Vickers hardness and Specific wear resistance of E glass reinforced poly methyl methacrylate

Merin Mathew, Kamalakanth Shenoy, Ravishankar K.S.

Abstract—Poly methyl methacrylate (PMMA) is one of the most widely accepted biomaterial in prosthetic dentistry due to its acceptable advantages. However the conventional PMMA used are far from being ideal because of their inferior mechanical properties. So the present study is to determine the Vickers Hardness and specific wear rate of the silane treated E glass fiber reinforced PMMA. Vickers Hardness number and specific wear rate of PMMA denture base can be determined by varying the weight percentage and aspect ratio of glass fiber. To measure the Vickers hardness, specimens prepared using a standard rectangular mold of 62mm length, 10 mm breadth and 2.5 mm thickness. Vickers Hardness number measured using Vickers hardness test apparatus having square based diamond pyramid as indenter. For wear analysis, specimens prepared using a standard cylindrical mold of 8mm diameter and 25mm length. Specific wear rate measured after measuring the weight loss in the pin on disc method by Wear and Friction Monitor TR-20ICL. Microstructure of the abraded surface observed through Trinocular inverted metallurgical microscope model Metji M1004. Detailed statistical analysis performed using One-way Analysis of Variance (ANOVA), Tukey-Kramer Multiple Comparisons Test. E glass reinforced PMMA shown superior Vickers hardness number compare to control and the specific wear rate for the reinforced groups were less compare to control.

Index Terms— Poly methyl methacrylate. PMMA, specific wear, Silane treated E glass fiber, Vickers hardness.

1 Introduction

Hardness is the mechanical property of a material that enables the material to resist plastic deformation predominantly by penetration, indentation, scratching, abrasion etc.[1] Hardness provides a possible indication of the abrasiveness of the dental material which is the characteristic of the ease of finishing the material as it is resistant to in - service scratching during cleansing or handling.[2] Hardness testing methods have become one of the most popular tools in mechanical testing because of their relatively straightforward, fast and sufficiently repeatable performance.[3] Poly methyl methacrylate is so well received biomaterial by dental profession due to its acceptable advantages. However the mechanical performances of these materials are not ideal.[4] So in order to enhance the mechanical characteristics of the conventional PMMA, surface treated e glass fibers incorporated with the PMMA matrix and a polymer composite is prepared. Hardness testing is one of the effective and relatively simple methods in understanding the mechanical behavior of these reinforced PMMA.

2 Aim

To determine hardness and specific wear resistance of PMMA material by varying the weight percentage of glass fiber. (2.5wt%, 5 wt%, 10 wt %), To determine hardness and specific wear resistance of PMMA material by varying the length/ thickness ratio of glass fiber.(3mm/ 20µm, 6mm/ 20µm, 12mm/ 20µm) and Comparison of the above and understand the optimum property of the PMMA material using the correct weight percentage and aspect ratio.

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3 Materials and Methods

3.1 Materials

Modelling wax, dental stone type III gypsum product, type II gypsum product, silane treated E glass fibers, heat polymerizing PMMA powder and monomer liquid, separating medium.

3.2 Methods

3.2.1. Preparation of gypsum moulds to obtain the acrylic specimen for hardness testing

Wax pattern (62mm X 10mm X 2.5mm) is prepared using modelling wax and invested in the dental flask in the conventional manner using dental stone and model plaster. After 1 hour the invested flask kept for dewaxing, then any waxy residue removed by washing the mould by hot water and then cleaned using soap solution, allowed to dry, thin layer of separating medium is applied in the mould space, allowed to dry. The mould was then ready to be used for the preparation of acrylic specimen.[5]

3.2.2. Preparation of gypsum moulds to obtain the acrylic specimen for wear analysis

Wax pattern (8mm diameter, 25mm length) is prepared using modeling wax and invested in the dental flask in the conventional manner using dental stone and model plaster. After 1 hour the invested flask kept for dewaxing, then any waxy residue removed by washing the mould by hot water and then cleaned using soap solution, allowed to dry, thin layer of separating medium is applied in the mould space, allowed to dry. The mould was then ready to be used for the preparation of acrylic specimen

3.2.3. Preparation of PMMA resin specimen:

3.2.3.1. Control group:

Control group test specimen made with conventional heat polymerized PMMA resin (DPI heat cure) polymer and monomer (2.4gm: 1ml) mixed and allowed to reach dough consistency. Dough is kneaded and then packed into the mould, flask is closed and a pressure of 1400 psi is given and bench cured for 30 minutes in hydraulic press apparatus. Then the flask is clamped and transferred it into the water bath. Temperature of the water bath elevated slowly to 72 °c, and maintained for 90 minutes. Then the temperature of the water bath elevated to 100°c and maintained for 60 minutes. After completion of polymerization cycle, the flask is allowed to cool in same water bath to room temperature, and the acrylic resin specimens are retrieved after deflasking. The specimen obtained were finished and polished in the conventional manner.[5]

3.2.3.2. Reinforced group:

Silane treated E glass fibers of varying length and concentration is taken and impregnated in the measured monomer for 5 minutes, then the polymer powder is weighed and mixed with monomer and E glass fiber and allowed to reach dough consistency. Then it is packed and a pressure of 1400 PSI is given and bench cured for 30 minutes in hydraulic press apparatus. Then the flask is clamped and transferred it into the water bath. Temperature of the water bath elevated slowly to 72 °c, and maintained for 90 minutes. Then the temperature of the water bath elevated to 100°c and maintained for 60 minutes. After completion of polymerization cycle, the flask is allowed to cool in the same water bath to room temperature, and the acrylic resin specimens are retrieved after deflasking. Specimens obtained were finished and polished in the conventional manner.[5]

3.2.4. Hardness testing:

Hardness was measured using Vickers hardness test apparatus. It has a square based diamond pyramid as indenter. The value of the load (50gm) and the time duration (10 seconds) that is to be applied was set. The test specimen was held firmly in position and lens were arranged to get the image clearly at its focal length, then the indentation made using set parameters. Indentations focused and the measuring lines were made to interact at two diagonally opposite corner. Readings were taken by pressing the read button. Similarly the lens was rotated and the measurement of diagonally opposite corner was measured.

3.2.5. Wear analysis:

Specific wear rate was measured using pin on disc method by Wear and Friction Monitor TR-20ICL. Weight of the specimen was measured and considered as initial weight W1.specimen was inserted into the holder and made sure that the end surface of the specimen and disc surface was parallel to each other. Holder was adjusted to get the desirable wear track radius (D=60mm). Load was given on the hang attached to the apparatus (300gm). Specimen securely tightened to the holder. By using the controller attached to the device the speed of rotation (200 rpm) and the time duration for the rotation (10 minutes) were selected. Then the data recorded controller device was switched on. Once the rotation completed after set duration 10 minutes, the weight of the specimen measured W2. The procedure was repeated and the weight measured as W3. Weight loss W1-W2 and W2-W3 measured and average weight loss measured as ΔW. The experiment was repeated for 500 gm, 1000gm load. The load was varied in order to understand the effect of load on the specific wear. Specific wear rate was obtained from the formula:

Specific Wear rate = ΔW / (load in Newton * sliding distance) gm/ Nm

Where ΔW = average weight loss

Sliding distance S = velocity m/sec*time sec

Sliding velocity (V) = (¶ D N)/(60*1000) m/sec

D= wear track diameter selected (60mm)

¶ = 3.14

N = speed of the rotating disc (rpm) (200)

The surface after wear was observed through Trinocular inverted metallurgical microscope model Metji M1004

Figure 1: Abraded surface of the control specimen observed at 50X magnification

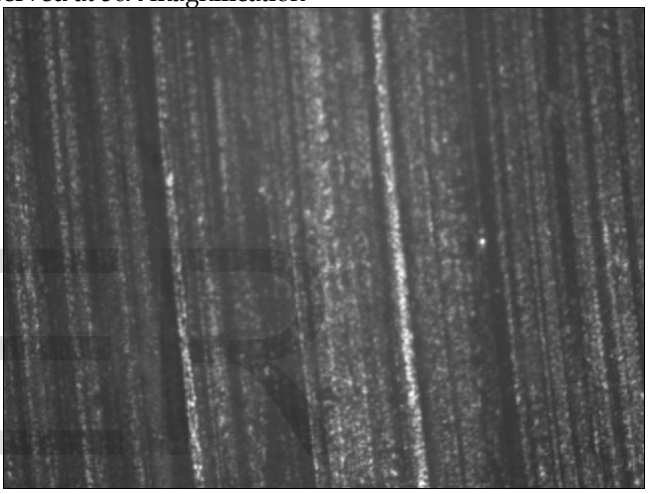
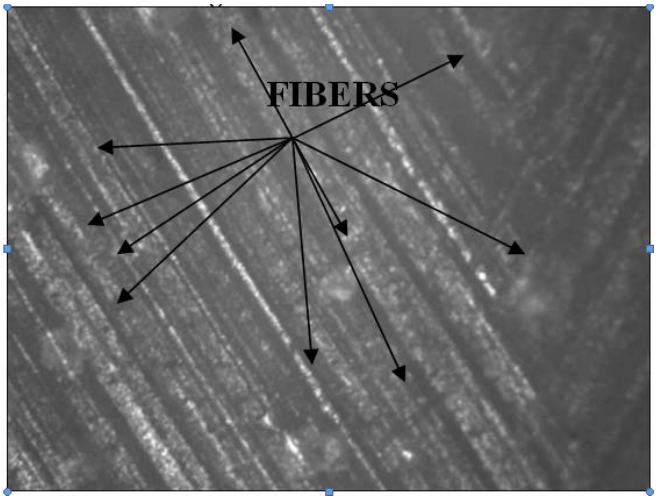


Figure 2: Abraded surface of the reinforced specimen observed at 50X magnification



4 RESULTS

•All modified groups shown significant increase in the Vick-

ers hardness number compared to control group having no fiber.(Table 1)

•Comparing different fiber weight with same fiber length there was significant increase in the Vickers hardness number.(Figure 3)

•Comparing different fiber length with same fiber weight percentage there was no significance in the Vickers Hardness number.(Figure 3)

•Under all given loads, specific wear rate was more for the control group when compare with other reinforced group. (Table 2)

•As the load increases from 300 gm to 1000gm there was significant increase in the wear rate of the control group.(Table 2)

•As the fiber concentration increases, specific wear rate observed was less. (Figure 4)

5 Discussion

Vickers hardness test was developed in 1921by Robert L. Smith and George E. Sandland. The advantage of using this method is that this test can be used for all materials and has the widest scale among the hardness tests and the result will be more accurate. The hardness number is determined by the load over the surface area of the indentation and not the area normal to the force, and is therefore not a pressure. Unit of hardness measured by this test is Vickers Pyramid number (HV) or Diamond Pyramid hardness (DPH) [6] Vickers hardness test is highly depend on the surface homogeneity, the operator's perception and mainly on the elastic recovery of the material. [7] So in order to overcome these problems, the readings were taken on well polished samples immediately after the indentation obtained.

Hardness is the resistance of a material against plastic deformation when the load is applied.[8] The greater the resistance to permanent deformation, the greater will be the hardness. so the hardness measurement of poly methyl methacrylate based prosthesis is important in such a way that those prosthesis having low hardness are tend to be scratched in the oral environment while brushing, chewing, cleaning etc. as a result surface become rough and chance of plaque accumulation, pigmentation and these will lead to compromise the aesthetics, hygiene and moreover the minute cracks or flaws on the surface will act as the initiator for crack propagation and material's fracture. So the hardness property is directly related to the service life of PMMA based materials.[9]

Hardness of the PMMA based materials directly related to the amount of residual monomer present after processing. Unreacted monomer will act as plasticizer and weaken the matrix. Heat cured PMMA can perform better in this aspect that the residual monomer content is less when compare to PMMA polymerized through other mechanisms. There is an increase in the hardness of the heat cured PMMA when glass fibers added to it. [10] So the present study demonstrates the effect of silane treated E glass fiber weight percentage and aspect ratio on the Vickers harness number of PMMA. Inor-

Table 1 : VHN- Vickers hardness number when 50 gm applied for 10 seconds

Sl no.	Control specimen	3mm long E glass fiber			6mm long E glass fiber			12mm long E glass fiber		
		2.5 fiber wt%	5 fiber wt%	10 fiber wt%	2.5 fiber wt%	5 fiber wt%	10 fiber wt%	2.5 fiber wt%	5 fiber wt%	10 fiber wt%
1	20.4	25	27	30.9	26.1	28.1	31	25.4	28.7	33.1
2	19.4	26.4	29	32.7	26.8	27.8	32.5	26	27.2	32.8
3	19.4	24	28.9	31.9	26	27.2	33	25.9	28.1	33.6
4	20.2	26.8	27.4	33	26.6	29	31.2	25.6	27	33
5	20.1	24.7	29.7	31.2	24.2	27.4	31.8	25	28.9	32.9
6	19.9	25.2	28	32.8	25.4	27.6	32.6	25.8	27.8	33.2
Mean	19.9	25.35	28.333	32.083	25.85	27.85	32.016	25.616	27.95	33.1
stdev	0.419	1.0578	1.0385	0.8886	0.946	0.6442	0.811	0.371	0.7714	0.28284

Table 2.Specific wear rate of the tested samples at different loads

	control	2.5 wt 3mm	5 wt 3mm	10 wt 3mm	2.5 wt 6mm	5 wt 6mm	10 wt 6mm	2.5 wt 12mm	5 wt 12mm	10 wt 12mm
Δw load 300 gm	0.001	0.0005	0.00025	0	0.00025	0	0	0.000125	0	0
Specific wear rate (x 10^{-7} g/NM)	9.02	4.51	2.26	0	2.26	0	0	1.12	0	0
Δw load 500gm	0.002	0.001	0.0005	0	0.0005	0	0	0.00025	0	0
Specific wear rate (x 10^{-7} g/NM)	10.83	5.4	2.7	0	2.7	0	0	1.35	0	0
Δw load 1000 gm	0.007	0.004	0.003	0.002	0.003	0.002	0.0005	0.002	0.001	0.0005
Specific wear rate (x 10^{-7} g/NM)	18.93	10.82	8.11	5.41	8.11	5.41	1.35	5.41	2.7	1.35

Key: Δw = average weight loss of specimen after wear test(w1- w2, w2-w3)

ganic materials like glass fibers have poor compatibility with the fiber matrix interface. Silane coupling agents can be used to improve the adhesion of these inorganic glass fibers to the polymeric matrix and in addition coupling agent aid in protecting fiber surface and prevent inhibition of polymerization by the solid surface. [11] The results suggest that there is significant increase in the hardness number when the fiber weight percentage is more and at the same time there is no much variation depends on the aspect ratio of the fiber; that may be due to the fact that hardness is a surface mechanical property and the micro hardness tests demonstrates the ability of the material to resist surface plastic deformation in a limited area.

Figure 3. Vickers hardness number of tested samples

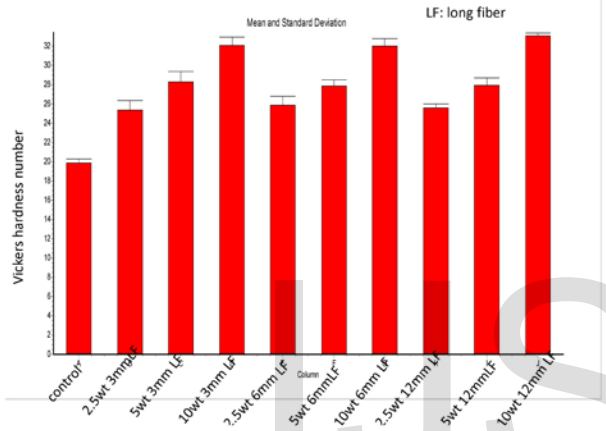
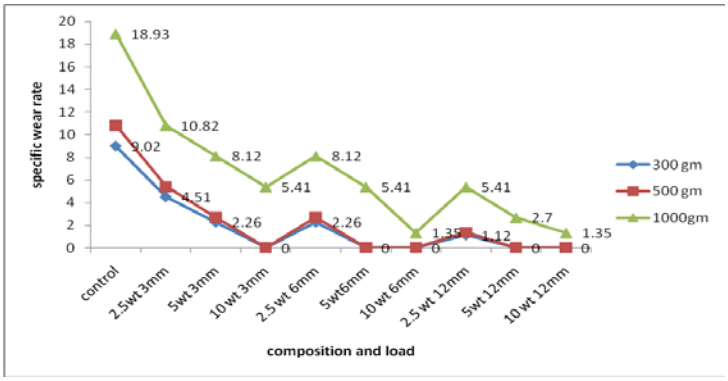


Figure 4. specific wear rate of the tested samples at different loads



Wear rate of the PMMA material is also important in addition with the hardness as it determines the longevity of the materials inside the oral cavity. [12] Wear rate and the hardness are related property, when the wear rate is more hardness is less. [13] Generally two different mechanisms of wear occur in polymers, namely cohesive and interfacial wear processes. The cohesive wear such as abrasion wear, fatigue wear and fretting mainly depend on the mechanical properties of the interacting material. On the other hand the interfacial wear such as transfer wear, chemical wear and corrosive wear depends on the chemistry of the surface involved. The cohesive abrasion wear, the two- body and three- body abrasion wear are the most common wear encountered in polymer composites. This is highly depending on the hardness of the materials in contact, applied load and sliding distance and the geometry of the abrasive particle. [14] In the present study the wear rate of the prepared polymer composites were calculated under different loads and under different applied loads there was significant difference in the wear rate of control specimen with no fiber in it. And there was no much variation in the wear rate of the reinforced groups indicating the fiber reinforced varieties are more resistant to abrasive wear than the control.

6 Conclusion

- Fiber reinforcement results in improved hardness.
- As the concentration of the fiber increases, Vickers hardness number also increases, but aspect ratio did not affect much on the hardness number.
- Specific wear rate observed was less for the reinforced groups compared to control group.
- As the load applied increases, wear rate also increases.

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