

SL.no:	Sample No:	L(mm)	W(mm)	B(mm)	Impact Strength (KJ/m ²)
1	1B(1)	80	4	10	4.12
2	1B(2)	80	4	10	3.82

Table 5: Measurement of fracture toughness of test group (Group 2A) - BioHPP

SL.no:	Sample No:	L(mm)	W(mm)	B(mm)	Fracture Toughness (MPa)
1	2A(1)	65	10.30	2.5	1749
2	2A(2)	65	10.30	2.5	1750

Table 6: Measurement of impact strength of test group (Group 2A) - BioHPP

SL.no:	Sample No:	L(mm)	W(mm)	B(mm)	Impact Strength (KJ/m ²)
1	2A(1)	80	4	10	22
2	2A(2)	80	4	10	20

Table 7: Measurement of fracture toughness of test group (Group 2B) - PMMA

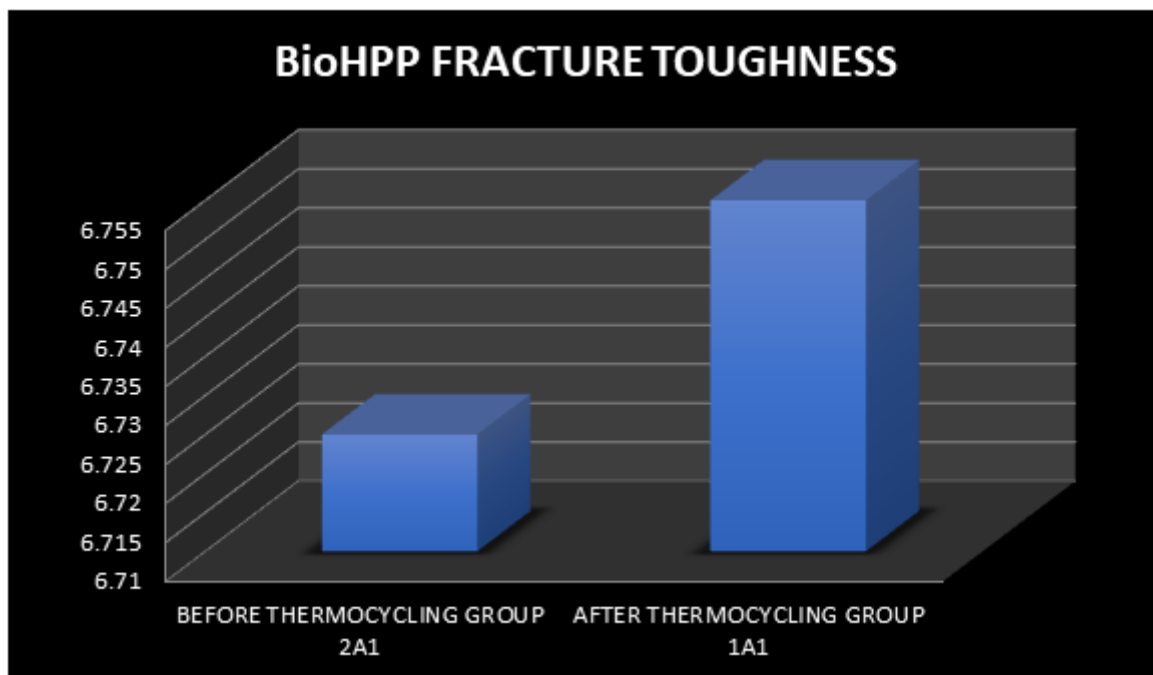
SL.no:	Sample No:	L(mm)	W(mm)	B(mm)	Fracture Toughness (MPa)
1	2B(1)	65	10.30	2.5	94
2	2B(2)	65	10.30	2.5	90

Table 8: Measurement of impact strength of test group (Group 2B) - PMMA

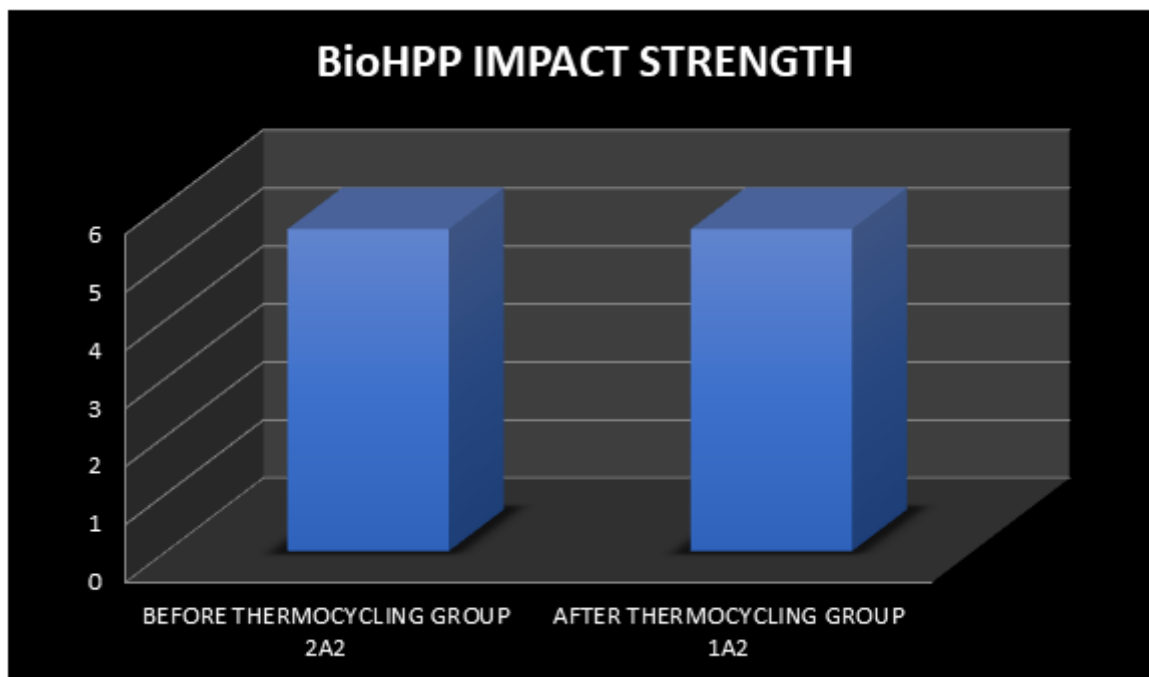
SL.no:	Sample No:	L(mm)	W(mm)	B(mm)	Impact Strength (KJ/m ²)
1	2B(1)	80	4	10	3.18
2	2B(2)	80	4	10	3.51

TABLE 9 :Comparison between before and after thermocycling of fracture toughness and impact strength in BioHPP and PMMA

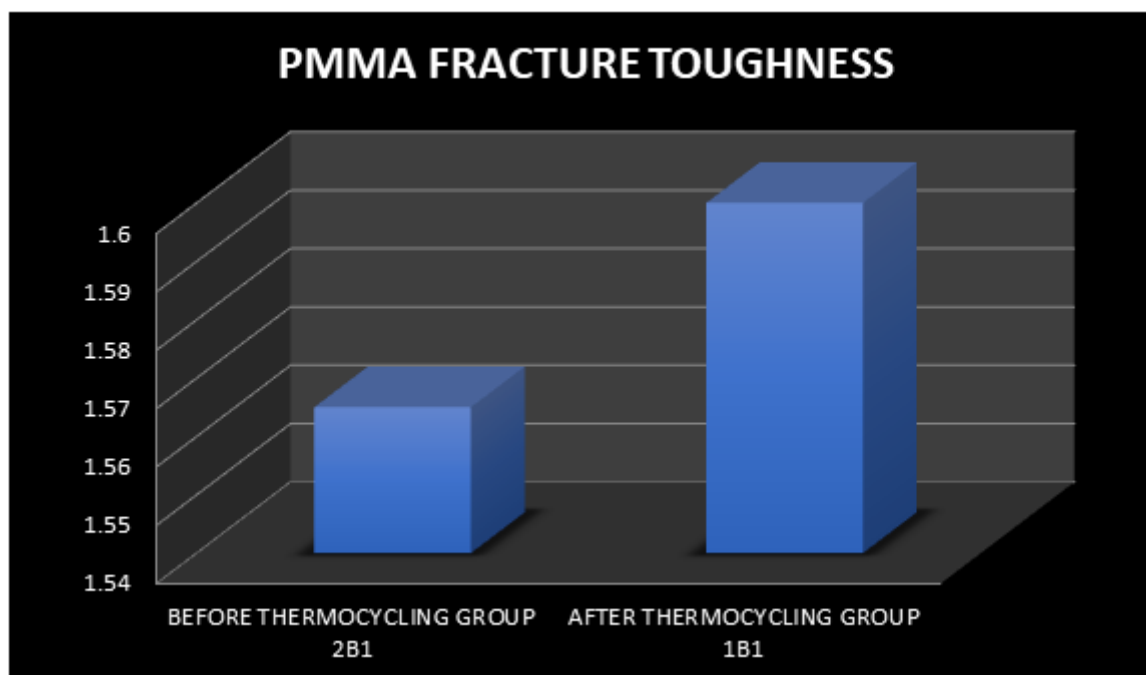
TYPE 1	TYPE 2	TIME PERIOD	GROUP	N	MEAN	STANDARD DEVIATION	P value
BioHPP	FRACTURE TOUGHNESS	BEFORE THERMOCYCLING	GROUP 2A1	2	6.725	0.021	0.374
		AFTER THERMOCYCLING	GROUP 1A1	2	6.755	0.007	
	IMPACT STRENGTH	BEFORE THERMOCYCLING	GROUP 2A2	2	5.55	0.070	1
		AFTER THERMOCYCLING	GROUP 1A2	2	5.55	0.212	
PMMA	FRACTURE TOUGHNESS	BEFORE THERMOCYCLING	GROUP 2B1	2	1.565	0.007	0.09
		AFTER THERMOCYCLING	GROUP 1B1	2	1.600	0.014	
	IMPACT STRENGTH	BEFORE THERMOCYCLING	GROUP 2B2	2	2.20	0.141	0.205
		AFTER THERMOCYCLING	GROUP 1B2	2	2.35	0.070	
TOTAL				16			



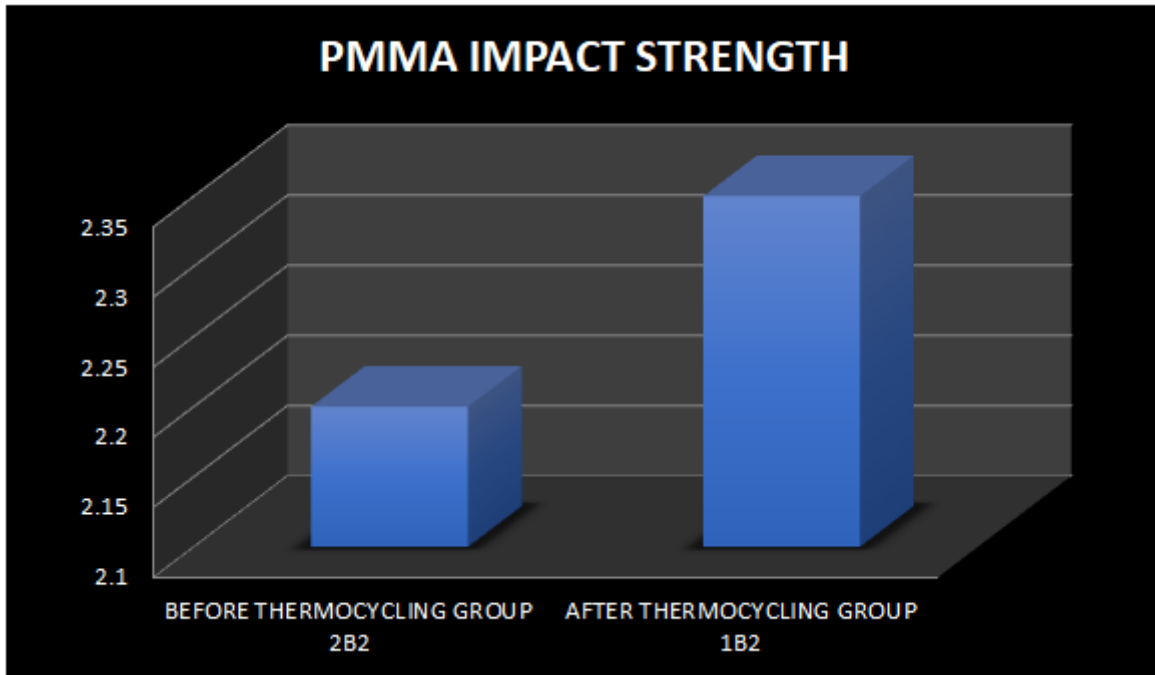
Graph 1:Fracture toughness of BioHPP before and after thermocycling



Graph 2: Impact strength of BioHPP before and after thermocycling



Graph 3: Fracture toughness of PMMA before and after thermocycling



Graph 4: Impact strength of PMMA before and after thermocycling