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The static strength and modulus of fiber reinforced denture base polymer

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Summary *Objectives*. Partial fiber reinforcements have been employed to strengthen dentures both during repair and in the manufacturing process. The reinforcing fibers can be evenly distributed in the denture base polymer or alternatively fiber-rich phase in the denture base polymer can form a separate structure. The aim of this study was to determinate static three-point flexural strength and modulus of denture base polymer that had been reinforced with different fiber reinforcements.

Methods. The test specimens ($3\times5\times50$ mm) were made of auto-polymerized denture base polymer and reinforced with different fiber reinforcements. The test groups were: (A) no fibers; (B) non-impregnated polyethylene fibers; (C) light-polymerized monomer impregnated glass fibers; (D) porous polymer preimpregnated glass fibers and (E) light-polymerized monomer–polymer impregnated glass fibers. The fibers were oriented parallel to the long axis of the specimen and embedded into the denture base resin on the compression side ($n=7$) or tension side ($n=7$). Dry specimens were tested with three-point static flexural strength test set-up at crosshead speed of 5 mm/min.

Results. The statistical analysis by two-way analysis of variance showed that the brand and the location of the fiber reinforcements significantly influenced the flexural strength ($p<0.0001$). However, the location of the fiber reinforcements did not influence the flexural modulus ($p<0.722$).

Significance. The results suggest that impregnated and preimpregnated fibers reinforce denture base polymer more than non-impregnated fibers. Fiber reinforcements placed on the tensile side resulted in considerably higher flexural strength and flexural modulus values compared with same quantity of fibers placed on the compression side.

Introduction

Acrylic resin, based on polymethylmethacrylate (PMMA), is one of the materials routinely used for

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the manufacture of removable dentures. The favorable working characteristics, ease of manipulation and polishability, its use in combination with inexpensive equipment, stability in the oral environment and the aesthetics of acrylic resin have resulted in its extensive us as a denture base polymer. However, the acrylic resin denture base polymer has not fulfilled all the requirements in terms of optimum mechanical properties [1] due to its brittle nature under its glass transition temperature (T_g) of approximately 110 °C [2], and its susceptibility to cyclic loading. For this reason, fatigue fracture of dentures is a common clinical manifestation [3–13]. It is unlikely that a complete denture would be broken by one heavy biting cycle due to the high volume of the denture base polymer and the geometry of the base plate [10]. However, the thin denture base plates of removable partial dentures can fracture by one loading cycle as a result of a poorly balanced occlusion [12] and a fracture of this kind could result from a static load.

Conventional methods employed to reinforce denture base polymers generally involve the use of either metal wires or plates, however, their influence is minor [3–6]. Fiber-reinforced composites (FRC) have been introduced [14–32] to overcome the problem of denture factures by improving the mechanical properties of the denture base polymer. Partial fiber reinforcements, namely accurate placement of a relatively small quantity of fibers in the denture base polymer, have been employed to strengthen dentures both during repair and in the manufacturing process [5,6].

The types of fibers that have been used to reinforce denture base polymers include aramid fibers [14,15], carbon/graphite fibers [16], ultra-high molecular weight polyethylene (UHMWP) fibers [17–20] and glass fibers [5,6,14,21–32]. Although UHMWP fibers have relatively good mechanical properties, there are reports that poor adhesion of the fibers to the polymer matrix, even with the aid of plasma treatment, does not considerably increase the mechanical properties of the denture base polymer [18]. It has been previously shown that glass fibers can be adhered to the resin by silane coupling agents and can be used as an effective reinforcement [32]. In recent years, several brands of fiber reinforcements have become available, however, in the opinion of the authors there is a distinct lack of comparative information of the reinforcing effect of the various fiber reinforcements available in the dental literature. Therefore, the aim of the current study was to determine the static three-point flexural strength and flexural modulus of a denture base polymer that has been reinforced with fiber reinforcements commonly available to dentist and dental technicians.

Materials and methods

The test specimens utilized in the current study ($3 \times 5 \times 50$ mm) were manufactured from clear auto-polymerizing denture base resin (Palapress, Heraeus Kulzer GmbH & Co. KG, Wehrheim, Germany) and reinforced with four different brands of fiber reinforcements (Table 1, Fig. 1). The powder/liquid ratio of resin was 107 g/ml and the resin was polymerized in distilled water maintained at 55 ± 2 °C under air pressure of 300 kPa for 15 mins in a pneumatic curing unit (Ivomat Typ IPR, Ivoclar,

Table 1 Classification of test groups and the fiber reinforcing materials used in the current study.

Group	Manufacturer	Fiber type	Type	Preimpregnation resin
A. Control		None		
B. Ribbond	Ribbond Inc., Seattle, USA	Woven ribbon polyethylene UHMWP[a]	Non-preimpregnated	-
C. FibreKor	Jeneric/Pentron, Wallingford, USA	Continuous unidirectional S-glass fiber	Light-polymerizing resin impregnated Shade Clear 2K bundle, 3 mm wide	Bis-GMA[b]
D. Everstick	StickTech Ltd, Turku, Finland	Continuous unidirectional E-glass fiber	Light-polymerizing resin impregnated	Bis-GMA with, PMMA[c]-bis-GMA matrix
E. Stick	StickTech Ltd, Turku, Finland	Continuous unidirectional E-glass fiber	Porous polymer preimpregnated fiber	PMMA

[a] Ultra-high molecular weight polyethylene fiber.
[b] 2, 2-bis[4-(2-hydroxy-3-methacryloxypropoxy)phenyl]-propane.
[c] Polymethylmetacrylate.

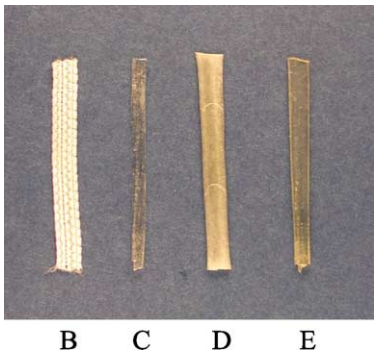

Figure 1 Fiber reinforcements used in test groups in the current study. Group (B) Ribbond; Group (C) FibreKor; Group (D) Stick; and Group (E) everStick fiber reinforcement.

Schaan, Liechtenstein). The test specimens in group A were manufactured without fiber reinforcement and were employed as the control group in the study. The light-polymerized resin impregnated fiber reinforcements, namely, FibreKor and everStick (group C and E, respectively,) were polymerized with a light-curing guide (Optilux 501, Sds Kerr, Danbury, CT). Output intensity was measured with an in-build radiometer to be 800–850 mW/cm^2. The fiber reinforcements were light-polymerized by placing the light-curing tip on the reinforcement. The 10 mm diameter of the light-curing tip was employed to light-polymerize over five overlapping areas for 40 s on both sides of the specimens prior to embedding the fiber reinforcements in the denture base resin. The non-preimpregnated fiber reinforcement, namely, Ribbond (group B) and PMMA preimpregnated Stick reinforcement (group D) were wetted with a mixture of denture base resin. All the fiber reinforcements were then embedded in a polyvinyl siloxane Lab-putty mold (Coltène, Alstätten, Switzerland) ($3.2\times5.2\times50.2$ mm) that had been partially filled with a 1 mm layer of denture base resin.

The major orientation of the fibers in groups C, D and E (FibreKor, Stick and everStick) was parallel to the long axis of the specimen, whereas ribbon type group B (Ribbond) fibers contained also fibers orientated against the long axis of the specimen. The fibers subsequently were placed on top of the denture base resin layer. In the case of group D specimens (Stick) the fibers were distributed and filled the entire volume of the mold as the denture base resin mixture swelled the polymer matrix preimpregnation of the reinforcement. In groups B, C and E the fiber reinforcement was covered with a further layer of denture base resin. Following polymerization, the specimens were then wet ground (Stuers LabPol-21, Stuers A/S, Copenhagen, Denmark) with successively finer grades of silicon carbide abrasive papers from P300 to P1200 (Stuers A/S, Copenhagen, Denmark) to the predermined dimensions of the specimens ($3\times5\times50$ mm) and the test specimens were stored dry prior to testing. Static three-point-flexural strength testing was performed in air to determine the ultimate flexural strength and the associated flexural modulus at a crosshead speed of 5 mm/min (Model LRX, Lloyds Instruments Ltd, Fareham, UK). The distance between the supports of the test specimens was 20 mm and the load–deflection curves were recorded with Nexygen-software (Lloyd Instruments Ltd, Fareham, UK). The ultimate flexural strength σ (MPa) was determined for the specimens that contained the main part of the fiber reinforcement when either placed in tension ($n=7$) or in compression ($n=7$) in accordance with Eq. (1), where F is the maximum load (N) applied at the highest point of load–deflection curve, l is the specimen span length (mm), and b is the breath of the specimen (mm) and h is the thickness of the test specimen (mm). The flexural modulus E (GPa) of the specimens was also determined when the fibers were placed in tension ($n=7$) or in compression ($n=7$) in accordance with Eq. (2) where S is the stiffness (N/m) and d is the deflection (mm) corresponding to a load F (N) at a point in the linear portion of the trace to provide the stiffness value.

$$\sigma = \frac{3Fl}{2bh^2} \tag{1}$$

$$E = \frac{Sl^3}{4bh^3d} \tag{2}$$

Following flexural strength testing, the specimens were visually examined in an attempt to differentiate the origin of the fracture and the failure mode. The failure modes were classified as (1) intact test specimen; (2) the fracture path was arrested at the fibers; (3) the fracture path passed the fibers but did not result in complete test specimen fracture; and (4) the test specimen was broken into two pieces.

The fiber content of the test specimens containing glass fiber reinforcement was measured by combustion analysis with a burnout furnace (Radiance Multi-stage msc, Jelrus International, Hicksville, NY, USA). The test specimens were dried in a desiccator for 2 days at room temperature and weighed to an accuracy of 0.001 mg prior to combustion. In order to combust the denture base polymer matrix, the temperature was raised 12 °C per min until the temperature reached 700 °C. Test specimens were maintained at 700 ± 25 °C for 1 h and reweighed. The fiber content as a percentage

by volume (V_f) (vol.%) was assessed in accordance with the equation

$$V_f = \frac{(W_f/D_f)}{(W_f/D_f) + (W_p/D_p)} \quad (3)$$

where W_f is the weight of the fiber, D_f is the density of the glass fiber (2.54 g/cm^3), W_p is the weight of the denture base resin, and D_p is the density of the denture base resin (1.19 g/cm^3).

The fiber volume of the group B (Ribbond) with the UHMWP fibers was determined based on fiber content as percentage by weight. The fiber content as percentage by volume was determined in accordance with the following equation

$$V_f = \frac{(W_f/D_f)}{V_s} 100\% \quad (4)$$

where W_f is the weight proportion of fiber reinforcement (weight of 50×4 mm Ribbond, 0.028 g), D_f is density of reinforcement (polyethylene, 0.97 g/cm^3) and V_s is the size of the specimen (3×5×50 mm = 0.75 cm^3).

The flexural strength and flexural modulus of the specimens tested with the fibers in compression or tested in tension were analyzed utilizing a two-way analysis of variance (ANOVA) at a 95% significance level using a statistical software package (Statistical Package for the Social Sciences for Windows, Rel. 10.0.5, Chicago, IL, USA).

Results

Testing fiber reinforcements in tension

The seven control specimens (group A) had a mean three-point flexural strength and associated SD of 128±3 MPa and three-point flexural modulus of

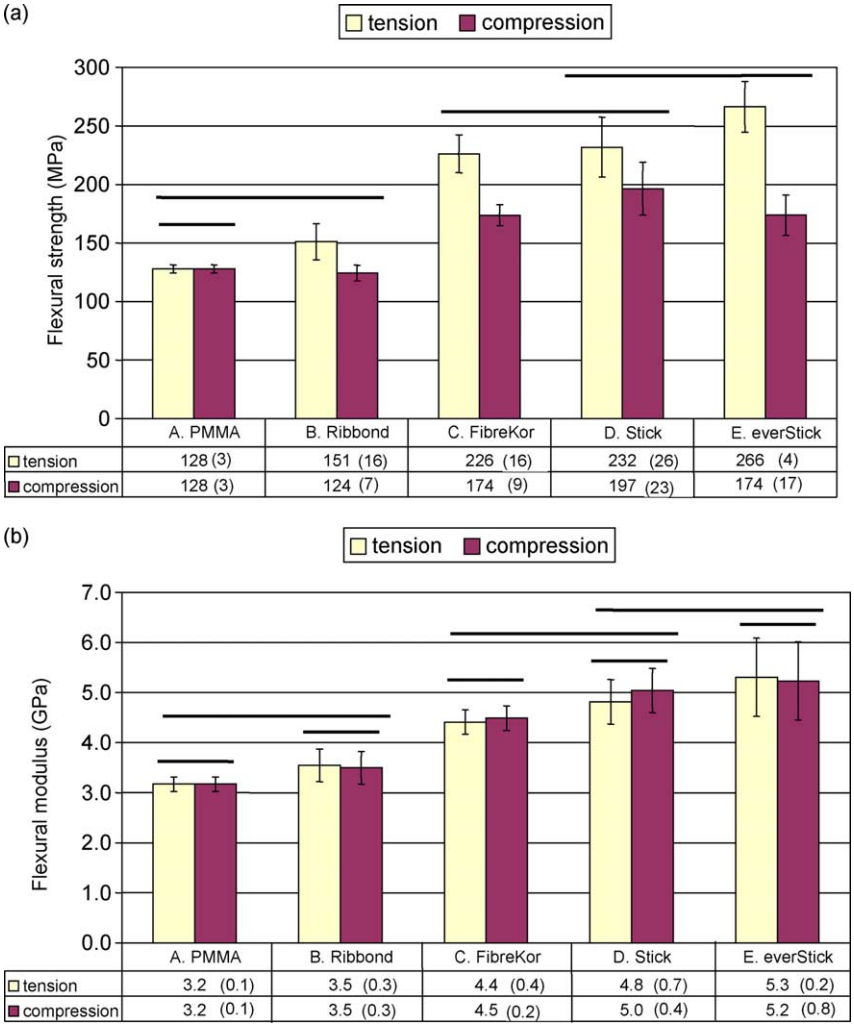

Figure 2 (a) Three-point flexural strength and (b) calculated three-point flexural modulus of the test specimens reinforced with various brands of fiber reinforcements and two fiber locations (compression or tension side). Vertical bars represent SD. Horizontal lines above the bars indicate groups that do not differ statistically from each other.

Table 2 The failure mode of the test specimens reinforced with different brands of fibre reinforcements located where the reinforcement was tested in tension or compression.

	Group A, PMMA	Group B, Ribbond	Group C, FibreKor	Group D, Stick	Group E, everStick
Intact test specimen					
Compression	-	-	-	7/7	-
Tension	-	-	-	-	-
Fracture stopped at fibers					
Compression	-	-	1/7	-	-
Tension	-	-	1/7	6/7	-
Fracture passed the fibers					
Compression	-	7/7	2/7	-	7/7
Tension	-	7/7	-	1/7	6/7
Broken into two pieces					
Compression	7/7	-	4/7	-	-
Tension	7/7	-	6/7	-	1/7

3.2 ± 0.1 GPa, respectively (Fig. 2a and b). Following visual examination, it was identified that all the specimens had fractured into two pieces (Table 2). When the light-polymerized resin impregnated glass fiber reinforced specimens (groups C and E, respectively,) were tested with the reinforcement placed in tension, the mean three-point flexural strength and calculated three-point flexural moduli were 226 ± 16 MPa and 4.4 ± 0.4 GPa, respectively, for group C and 266 ± 4 MPa and 5.3 ± 0.2 GPa, respectively, for group E specimens (Fig. 2a and b). In group C, six of the seven test specimens fractured into two pieces and in one of the specimens the fracture had arrested at the fibers. In group E, fracture path passed the fibers in six specimens but did not result in specimen fracture and the remaining test specimen was fractured broken into two pieces (Table 2). The non-preimpregnated UHMWP fiber reinforcement for group B specimens resulted in a mean three-point flexural strength of 151 ± 16 MPa and a calculated three-point flexural modulus of 3.5 ± 0.3 GPa (Fig. 2a and b). The polymer preimpregnated glass fiber reinforced test specimens (group D) resulted in a three-point flexural strength of 232 ± 26 MPa and calculated three-point flexural modulus of 4.8 ± 0.7 GPa (Fig. 2a and b). In all of the specimens in group B, the fracture path passed the fibers but did not result in specimen fracture while in six specimens of group D the fracture path was arrested at the fibers and in one specimen fracture path had passed the fibers (Table 2).

Testing with the fibers in compression

When the fiber reinforcement was located in the compression side the light-polymerized resin preimpregnated glass fiber reinforcements (groups C and E) resulted in a mean three-point flexural strength of 174 ± 9 MPa and a calculated three-point flexural moduli of and 4.5 ± 0.2 GPa for group C, and 174 ± 17 MPa and 5.2 ± 0.8 GPa, respectively, for group E (Fig. 2a and b). Following visual examination, it was identified that four of the specimens in group C were fractured into two pieces, in two specimens fracture had passed the fibers and in one test specimen fracture had arrested at the fibers. In group E, all of the test specimen fracture path had passed the fibers (Table 2). The non-preimpregnated UHMWP fiber reinforcement for group B resulted in a three-point flexural strength of 124 ± 7 MPa and a calculated three-point flexural modulus of 3.5 ± 0.3 GPa (Fig. 2a and b). In all of the test specimen fracture had passed the fibers (Table 2). The polymer preimpregnated glass fiber reinforcements (group D) had values of 197 ± 23 MPa and 5.0 ± 0.4 GPa, respectively, for the three-point flexural strength and modulus (Fig. 2a and b). The test specimens were intact in group D (Table 2).

The statistical analysis by two-way ANOVA showed that the brand and the location of the fiber reinforcements significantly influenced the flexural strength ($p < 0.0001$). Utilizing a two-way ANOVA at a 95% significance level the mean three-point flexure strength values were identified to be significantly increased for groups B, C, D and E when the fibers were placed in tension compared when the fibers were placed in compression. The two-way ANOVA showed also that only brand of the fiber reinforcement, had significant effect on flexural modulus ($p < 0.0001$), the location of the fiber reinforcements did not influence the flexural modulus ($p < 0.722$).

Combustion analysis studies highlighted that the fiber content as a percentage by weight (wt %)of the group B (Ribbond) specimens was 3.2 wt% which corresponded to 3.8 vol.%, for group C (FibreKor) 2.3 wt% and 1.1 vol.%, for group D (Stick) 12 wt% and 5.8 vol.% and for the group E (everStick) test specimens 12 wt% and 5.9 vol.%.

Discussion

This study demonstrated the effect of fiber reinforcement on the static flexural properties of a denture base polymer. Fiber reinforcements placed on the tensile side of the test specimens under loading resulted in considerably higher flexural strength and flexural modulus values compared with specimens that contained the same quantity of fibers placed on the compression side. It is suggested that this is due to the higher tensile strength of the reinforcing fibers, which could more effectively be used if the location of the fibers was close to the highest tensile stress of the test specimen. As a result, in clinical situations the fiber reinforcements should also be placed near the position of highest tensile stress under functional loading of a denture. An example for removable complete denture in a maxillary jaw would be close to the oral surface of the denture and perpendicular to the midline.

Methodologically, one limitation of the present study is related to the testing of dry specimens only. Several studies have already shown the influence of water saturation on the flexural properties of reinforced denture base polymer and other dental FRCs [25,30,31]. Because this study was aimed at the differences between fiber reinforcements and their locations on the flexural properties, the results can be interpreted accordingly. It has also been reported previously that the interfacial adhesion of glass fibers to the polymer matrix through silane coupling agent can be deteriorated by water storage. The degree of deterioration was related to the glass fiber sizing and silanation. Therefore, comparison of different brands of the fiber reinforcement can be questioned to some extend in terms of clinical significance. However, it has also been shown that properly sized and silanized glass fibers in denture base polymer did not weaken in water for several years [30].

Another methodological matter is that the three-point flexural strength and modulus are used as comparative values to evaluate differences between groups. Eqs. (1) and (2) do not take into account the layered structure of the test specimens investigated. In a homogenous polymer matrix test

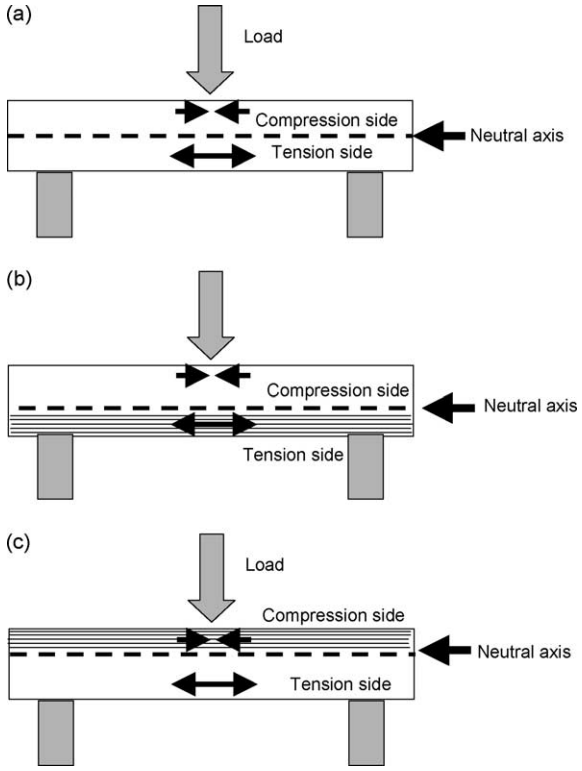

Figure 3 Schematic representation of highlighting the areas of tensile and compression side. If specimen is homogenous without any reinforcement, neutral axis located in the middle of test specimen (a), but if the fiber reinforcement is placed in tension side of the test specimen (b), or in the compression side (c), neutral axis is moved towards fiber reinforcement layer.

specimen, the neutral axis is located in the middle of the test specimen (Fig. 3a). However, the neutral axis is moved towards the fiber layer, when the test specimen consists of reinforcing fibers and a homogeneous polymer layer [33] (Fig. 3b and c). When the reinforcing fibers are positioned on the compressive side of the test specimen, the neutral axis is moved close to the fiber layer and higher tensile stresses are exposed to the homogeneous polymer, which causes fracture of the test specimen at lower loads (Fig. 3c). Depending on the ratio between the modulus of the fiber reinforcement and the homogeneous polymer layer, the neural axis location even inside the fiber layer, can cause the weaker polymer layer to be exposed to higher tensile stresses. This might be one explanation for the advantageous behavior of group D (Stick), where the fibers were more evenly distributed and filled the entire volume of the mold resulting in an almost homogenous structure.

Also the fact that the ratio of the thickness of the test specimen versus span length is relatively high should be taken into consideration. This has likely contributed more shear stress in the test specimen

than tested previously [31] according to ISO standard. However, the thickness of the removable denture base at the correct location of the fiber reinforcement corresponds to the thickness of the test specimens used in the present study.

The difference in the three-point flexural strength of the test specimens when the reinforcement was placed in tension compared with compression was marked in groups C (FibreKor) and E (everStick), the light-polymerized resin impregnated fiber reinforced specimens. This could be attributed to having a high quantity of fibers packed efficiently such that the one side of the specimen did not contain fiber reinforcements at all. Interestingly, group D (Stick) specimens showed less difference when the reinforcement was placed in tension or compression during testing. In this group, the polymer preimpregnated fiber reinforcement swelled during specimen fabrication and caused a relatively even distribution of fibers throughout the cross section of specimen resulting in effective crack stopping behavior. The relatively modest reinforcing effect of the UHMWP fibers (group B), irrespective of whether the fibers were placed in tension or compression suggested possibly that there was inadequate interfacial adhesion between the denture base polymer and the fibers. It was also possible that the impregnation of tightly bound UHMWP fibers by the highly viscous denture base resin was inadequate. This problem has been discussed previously by Vallittu [27,34]. It is proposed that the combination of inadequate interfacial adhesion and the inadequate impregnation may hinder stress transfer from the homogeneous polymer matrix to the fiber reinforcements, and therefore only minor difference between the test specimens with fiber locations could be found. Fiber reinforcement in group B (Ribbond) was not unidirectionally oriented as in the other fiber reinforcements using in the current study. Group B fiber reinforcement can be described as knotted as seen in Fig. 1. According to fiber geometries and Krenchel's factor, unidirectional reinforcement has the most efficient reinforcing factor than any other form of reinforcement [35], which can be one reason for the lower three-point flexure strength of group B.

It is of importance to note that the flexural modulus of the test specimen was attributed by the brand of the reinforcement but not by the location of reinforcement. This can be explained by the fact that the stiffness of the fiber reinforcement layer is dominant over the homogeneous polymer layer. As a result the three-point flexural modulus value recorded is indicative of the fiber reinforcement layer, because the stiffness of a fiber reinforcement is approximately 5–10 times higher than of PMMA [31]. This can be explained by the capability of the reinforcing fibers to withstand compressive stresses due to the higher compressive moduli of reinforcing fibers compared with the homogeneous polymer PMMA.

The visual examination of the test specimens was undertaken to differentiate the origin of the fracture and the failure mode. In the test specimens of group D (Stick) the test specimens were intact, whereas in group C (FibreKor) most of the test specimens were fractured into two pieces and in group B (Ribbond) the fracture passed all the fiber reinforcements. This could be explained by the relatively even distribution of fibers throughout the test specimens in group D. In clinical situations, a denture fracturing in two pieces is more drastic for a patient. A fracture line that has arrested at the fibers still allows the denture to function. The combustion analysis showed that the fiber amount in group C was significantly lower than in other groups and this could have been one reason why most of the test specimens were broken into two pieces.

Clinically the use of a fiber reinforcement that swells during manufacture and enables a more even distribution of the fibers could provide a more effective and less technique sensitive method to reinforce denture bases than using a high quantity of densely packed fibers that may have been placed incorrectly. Further research is needed to verify the present static test results with a dynamic loading test.

Conclusion

The results suggest that impregnated and preimpregnated fiber reinforcements reinforce denture base polymer more than non-impregnated fiber reinforcements. Fiber reinforcements placed on the tensile side resulted in considerably higher flexural strength and flexural modulus values compared with same quantity of fiber reinforcements placed on the compression side.

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