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The evaluation of some flexural properties of a denture base resin reinforced with various aesthetic fibers

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Abstract This study was performed to determine whether some flexural properties of a denture base resin material could be improved through reinforcement with five types of aesthetic fibers at 3% concentration by weight and in 2, 4, and 6 mm length. Five specimens of similar dimensions were prepared for each of the test groups; base resin and the same resin with glass, rayon, polyester, nylon 6 and nylon 6,6 fibers in three different lengths. Flexural properties were evaluated by using a 3-point bending test. A visual examination was also made to determine mode of fracture of the specimens. The incorporation of different fibers in varying lengths had no significant effect on flexural strength of the resin. The specimens reinforced with nylon 6,6 fibers of 6 mm length showed the highest flexural strength. Young's modulus and maximum load suggests that such reinforcement makes resin resistant to fracture.

1 Introduction

Denture fractures in clinical use may result from a large transitory force caused by an accident or a small force during repeated chewing [1]. Flexural failure of denture base materials is considered to be the primary mode of clinical failure and has been explained by the development of microscopic cracks in the areas of stress concentration [2]. With continued loading, these cracks fuse to an ever growing fissure that weakens the material. Catastrophic failure results from a final loading cycle that exceeds the mechanical capacity of the remaining sound portion of the material [3]. Flexural strength test is thought to be relevant, since it reflects the load arrangement in the clinical situation and it also gives an indication of the rigidity which is useful in comparing the denture base materials [1, 2, 4, 5].

Fracture of the dentures can be reduced by increasing the strength of the poly methyl methacrylate (PMMA) resin which is commonly used as a base material. After the many attempts made to improve the mechanical properties of PMMA resin, interest has turned to fiber reinforcement. The incorporation of various fibers into polymer matrix, such as carbon, aramid, ultra-high molecular weight polyethylene and glass fibers, has provided substantial improvements on impact and flexural strength, and fatigue resistance [6–28]. The fiber reinforcing mechanism has been explained by the principle that a relatively soft ductile polymer matrix is fully capable transferring an applied load onto the fibers via shear forces at the interface. In such a composite, the fibers will be the main load-bearing constituents while the matrix forms a continuous phase to surround and hold the fibers in place [6].

There are many studies which have focused on the effect of glass fibers on the mechanical properties of PMMA resin [1, 14, 18, 19, 21–27]. Reinforcement of the resin has been clinically successful, with good strength results, and glass fibers were considered to be aesthetically suitable for this purpose [3, 4, 21–27]. However, there is ongoing search for alternative base materials with better mechanical properties

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than the commonly used PMMA. Yunus et al. [5] have compared some flexural properties of a nylon-based denture base material with conventional base resins and found that it was less rigid. They have suggested that it could be used in cases where flexibility is desired. John et al. [15] have used nylon fibers as reinforcing agents and concluded that addition of these fibers to polymer matrix affected the flexural strength of composite positively. In the studies by Chen et al. [10, 29] the incorporation of polyester fibers has shown that reinforcement of the acrylic resin increased the impact strength many fold, but that larger amounts of the fiber decreased the surface hardness and had no significant effect on bending strength. Katsikas et al. [30] have studied some rheological properties of a conventional denture base resin reinforced with viscose rayon fibers, 2 mm in length and at four different concentrations. They have showed that increasing the percentage of fibers decreased the flow of the material and reduced the doughing time.

To date, no study has compared the flexural properties of acrylic resin reinforced with some aesthetic fibers. Therefore the present study was undertaken to evaluate the flexural effect of E-glass, polyester, rayon, nylon 6 and nylon 6,6 fibers as strengtheners. The notion behind the study was that the specimens reinforced with these fibers would give higher flexural strength values than the specimens without fiber. It was also thought that the strength values would increase in proportion to the length of the fiber used.

2 Materials and methods

2.1 Materials used

Five kinds of fibers supplied as threads [(E-glass (SMC3) (Cam Elyaf Sanayi, Kocaeli, Turkey); polyester (PE), rayon (RY), nylon 6 (N6) and nylon 6,6 (SM6) fibers (Kordsa, Kocaeli, Turkey)] were chosen to reinforce a conventional heat-polymerized acrylic resin (Meliodent, Heraus Kulzer, Germany). Rayon fiber is yellow in color, the others are white. The fibers were cut to lengths of 2, 4, and 6 mm without any surface treatment. The fiber content was determined as 3% by weight for trial groups. The amount of fiber added was based only on the premixed, measured resin powder weight (Sartorious AG, Gottingen, Germany), not on the combined powder and liquid or mixed resin weight.

2.2 Test specimen fabrication and conditioning

Five specimens were prepared for each length of the fibers tested and also for the control PMMA resin. A stainless steel mold was constructed as specified in the ASTM D790 M-92 [31] for flexural test. It had dimensions of 70 × 25 × 2 mm and rectangular shape. Wax patterns in sufficient number obtained using this mold were flasked and eliminated in the conventional manner.

Acrylic resin with or without fiber was mixed thoroughly at a powder/liquid ratio of 2.34 g/L mL in an agate mortar manually. After the acrylic dough reached a consistency, the mixture was packed into the gypsum mold created before by wax patterns, and the flask was placed under a hydraulic press (Rucker PHI, Birmingham, UK) and left for 5 min to remove any voids. Excess flash was trimmed away on trial packing. The flasks were fixed with clamps and cured in a 70 °C water bath for 1 h, then in boiling water for 30 min. After the completion of polymerization, the flasks were left to cool at room temperature before being opened. Deflasked specimens were manually polished with a 600-grit water-proof silicone carbide paper under the tap-water. All specimens were stored in distilled water at 37 °C for 24 h before the mechanical test. The test was performed at laboratory conditions.

2.3 Mechanical testing

Each group was subjected to flexural test under three point loading by using a cross head speed of 50 mm min^{-1} with a universal testing machine (Lloyd NK5, Lloyd instruments ltd., Fareham, Hampshire, UK), maximum load at break (N), deflection at maximum load (mm), flexural strength (MPa) and Young's modulus (MPa) were recorded. The flexural strength (F_s) and Young's modulus (E) were calculated using the following formulas:

$$F_s = 3Fl/2bh^2 \quad E = Fl^3/4bh^3d,$$

where F = the maximum load, l = the span length, b = the width of the test specimen, h = the thickness of the test specimen, and d = the deflection corresponding to load F at a point in the straight line portion of the trace.

2.4 Visual examination

After the flexural test, the mode of fractures on the specimens was examined visually. Photographs of some typical views were taken by a digital camera (Konica Minolta, Dimage Z3, Japan).

2.5 Statistical methods

After the collection of data, mean values and standard deviations were calculated in a SPSS statistical software program (10.0 version, SPSS Inc., Chicago, USA). The differences of control and the fiber groups at given lengths; and also effect of different lengths for the same fiber group were evaluated by the Kruskall Wallis analysis of variance and Friedman tests, respectively. For pairwise comparison of the same length of fibers Mann–Whitney *U* test, for the different length of fibers Wilcoxon test was used.

3 Results

3.1 Flexural test results

The results of the flexural test measurements of trial groups are given in Tables 1–4 showing in maximum load at break, deflection at maximum load, Young's modulus and flexural strength values for each combinations of fibers. The groups which had statistically significant differences by Mann–Whitney *U* test at 5% level are indicated using the same superscripted letters.

The Kruskall Wallis analysis of variance indicated that there were differences between control and test specimens reinforced with 2 mm length of fibers in terms of maximum load at break ($p = 0.008$) (Table 1), and flexural strength values ($p = 0.015$) (Table 4); whereas deflection at maximum load and Young's moduli values did not show any difference ($p = 0.349$ and 0.095, respectively) (Tables 2, 3). The highest maximum load at break and flexural strength values were recorded for control specimens (117.1 ± 11.0 N and 79.5 ± 7.5 MPa) and the lowest values for SM6 (90.5 ± 16.0 N and 61.7 ± 11.0 MPa). Mann–Whitney *U* test revealed statistical differences between control group and SM6, and RY fiber-reinforced specimens in terms of above parameters, and difference of maximum load at break was also found statistically significant between SMC3 fiber- and RY fiber-; SMC3 fiber- and SM6 fiber-reinforced specimens, respectively ($p < 0.05$).

With the use of 4 mm length of fiber, among the groups tested, no difference was observed in maximum load at

Table 1 The maximum load at break values of each group (N)

Groups	Length			Statistical test	
	2 mm ($\bar{x}$ ± SD)	4 mm ($\bar{x}$ ± SD)	6 mm ($\bar{x}$ ± SD)	Friedman	Wilcoxon
N6	104.7 ± 2.8	99.9 ± 9.8	93.6 ± 9.0^{e,g}	$p = 0.247$	$p > 0.05$
RY	90.6 ± 8.1^{a,c}	87.8 ± 14.9	94.6 ± 6.6^{f,h}	$p = 0.549$	$p > 0.05$
SMC3	115.1 ± 6.2^{c,d}	102.6 ± 13.0	100.8 ± 11.4^{i}	$p = 0.074$	$p > 0.05$
SM6	90.5 ± 16.0^{b,d}	102.2 ± 15.5	122.1 ± 11.4^{g,h,i,j,k}	$p = 0.015$	$p < 0.05$
PE	104.6 ± 19.0	96.2 ± 11.3	94.4 ± 12. 9^{j}	$p = 0.549$	$p > 0.05$
Control	117.1 ± 11.0^{a,b}	117.1 ± 11.0	117.1 ± 11.0^{e,f,k}		
	KW = 15.77	KW = 10.49	KW = 16.58		
	$p = 0.008$	$p = 0.064$	$p = 0.005$		

$n = 5$

The groups with same superscripted letters are significant by Mann–Whitney *U* test, at 5% level ($p < 0.05$)

Table 2 The maximum deflection mean values of each group (mm)

Groups	Length			Statistical test	
	2 mm ($\bar{x}$ ± SD)	4 mm ($\bar{x}$ ± SD)	6 mm ($\bar{x}$ ± SD)	Friedman	Wilcoxon
N6	6.5 ± 1.1	7.8 ± 0.6	7.4 ± 1.5	$p = 0.058$	$p > 0.05$
RY	7.1 ± 0.8	7.0 ± 0.3	6.5 ± 0.8	$p = 0.449$	$p > 0.05$
SMC3	6.6 ± 0.5	6.0 ± 0.8	6.0 ± 0.9	$p = 0.549$	$p > 0.05$
SM6	6.3 ± 0.6	6.4 ± 1.0	5.4 ± 0.3	$p = 0.165$	$p > 0.05$
PE	6.3 ± 0.5	6.9 ± 0.8	7.0 ± 1.1	$p = 0.550$	$p > 0.05$
Control	7.1 ± 1.1	7.1 ± 1.1	7.1 ± 1.1		
	KW = 5.58	KW = 10.38	KW = 10.50		
	$p = 0.349$	$p = 0.065$	$p = 0.063$		
	$p > 0.05$	$p > 0.05$	$p > 0.05$		

$n = 5$

Table 3 The Young's modulus mean values of each group (MPa)

Groups	Length			Statistical test	
	2 mm ($\bar{x} \pm$ SD)	4 mm ($\bar{x} \pm$ SD)	6 mm ($\bar{x} \pm$ SD)	Friedman	Wilcoxon
N6	1786.7 ± 251.7	1469.6 ± 248.3^{a,d,g}	1437.2 ± 209.6^{j}	$p = 0.247$	$p > 0.05$
RY	1522.1 ± 231.5	1393.7 ± 341.8^{b,e,h}	1508.6 ± 150.3^{k}	$p = 0.449$	$p > 0.05$
SMC3	2060.9 ± 169.0	1803.7 ± 193.7^{d,e,f}	1830.3 ± 69.7^{l}	$p = 0.247$	$p > 0.05$
SM6	1554.4 ± 245.4	1792.5 ± 223.1^{g,h,i}	2365.4 ± 310.8^{j,k,l,m,n}	$p = 0.015$	$p < 0.05$
PE	1778.1 ± 289.0	1372.3 ± 269.0^{c,f,i}	1677. 9 ± 291.6^{m}	$p = 0.022$	$p < 0.05$
Control	1761.5 ± 351.7	1761.5 ± 351.7^{a,b,c}	1761.5 ± 351.7^{n}		
	KW = 9.37	KW = 11.87	KW = 17.29		
	$p = 0.095$	$p = 0.037$	$p = 0.001$		

$n = 5$

The groups with same superscripted letters are significant by Mann–Whitney U test, at 5% level ($p < 0.05$)

Table 4 The flexural strength mean values of each group (MPa)

Groups	Length			Statistical test	
	2 mm ($\bar{x} \pm$ SD)	4 mm ($\bar{x} \pm$ SD)	6 mm ($\bar{x} \pm$ SD)	Friedman	Wilcoxon
N6	71.8 ± 4.7	68.0 ± 6.7	63.7 ± 6.0^{c,e}	$p = 0.247$	$p > 0.05$
RY	62.0 ± 5.5^{a}	61.2 ± 12.7	63.8 ± 4.8^{d,f}	$p = 0.819$	$p > 0.05$
SMC3	77.4 ± 5.1	71.2 ± 7.9	71.3 ± 7.3	$p = 0.015$	$p < 0.05$
PE	71.3 ± 13.0	65.8 ± 7.7	67.0 ± 6.9^{g}	$p = 0.449$	$p > 0.05$
Control	79.5 ± 7.5^{a,b}	79.5 ± 7.5	79.5 ± 7.5^{c,d}		
	KW = 14.05	KW = 9.25	KW = 17.29		
	$p = 0.015$	$p = 0.099$	$p = 0.001$		

$n = 5$

The groups with same superscripted letters are significant by Mann–Whitney U test, at 5% level ($p < 0.05$)

fracture, deflection at maximum load, and flexural strength values ($p > 0.05$), however they were found to be different in respect to Young's moduli ($p = 0.037$) (Tables 1–4). SMC3 fiber-reinforced specimens yielded the highest value (1803.7 ± 193.7 MPa) and the pairwise comparison showed that this group was statistically different from RY fiber-, PE fiber- and N6 fiber-reinforced specimens. Moreover, statistically significant differences were also detected between following specimens reinforced with fibers of SM6 and N6, RY, PE; between control specimens and RY, N6, PE, respectively ($p < 0.05$) (Table 3).

Control and trial groups reinforced with 6 mm length of fibers, which were analyzed by Kruskal Wallis of variance, displayed significant differences within themselves in terms of maximum load at break, Young's moduli as well as in flexural strength values ($p = 0.005$, 0.001, and 0.001) (Tables 1, 3, 4). The mean values of deflection at maximum load were not different ($p = 0.063$). The force required to break the specimens was found to be highest for the SM6 fiber-reinforced specimens (122.1 ± 11.4 N), and Young's modulus and flexural strength values of these specimens were also higher than those of the others (2365.4 ± 310.8 and 83.2 ± 7.8 MPa, respectively). Young's modulus and maximum load values of SM6 fiber-reinforced specimens were found to be statistically different from others groups tested ($p < 0.05$) (Tables 1–3).

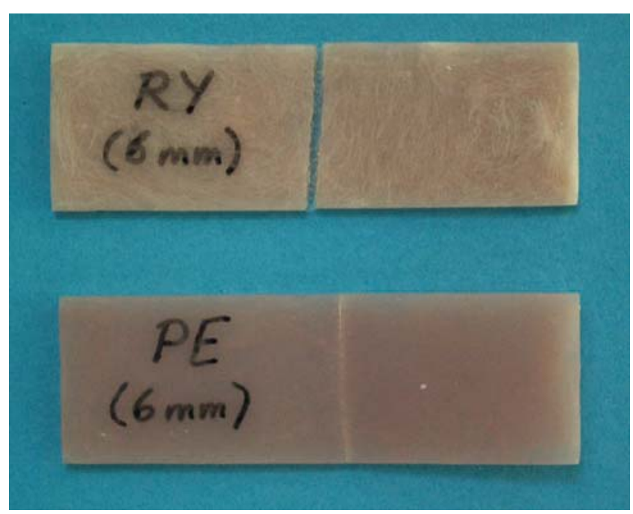

Fig. 1 Rayon and polyester fiber-reinforced specimens after fracture with fibers holding the sections together

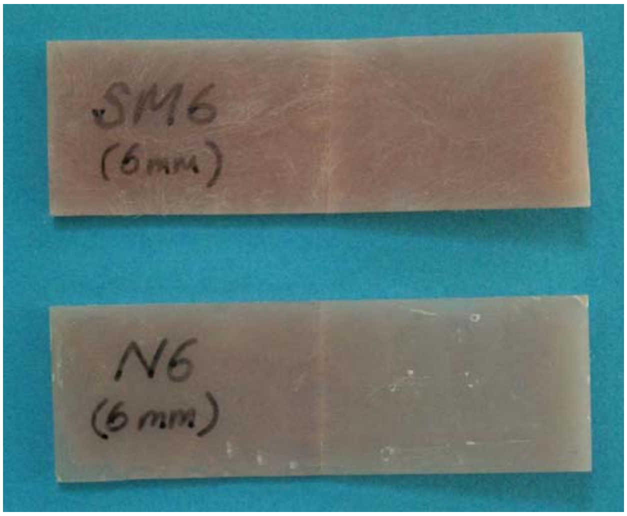

Fig. 2 Nylon 6,6 and nylon 6 fiber-reinforced specimens after fracture. Specimens remained bound together by fibers across the fracture line

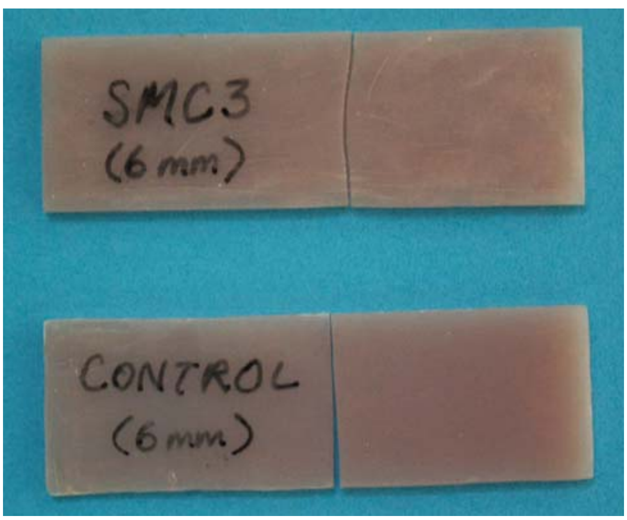

Fig. 3 Completely fractured specimens reinforced with glass fiber and without fiber

Table 5 The results of visual evaluation of fracture lines

Groups	Mode of fractures		
	2 mm	4 mm	6 mm
N6	4 CF/1 NCF	–/5 NCF	–/5 NCF
RY	5 CF/–	5 CF/–	1 CF/4 NCF
SMC3	5 CF/–	5 CF/–	5 CF/–
SM6	2 CF/3 NCF	–/5 NCF	–/5 NCF
PE	4 CF/1 NCF	4 C/1 NCF	–/5 NCF
Control	5 CF/–		

CF = Completely fracture, NCF = Not completely fracture

The use of different lengths of fibers had no noticeable effect on flexural properties of the trial groups, except SM6 fiber-reinforced specimens which showed important increase in flexural strength and maximum load at break and Young's moduli as length of fiber increased ($p < 0.05$) (Tables 1, 3, 4). These specimens also showed the lowest deflection at maximum load (5.4 ± 0.3 mm) when 6 mm length of fiber was used, though the difference was not significant ($p > 0.05$). Increased lengths of fibers led to a decrease in Young's moduli on the PE fiber-reinforced specimens ($p < 0.05$) (Table 3).

3.2 Visual evaluation

Visual inspections of fractures on specimens showed that although the specimens with reinforced 6 mm length of fibers were fractured, the fractures were held together via the extended fibers. However, control and SMC3 fiber-reinforced specimens appeared to be fractured completely (Figs. 1–3) (Table 5).

4 Discussion

Development of the fibrous composite materials in industry has inspired a new approach to improve the performance of acrylic resins [16]. By the addition of various fibers to resins, successful results have been reported on the strength of denture base polymers.

This study investigated the strengthening capacity of five different aesthetic fibers in the acrylic resin and also the effect of fiber length on flexural strength. Nylon 6, rayon, E-glass, nylon 6,6 and polyester fibers which were equivalent to commercial formulations were used 2, 4, and 6 mm in length as the acrylic resin reinforcers. They were added into resin at 3% concentration by taking into account the results obtained from previous studies [12, 30]. Gutteridge [12] has used 6 mm polyethylene fibers for acrylic resin reinforcement and tested the impact strength with favorable results. He has reported that if the fiber concentration were higher than 4%, it would become difficult to manipulate. Katsikas et al. [30] used 2 mm rayon fibers as an acrylic resin reinforcer at different concentrations ranging from 0.1% to 3% and found that the viscosity was increased with the amount of fiber incorporated.

Results of the present study revealed that the use of randomly oriented 2 mm length of fiber incorporation did not improve the flexural strength of resin specimens, because the highest value recorded was for PMMA specimens without fiber (79.5 MPa). Among the trial groups, glass fibers were generally found to be most effective in reinforcing the resin, thus giving the highest strength value for the specimens reinforced with 2 mm fiber (77.4 MPa). Increase in fiber length led to a decrease in the flexural strength of all fiber-reinforced specimens except for nylon 6,6 which gave the highest value in 6 mm length (83.2 MPa) (Table 4).

The decrease in flexural strength values of our specimens may be attributed to several factors. The first of these could be using the fibers without surface treatment. Untreated fibers are known to act as inclusion bodies and actually weaken the resin system [18]. Also, the fact that the fibers are free to orient themselves randomly means that they do not contribute significantly to improving the strength. Stipho [19] has evaluated the effect of 2 mm length of glass fiber reinforcement on the transverse strength of an autopolymerizing resin and found that 1% by weight glass fiber had the best effect, but that higher percentage of glass fiber weakened the resin significantly. The addition of fibers might disturb the main matrix continuity and interfere with stress transfer between two materials (fiber and polymer), or within the same material.

Discontinuities and weak adhesion between PMMA and fiber might also result in a decrease in flexural strength [20]. The high viscosity of the acrylic dough may lead to poor wetting of fibers [3, 7, 30]. Polymers used in prosthetic dentistry are often multiphase acrylic resin systems made from prepolymerized powder beads (predominantly PMMA) and a liquid of monomers such as methyl methacrylate (MMA) with a cross-linking monomer. Because such a polymer–monomer mixture or dough has relatively high viscosity, adequate impregnation of reinforcing fibers with resins has been difficult to achieve. Impregnation of reinforcing fibers with the resin allows fibers to come into contact with polymer matrix. This is a prerequisite for bonding of fibers to polymer matrix, and thus for strength of the composite [27]. In the present study, the use of untreated fibers might lead to a weak bonding with resin, and may explain lower flexural strength values of fiber-reinforced groups compared to PMMA without fiber group.

The differences in the flexural strength of specimens tested might reflect the type of reinforcement and these findings were in agreement with results in the literature [3, 4, 10, 15, 18, 29]. John et al. [15] have compared the flexural strength of conventional PMMA resin against that reinforced with glass, aramid, and nylon fibers in loose form. They have found that all fiber-reinforced specimen bases had a higher fracture resistance than non-reinforced PMMA specimens. However, Chen et al. [10] have not observed any remarkable difference in flexural strength with the use of polyester, glass, and aramid fibers in different lengths or concentrations. They have reported that large amounts of fiber incorporation had little effect on bending strength. Although the flexural strength values of glass-reinforced specimens were not found to be statistically different from those of other specimens, this group exhibited better flexural strength than the other fiber-reinforced specimen groups in our study. The composition of E-glass fibers consisted of 55% SiO_2, 22% CaO, 15% Al_2O_3, 6% B_2O_3, 0.5% MgO, <1.0% Fe + Na + K (manufacturer's information). Because of high alumina and low alkali and borosilicate content, E-glass fibers have been claimed to be superior in flexural strength [18]. Moreover, because modulus of elasticity of glass fibers is very high, most of the stresses are received by them without deformation [15].

In the present study, the best flexural strength value was obtained by the use of nylon 6,6 fibers in 6 mm length. Nylon fibers are polyamide in nature and are based primarily on aliphatic chains [15]. Amide groups are extremely polar and hydrogen bonded with each other. The backbone of nylon is regular and symmetrical, so forms very good fibers [33, 34]. The chief advantage of nylon lies in its resistance to shocks and repeating stress [15]. Depending on the carbon chain length of the diacide, different types of nylon can be produced. Nylon of different types can have distinct physical properties [33, 34]. John et al. [15] have found that nylon fiber-reinforced specimens had better flexural strength than unreinforced specimens. In contrast, we found that the specimens reinforced with nylon 6 had lower flexural properties than those of control resin. This could be due to differences in test conditions and/or materials used. However, strengthening performance of nylon 6,6 fibers was found to be higher than nylon 6. This could be due to their different chemical structure.

For polyester fibers, Chen et al. [10, 29] have reported that when short length polyester fibers were added in a randomly oriented fashion, the denture was processed easily by the traditional procedure without causing any aesthetic problems. They have also found multiple-fold improvement on impact strength, but little effect on bending strength. The flexural strength results obtained in this study were similar to those of glass fiber reinforcements [10, 29]. In contrast, polyester fibers did not provide significant increase in strength value with the increase of fiber length in the present study.

Our previous study [35] have been conducted to observe the changes in impact resistance of a denture base resin reinforced with the same fibers. Results have indicated that the impact energy tended to increase with fiber length and that the highest value was recorded for rayon fiber reinforced specimens of 6 mm length. However, in the present study the rayon fiber reinforcement produced lower strength results than the other trial groups. In addition, it appeared that incorporation of these fibers into resin led to a rust brown color which may not be aesthetically acceptable in visible locations.

It has been stated that the average breaking force of acrylic resin should not be less than 55N [2, 13]. In this respect, all specimens satisfied this requirement and the highest value was recorded for the specimens reinforced with nylon 6,6 fibers in 6 mm length (122.1 N). Deflection results revealed that the specimens reinforced with this fiber had the least deformation before the fracture (Tables 1–4). The greatest permanent deformation was exhibited by the specimens reinforced with nylon 6 fibers, followed by those with rayon fibers (Table 2). This might suggest that the use of nylon 6 and rayon fibers as resin strengtheners may lead to a greater degree of permanent deformation of denture base without obvious clinical feature.

The Young's modulus is a constant that relates to the stress and strain in the linear elastic region and is a measure of the stiffness of the material in terms of transfer of stress [31]. Ideally, the Young's modulus of the fibers should be greater than the Young's modulus of the matrix so that at a given strain, the fibers absorb far more stress [14]. In the trial groups reinforced with 6 mm length of fiber, Young's modulus of the specimens with nylon 6,6 fibers increased markedly; this might indicate that such a reinforced denture base material might withstand the applied stress without permanent deformation.

Visual examination showed that all types of fibers, except for rayon, did not compromise the aesthetic qualities of PMMA. One of the interesting observations was the mode of crack propagation. The specimens without any fiber and reinforced with glass fiber were broken into two pieces under maximum load, and showed catastrophic failure. However, in the specimens reinforced with other fibers, a crack occured mostly on the tension side, but did not propagate through the compression line. In spite of decreasing the flexural strength, the extended fibers appeared to hold two pieces together (Figs. 1–3).

Further work is needed to better understand the nature of the reinforcement conferred by these fibers on the resin system and additional mechanical properties should also be studied.

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