Improving the Strength Properties of Denture Base Acrylic Resins Using Hibiscus Sabdariffa Natural Fiber

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Abstract

Various materials have been reported to improve the strength and fatigue resistance of poly methyl methacrylate (PMMA) denture base resins, however there is limited evidence on using natural fibers. This study determined the flexural and impact strengths of Hibiscus sabdariffa fiber-reinforced PMMA acrylic resins. From the 50 PMMA specimens fabricated, 40 were reinforced with Hibiscus sabdariffa fibers of different weight percentages (wt%) and 10 were unreinforced. There were two sample groups, each consisting of 25 PMMA specimens for flexural and impact strengths testing, respectively. Statistically, ANOVA and Bonferroni tests showed significant differences among the groups. Flexural strength results revealed that PMMA reinforced with 7.5 wt% fibers produced the highest mean value (101.20 ±3.654) MPa, followed by the 10 wt% (88.67±13.752) MPa, unreinforced (83.55±0.618) MPa, 5 wt% (75.58 ±3.676) MPa, and 2.5 wt% (68.84 ±4.293) MPa. Impact test results revealed that PMMA reinforced with 7.5 wt% fibers had the highest mean value (32.01±0.015) KJ/M2, followed by 10 wt% (24.48 ±0.010) KJ/M2, 5 wt% (23.54 ±0.006) KJ/M2, 2.5 wt% (17.89 ±0.006) KJ/M2 and the unreinforced (13.12 ±0.003) KJ/M2. Essentially, the inclusion of 7.5 wt% Hibiscus sabdariffa fibers improved the strength properties of PMMA denture base resins.

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Introduction

Polymethyl methacrylate (PMMA) denture base resin is commonly used in the fabrication of dentures as it is easy to manipulate and repair, is non-toxic, light in weight and can closely reproduce oral surface details[1,2,3.4.5,6]. PMMA denture base resins however, have low impact strength and poor fatigue resistance[7,8,9]. Modification of acrylic resins to improve the strength has therefore been advocated. Notably, different materials such as metals (powders, nets, plates and wires) and synthetic fibers (polyethylene, glass, carbon, kevlar and aramid) are used to improve the strength properties of PMMA dentures[10,11,12,13,14,15,16]. These reinforcing materials are difficult to manipulate during fabrication processes, and have a weak interfacial bond with the polymer matrix. Equally important, some of the aforementioned reinforcing materials are reported to be cytotoxic in the mouth[17,18,19.20,21,22,23,24].

The synthesis of natural fibers such as hibiscus sabdariffa (*H. sabdariffa*) in polymeric materials is reported to improve the overall strength properties of polymeric materials. Notably, natural fibers are non-toxic, lightweight, readily available, economical and are environmentally friendly to use[25,26]. As documented in the literature[27,28,29,30] *H. sabdariffa* fibers have high strength, high toughness and stiffness, low density and good tensile modulus. These fibers have been used in the reinforcement of polymeric materials such as urea formaldehyde, polyester, resorcinol formaldehyde and bio-epoxy resin. Despite the positive attributes of *H. sabdariffa* fibers there is less evidence on its use to reinforce PMMA

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denture base acrylic resins. This paper therefore aimed to examine the flexural and impact strengths of *H. sabdariffa* fiber-reinforced PMMA denture bases.

Materials and methods

Preparation of *H. sabdariffa* fibers

Hot water retting was used to manually extract fibers from the *H. sabdariffa* stems, which were collected from Hoedspruit, Mopani District of the Limpopo Province in South Africa. The fibers were heated in 3% hydrogen peroxide (H_2O_2) solution at 100°C for 45 minutes to remove dirt and other impurities. Subsequently, the fibers were rinsed several times by over flowing tap water and then dried at 50°C for 25 minutes. The fibers were then mercerized by soaking them in 8% NaOH solution for 8 hours. After the soaking time, the fibers were thoroughly washed in distilled water and dried in the open air for 24 hours. Further drying of the fibers at 70°C for 24 hours followed. Taking Xu *et al.*,[31] recommendation into consideration, the mercerized fibers were subsequently cut into 3mm fiber lengths.

Fabrication and Curing of PMMA Acrylic Specimens

As shown in Table 1, 50 PMMA (Vertex™, Vertex-Dental B. V., The Netherlands) acrylic specimens were fabricated. There were two sample groups, each containing 25 PMMA acrylic specimens for flexural strength and impact strength testing, respectively. Within each group, there were 5 unreinforced and 20 fiber-reinforced specimens of different weight percentages (wt%) namely, 2.5 wt%, 5.0 wt%, 7.5 wt%, and 10 wt%. Each wt% sub-category had 5 specimens. The monomer/polymer mixing ratio used was 0.60ml: 1g. A noteworthy point is that the pre-weighed fibers were soaked in monomer for 10 minutes to promote the complete wetting of fibers and its subsequent adhesion with acrylic powder. The unreinforced PMMA acrylic specimens (control group) were fabricated with strict compliance to the manufacturer's recommended mixing of monomer/polymer ratio that is 0.33ml: 1g.

Following ISO 1567: 1999 (ANSI/ADA specification 12) for denture base materials, two different sized rectangular metal dies were manufactured to ensure consistent molds were formed during the flasking and packing processes. PMMA acrylic specimens measuring 60mm x 10mm x 3mm (length x width x thickness) were fabricated for flexural strength testing, while 50mm x 10mm x 3mm (length x width x thickness) PMMA acrylic specimens were used for impact strength testing.

Tests performed (***Sample Groups***)	**Number of specimens**	
Flexural Strength (***Sample Group 1***)	Unreinforced	5
	Fiber-reinforced 2.5 wt%	5
	Fiber-reinforced 5.0 wt%	5
	Fiber-reinforced 7.5 wt%	5
	Fiber-reinforced 10 wt%	5
Impact Strength (***Sample Group 2***)	Unreinforced	5
	Fiber-reinforced 2.5 wt%	5
	Fiber-reinforced 5.0 wt%	5
	Fiber-reinforced 7.5 wt%	5
	Fiber-reinforced 10 wt%	5
	n =	**50**

Table 1. Overview of PMMA tests specimens.

Prior to the flasking process, the lubricated metal dies were invested to form plaster molds in one-half of the denture flask (Figure 1A). Once the plaster molds were dry-set, separating medium (Vertex™ Divosep liquid) was applied to the mold surfaces and allowed to dry. The second half of the denture flask was positioned and filled with plaster. After the final set of the plaster molds, the denture flasks were opened using a plaster knife and the metal dies were carefully removed from the molds (Figure 1B).

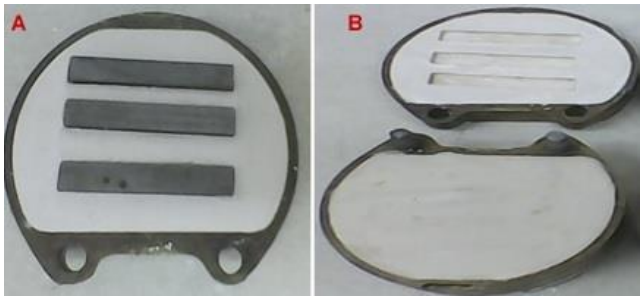

Figure 1. Metal dies investment process: **(A)** rectangular dies invested in flask; **(B)** Mold ready for packing.

The mold cavities were cleaned and coated with separating medium. Using the mixing ratios mentioned above, heat cure monomer and powder were mixed. The mix was thoroughly stirred to ensure random distribution of the fibers and the homogeneous mixing of the resin. At the dough stage of the acrylic mixture, the plaster molds were packed and the flasks were closed. It must be noted that the closed flasks were clamped, applying slow but incremental pressure until there was an edge-to-edge contact between the two halves of the flasks. The clamped flasks were placed into a curing bath (Mestra M-9™, Sondika- Bilbao, Spain) to cure the PMMA specimens according to the manufacturer's recommendations.

After curing, the clamped flasks were removed and left to bench cool. PMMA acrylic specimens were retrieved from the plaster molds and trimmed using a tungsten carbide bur (Cross-cut coarse – ISO 5001042370654; Bredent GmbH & Co KG) at 18000 rpm. All specimens were finished using a Silicon carbide abrasive paper (CC768; Deer Abrasives). A Vernier caliper was used to measure and ensure that the dimensions of the specimens in all planes were maintained. PMMA acrylic specimens which did not conform to the aforementioned measurements for flexural and impact testing were excluded. The PMMA specimens for impact tests were prepared with a 2mm V-notch at the center. All specimens were stored in distilled water at 37°C for 72 hours prior to mechanical testing at room temperature.

Mechanical Testing of PMMA Acrylic Specimens

In line with ASTM D 790 method, a three-point bending flexural testing was performed on PMMA acrylic specimens using a Lloyd™ testing machine (Lloyd Instruments Ltd, West Sussex, UK) at a crosshead speed of 5mm/min. The distance between the test jig supports was 50mm. The specimens were deflected at the center until fracture occurred. The force applied was recorded by the software attached to the testing machine. The strength was calculated using Equation 1 and the average results were recorded.

$$FS = 3FL/2bh^2 \ (MPa)^6 \qquad \textbf{(1)}$$

In Equation 1, FS is the flexural strength expressed in mega pascal (MPa); F is the maximum force applied expressed in newton (N); L is the distance between the support units expressed in millimeters (mm); b is the width of specimen expressed in millimeters (mm); and h is the height or thickness of specimen, prior to storage in water, expressed in millimeters (mm).

A Hounsfield™ charpy tester (Tensometer Ltd; Croydon, England) was used for impact testing in line with the ASTM D 256 testing method. Using Equation 2, the impact strength (IS) expressed in kilojoules per meter square (KJ/M^2) of the tested PMMA specimens were calculated and average results recorded.

$$IS = AE/T\ W \times 10^3 \quad (KJ/M^2)^{35} \qquad \textbf{(2)}$$

In Equation 2, AE is the absorbed energy; T is the thickness of the specimens expressed in millimeters; and W is the remaining width of specimen at the notch area expressed in millimeters.

Microscopic analysis

A scanning electron microscope (SEM Carl Zeiss, GmBH) that operated under control atmospheric conditions at 5 kV was used to analyze the fracture surfaces of specimens. Prior to the SEM analysis, samples were sputter coated with a thin layer of gold to ensure good conductivity of the materials and to prevent accumulation of electrostatic charge.

Statistical analysis

A one-way ANOVA test ($\alpha \leq 0.05$) was used to compare the average results between the sample groups. Bonferroni multiple comparison tests ($\alpha \leq 0.05$) were also used.

Results

Sample Groups	n	Mean (Std Dev)	Std Error	ANOVA p - value
Unreinforced	5	83.55 (0.61818)	0.27646	
Reinforced (2.5 wt%)	5	68.84 (4.29366)	1.92018	
Reinforced (5.0 wt%)	5	75.58 (3.67622)	1.64406	0.0001
Reinforced (7.5 wt%)	5	101.20(3.63543)	1.62581	
Reinforced (10 wt%)	5	88.67(13.75215)	6.15015	

Table 2. ANOVA flexural strength results.

Tables 2 and 3, and Figure 2 illustrate the flexural strength results of the unreinforced and fiber reinforced PMMA specimens. Statistically, and as seen in Table 2, there were significant differences ($p < 0.001$) in the flexural strengths between the unreinforced and the 7.5 wt% fiber-

reinforced PMMA specimens. Essentially, the 7.5 wt% fiber-reinforced PMMA specimens had the highest mean flexural strength (101.20MPa). This is a 21% improvement in comparison with the unreinforced specimens. The flexural strength of the 10wt% fiber-reinforced PMMA specimens was 88.67MPa, which is 6% improvement. The flexural strength of the PMMA specimens reinforced with 5.0 wt% and 2.5 wt% fibers were 9% (75.58MPa) and 17% (68.84MPa), lower than the unreinforced specimens respectively. The mean flexural strength of the unreinforced sample group was 83.55MPa.

Sample Groups	p- value
Unreinforced – Reinforced 2.5wt%	1.000
Unreinforced – Reinforced 5 wt%	1.000
Unreinforced – Reinforced 7.5 wt%	0.001
Unreinforced – Reinforced 10 wt%	1.000
2.5 wt% - 5.0 wt% Reinforced	1.000
2.5 wt% -7.5 wt% Reinforced	1.000
2.5 wt% - 10 wt% Reinforced	1.000
5.0 wt% - 7.5 wt% Reinforced	1.000
5.0 wt% - 10 wt% Reinforced	1.000
7.5 wt% - 10 wt% Reinforced	1.000

Table 3. Flexural strength results using Bonferroni test.

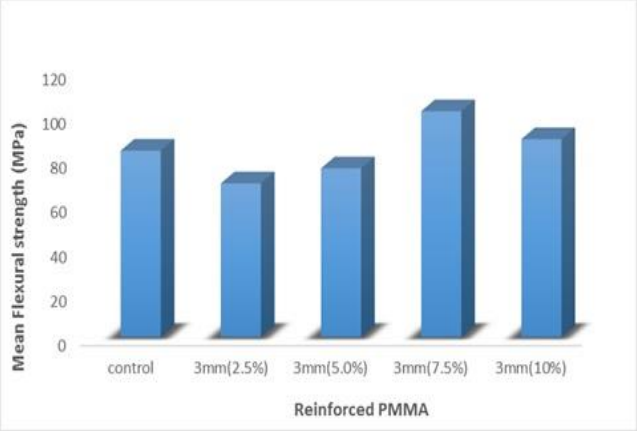

Figure 2. The mean flexural strength of the unreinforced and fiber-reinforced PMMA Specimens.

As presented in Tables 4 and 5, and Figure 3, all reinforced PMMA specimens had higher impact strengths than the unreinforced specimens ($p < 0.013$). The PMMA specimens reinforced with 7.5 wt% of fibers showed the highest mean impact strength (32.01KJ/m^2), followed by the 10 wt% (24.48KJ/m^2), 5 wt% (23.54KJ/m^2), and by the 2.5 wt% (17.89KJ/m^2) fiber-reinforced PMMA specimens. The unreinforced PMMA specimens had the lowest impact strength of 14.12KJ/m^2.

Sample Groups	n	Mean (Std. Dev)	Std Error	ANOVA p- value
Unreinforced	5	14.12 (0.00346)	0.00155	0.013
Reinforced (2.5 wt%)	5	17.89 (0.00695)	0.00311	
Reinforced (5.0 wt%)	5	23.54 (0.00640)	0.00286	
Reinforced (7.5 wt%)	5	32.01 (0.01536)	0.00687	
Reinforced (10 wt%)	5	24.48 (0.01022)	0.00457	

Table 4. ANOVA impact strength results.

Sample Groups	p - value
Unreinforced - 2.5 wt%	1.000
Unreinforced - 5.0 wt%	1.000
Unreinforced - 7.5 wt%	0.038
Unreinforced – 10 wt%	1.000
2.5 wt% - 5.0 wt% Reinforced	1.000
2.5 wt% - 7.5 wt% Reinforced	1.000
2.5 wt% - 10 wt% Reinforced	1.000
5.0 wt% - 7.5 wt% Reinforced	1.000
5.0 wt% - 10 wt% Reinforced	1.000
7.5 wt% - 10 wt% Reinforced	1.000

Table 5. Impact strength results using Bonferroni test.

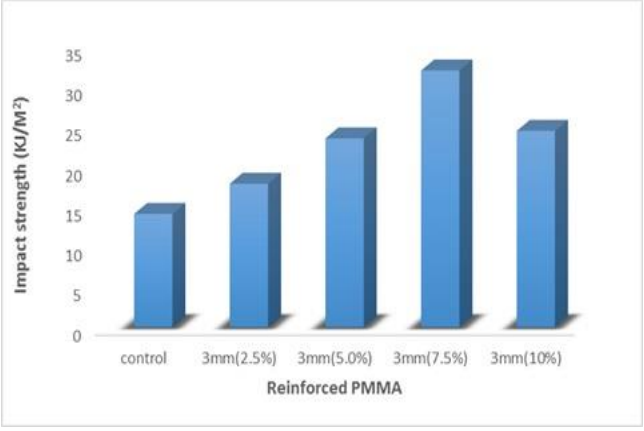

Figure 3. The mean impact strength of unreinforced and fiber-reinforced PMMA specimens.

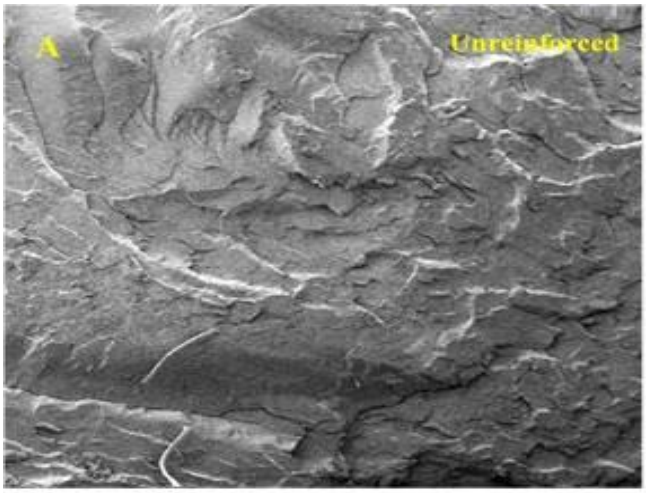

Figure 4. A

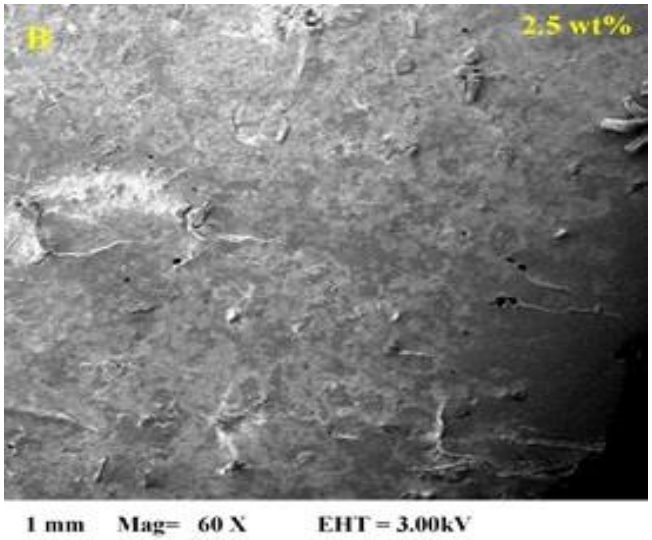

Figure 4. B

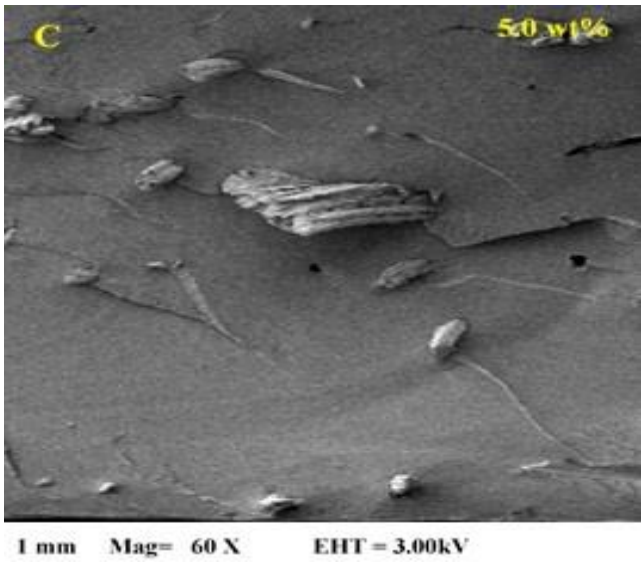

Figure 4. C

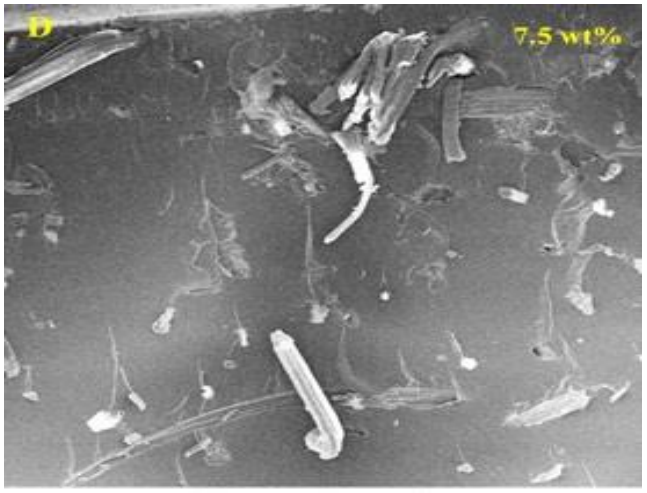

Figure 4. D

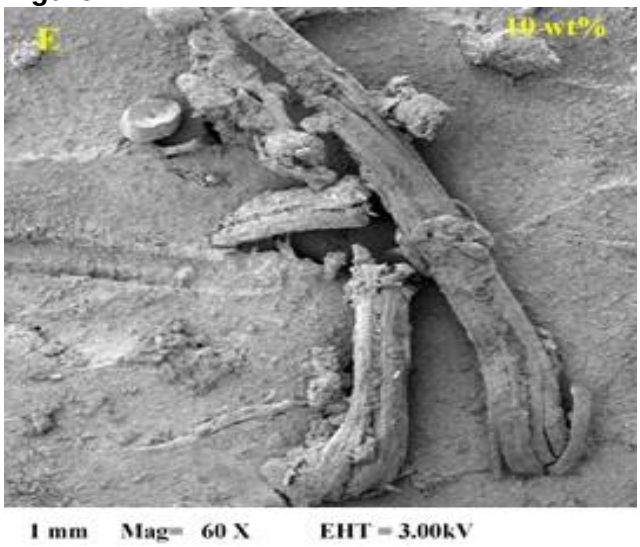

Figure 4. E

Figure 4. Micrograph of PMMA fracture surfaces. **(A)** Unreinforced; **(B)** 2.5 wt% fiber-reinforced; **(C)** 5.0 wt% fiber-reinforced; **(D)** 7.5% fiber-reinforced; and **(E)** 10% fiber-reinforced.

SEM analysis of the unreinforced PMMA specimens revealed a non-compact and irregular microstructure with grooves internally (Figure 4A). In contrast, and in relation to the wt% of fiber used, the microstructure of the PMMA specimens varied from rougher surfaces with micro voids and crack progression (Figures 4B and 4C); fiber agglomerated surface (Figure 4E); to smoothly organized and compact fracture surface devoid of micro voids (Figure 4D).

Discussion.

The strength properties of plant fiber-reinforced acrylics depends on, but are not limited to the fiber length, orientation, fiber content and fiber matrix interfacial bond. Arguably, the presence of the hydroxyl (OH) groups in natural fibers encourages the absorption of moisture. This causes poor wettability of the fiber and leads to weak interfacial bonding between the hydrophilic fibers and the hydrophobic polymer matrices. Mercerization therefore assists in achieving an effective interfacial bond between the fibers and polymers by critically imparting hydrophobic properties to the fibers. This supports the argument of Kabir et al.[23] that chemical treatment imparts effective optimization of interfacial bonding between fibers and matrices. The inclusion of mercerized *H. sabdariffa* fibers, particularly 7.5 wt% and 3mm in fiber length significantly increased the flexural and impact strengths of PMMA denture base resins.

The improvement in flexural strength could be attributed to pre-impregnating the fibers with the monomer, which enabled the dispersion of *H. sabdariffa* fibers within the polymer. Consequently, the interfacial bond between the fibers and the acrylic resin improved as pre-impregnation prepared the fiber surfaces to intimately bond with the polymer matrix. This argument is consistent with the research conducted by Murthy et al.[34], Ismaeel et al.[35] and Fatihallah[36], among other authors[2,8,18,32,33].

The ultimate bond strength between the *H. sabdariffa* fibers and the acrylic therefore determined the ability of the matrix to transfer load to the fibers[26]. Arguably, the decrease in the strength properties in the reinforced groups with 2.5 wt% and 5 wt% of *H. sabdariffa* fibers (Figures 4B and 4C) can be attributed to the insufficient quantity of fibers used. This critically breaks the molecular bond between the polymer chains thereby reducing the ability to transfer stress within the entire mass and subsequently leads to mechanical failure. The decrease in the strength of the 10 wt% of *H. sabdariffa* fiber-reinforced acrylics could be caused by agglomeration that is increased fiber to fiber contact (Figure 4E), which disrupts the transfer of stress to the reinforcing fibers. This resonates with Singha and Thakur[25] that excess fibers used decreases the strength properties of polymer matrix.

Ultimately, interfacial bonding between the *H. sabdariffa* fibers and the acrylics was achieved by adequate dispersion of fibers within the mass of resin. This reduced voids and prevented crack propagation thereby effectively improving the strength properties of denture base resins.

Conclusions

The prominent features of this paper showed that:

1. Using 3mm *H. sabdariffa* fiber lengths and fiber content of 7.5 wt% improves the flexural and impact strengths of fiber-reinforced PMMA acrylics.
2. Fiber content above 7.5 wt% reduces the strength properties of PMMA acrylics due to increased fiber to fiber contact and subsequent inability of the matrix to effectively transfer stress to the fibers.

Acknowledgements

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Declaration of Interest

The authors report no conflict of interest.

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