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Fibre reinforcement in heat-cured, microwave-cured and visible light-cured base resins

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The mechanical properties at transverse deflection, bending and impact tests were examined in three fibre reinforced resins including reinforcing materials such as inorganic glass fibre and cloth, organic Kevlar fibre and combined fibres. Both heat-cured and microwave-cured reinforced materials had almost the similar pattern to the changes in the mechanical properties, as compared with those in visible light-cured reinforced resin. Their reinforcing materials increased modulus and impact energy by about two times as compared with the bases. The visible light-cured resin was, however, not effective as a base resin when reinforced with fibres tested here, indicating that the transverse deflection and modulus values in light-cured reinforced resin were similar to that in the plain base resin.

1. Introduction

The reinforcement of poly(methylmethacrylate) has been approached by using graft copolymers for high impact resin [1], and reinforcing materials have been used for acrylic dough project [2–5]. Dentures should be made by a quick and simple technique without time consuming work. In general, carbon fibres, polyethylene fibres and polyaramid Kevlar fibre have been frequently tried to reinforce acrylic base resins as reported in [1, 2, 6, 7]. Their results indicate that the fibre reinforced resins have impact strengths and bending strengths equal to or greater than the plain acrylic resin. The other types of resins have hardly been reported, although the visible light-cured (LC) and microwave-cured (MC) resins have been used as base resins for reinforcement. The main applications in the dental field are as protheses [8] and removable orthodontic appliances [9]. MC and LC bases were, respectively, composed of an acrylic base resin and a matrix of urethane dimethacrylate plus microfine silica [8, 9]. This study was to investigate the usefulness of commercial denture base resins such as heat-curing (HC), MC and LC bases with the reinforcing materials. The fibre reinforced resins were examined with three types of tests; transverse deflection, bending and Charpy impact tests.

2. Materials and methods

The three types of base resins used were HC, MC and LC resin. The HC resin was the same as that reported in [3–5] (Natural resin, Nissin Co., Osaka). The resin was commercially available as ACRON MC (GC Dental Ind Corp. Co., Tokyo) and the visible LC resin as TRIAD (Dentsply Int, USA). These resins were reinforced with two kinds of fibres, namely, inorganic glass fibre and cloth (NITTOBO Co., Tokyo) and the organic Kevlar fibre (du Pont, USA). Their linear fibres were also incorporated as a reinforcing material as indicated in Table I. As a morphological feature lined fibres and cloth were used. The cloth which was frequently reported in [5] was a twill weave WLB, which gave superior bending properties to those in a plain acrylic resin. All reinforcing materials, which were coated with a silane coupling treatment, were set at the centre of a rectangular specimen which had the dimensions 10 mm × 65 mm × 3 mm. After curing and polishing, the specimens (10 mm × 65 mm × 2.5 mm) for bending and Charpy impact tests were obtained as reported in [4, 5]. The sizes of reinforcing materials were 55 mm length for glass linear fibre, 9.5 mm × 55 mm for glass cloth WLB, and 55 mm length for Kevlar fibre. Each fibre used had weights of 0.12 g (glass fibre), 0.04 g (Kevlar fibre) and 0.16 g (combined fibres) when added to each base resin, and the glass cloth's weight was 0.12 g.

The curing for each denture base resin was done according to the manufacturer's instruction. In particular, the MC resin was cured using the specially made FRP flask (HK type, GC Dental Ind. Corp., Tokyo), and the visible LC resin was done using the visible-light curing unit. The preparation of specimens was carried out by making the gypsum mould with a specimen dimension within each flask.

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TABLE I Maximum strength and impact energy in fibre reinforced resins and unfibred resin (base resin; HC resin, after, 30 days and 60 days, mean the resins after curing and immersing days in distilled water at 37 °C). Sample size = 5

Reinforcing materials	Morphology	Maximum strength (MPa) After	30 days	60 days	Impact energy (J) After	30 days	60 days
Glass fibre	Linear	118.6 ± 4.2	123.9 ± 7.1	101.1 ± 9.4	3.5 ± 0.6	1.5 ± 0.3	1.5 ± 0.2
	Cloth	112.9 ± 10.8	81.7 ± 4.0	93.7 ± 7.1	2.6 ± 0.3	1.2 ± 0.1	1.5 ± 0.3
Kevlar fibre	Linear	112.6 ± 6.5	109.0 ± 22.3	118.0 ± 9.6	4.1 ± 0.1	2.6 ± 0.6	2.4 ± 0.1
Glass and Kevlar fibre	Combined (Linear)	117.2 ± 7.2	110.2 ± 13.4	112.5 ± 13.9	2.4 ± 0.2	2.4 ± 0.1	1.8 ± 0.1
Cont (Unfibred)	–	94.9 ± 1.9	88.2 ± 9.0	86.2 ± 8.5	2.1 ± 0.1	0.6 ± 0.1	0.9 ± 0.1

Three kinds of tests were carried out in this study: Bending test (Autograph DCS-500, Shimadzu Co., Kyoto), Charpy impact test (Charpy impact machine, Japan Charpy Co., Tokyo) and transverse test (transverse bending tester, Seiki-shya, Tokyo). The former two were done as described in [4, 5], and the values from the latter were obtained from deflections at both 1.5 to 3.5 and 1.5 to 5.0 kg load increments as the standard method in [10]. Both maximum strength and impact energy were analysed under the following conditions: the values were measured, immediately after curing, after 30 days and after 60 days immersions in distilled water at 37 °C. The value of modulus at bending test was calculated by $WL^3/4bh^3d$, where W and d are the load and deflection at proportional limit, L the distance between the supports, b and h the width and thickness of the bending specimen [5, 10]. The stress and deformation curve at bending test was rewritten from the load and deflection curve, using bending strength (3 $WL/2bh^2$, under the applied load at each deflection). In the transverse test the modulus value was obtained from the amount of deflection at 3.5 to 5.0 kg load increment.

3. Results

The results of the transverse tests are shown in Figs 1a, b and 2. The transverse deflection values at 1.5 to 3.5 and 1.5 to 5.0 kg load increments, in a control plain specimen denoted as C for each base resin were larger than those in fibre reinforced specimens (G_L: glass fibre with a linear shape; G_C: glass cloth WLB; K: Kevlar fibre, and G_L + K; combined fibres of glass fibre and Kevlar fibre). At 1.5 to 3.5 kg load increment, the average values in three different types of base resins were about 1.6 mm, whereas those in the fibre reinforced specimens ranged from 0.9 to 1.4 mm. With increasing applied load the values of transverse deflection increased more than those at a load increment of 1.5 to 3.5 kg. At two load increments the values in LC reinforced resin became larger than those in HC and MC reinforced resins. In Fig. 2 (elastic modulus) the average values ranged from 3.2 to 6.1 GPa, showing that the HC reinforced resin had larger values than those in MC and LC reinforced ones. The base resins used had almost the same values ranging from 3.2 to 3.4 GPa. Typical stress and deformation curves are shown schematically in Fig. 3 (the curve obtained for the HC, MC and LC specimens after curing and preparation) and Fig. 4 (after curing, and immersion in distilled water for 30 and 60 days at 37 °C). The amounts of deformation at fracture of a bending test when changed to a dimension of length were above

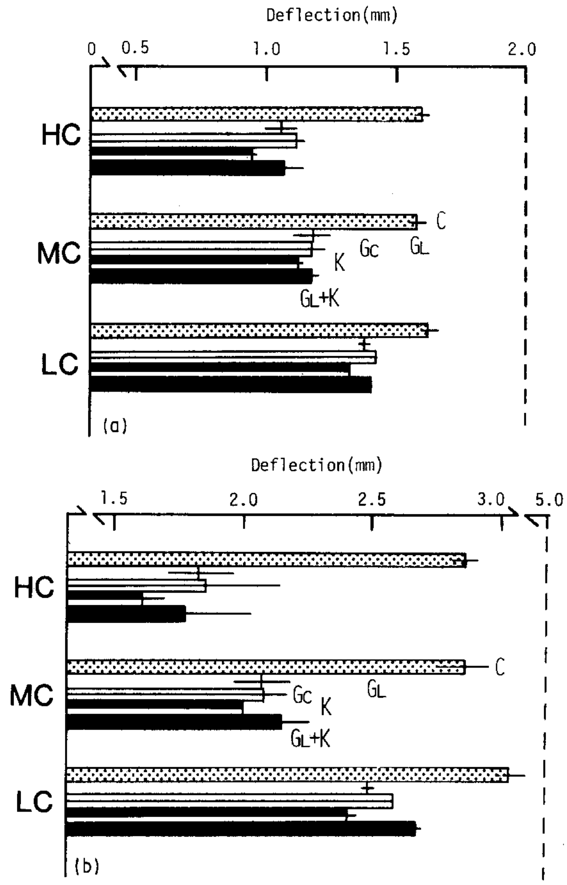

Figure 1 Deflection values in a transverse deflection test at each load increment for fibre reinforced resins: (a) increment of 1.5 to 3.5 kg load and (b) 1.5 to 5.0 kg load.

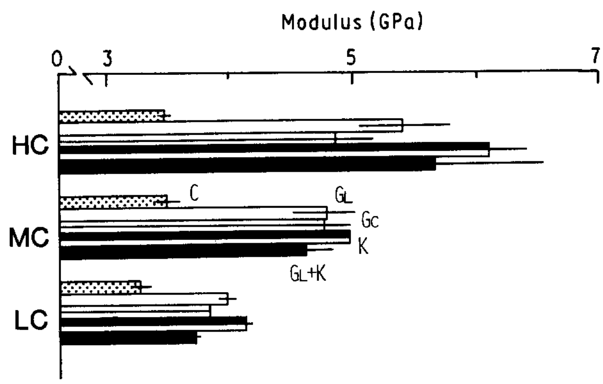

Figure 2 Elastic modulus from the transverse deflection test in HC, MC and LC fibre reinforced resins.

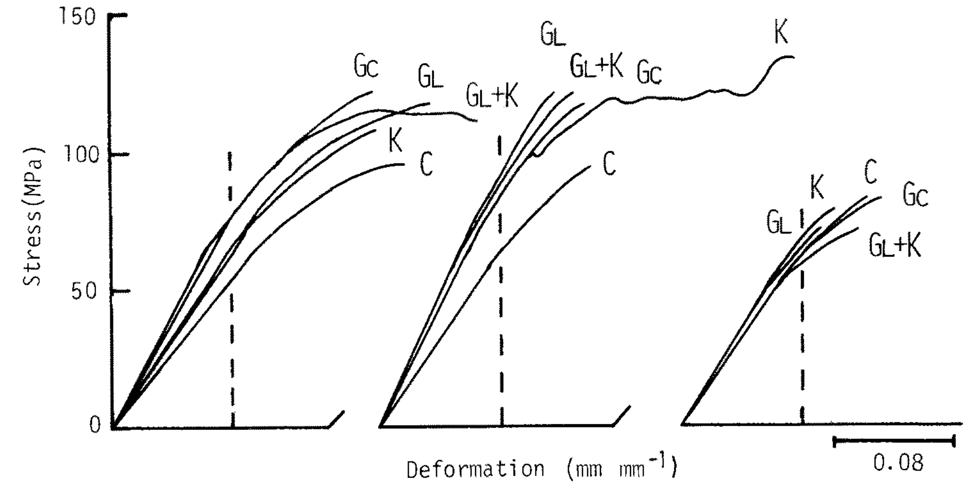

Figure 3 Graphic representation of stress and deformation at bending test in each fibre reinforced resin after curing (left: HC resin; middle: MC resin, and right: LC resin as a base)

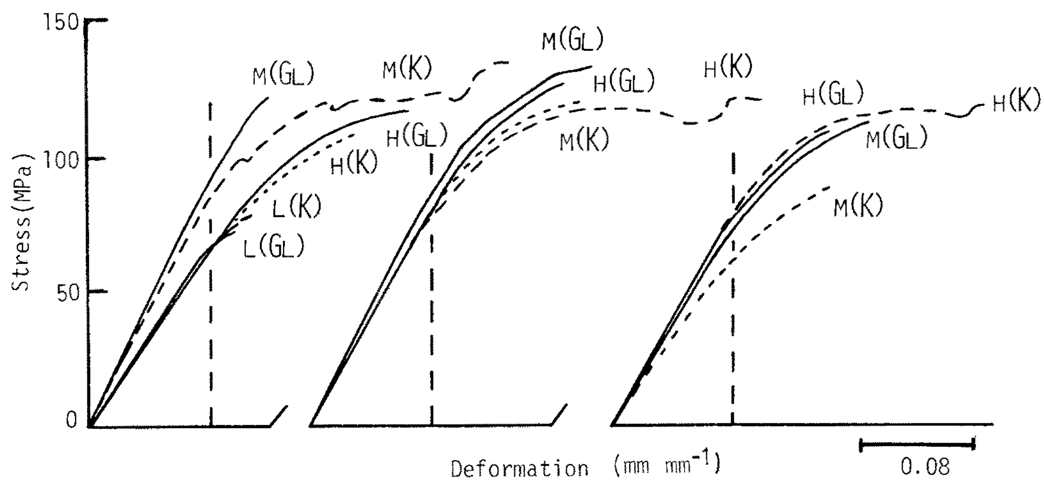

Figure 4 Graphic representation at bending test of glass fibre (linear)- and Kevlar fibre reinforced resins. Left: the resins after curing; middle: the resins after immersion for 30 days in distilled water at 37 °C; right: the resins after immersion for 60 days in distilled water at 37 °C. H, M and L mean HC, MC and LC fibred resins, respectively.

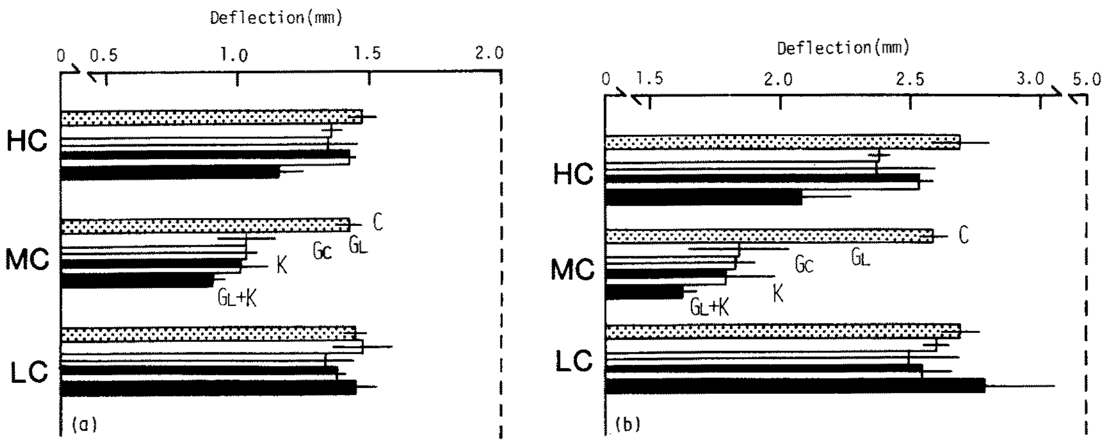

Figure 5 Amount of transverse deflection in HC, MC and LC fibred resins at: (a) 1.5 to 3.5 kg load increment and (b) 1.5 to 5.0 kg load increment.

2.0 mm (denoted as dotted line) for all specimens. Figs 5a, b, and 6 show, respectively, the deflection values at two load increments and elastic moduli for HC, LC and MC specimens. The transverse deflection in MC reinforced specimen was larger than those in HC and LC reinforced ones at two load increments, whereas the modulus value in MC fibre reinforced specimen was about 1.5 times those in the latter two specimens, indicating that the modulus value in latter was about 3.9 GPa. In Tables I, II and III the maximum strength determined as a bending strength and impact energy value are indicated, respectively, in HC, MC and LC

TABLE II Maximum strength and impact energy in fibre reinforced resins and unfibred resin (base resin; MC resin). Sample size = 5

Reinforcing materials	Morphology	Maximum strength (MPa)			Impact energy (J)		
		After	30 days	60 days	After	30 days	60 days
Glass fibre	Linear	111.0 ± 13.2	125.4 ± 11.4	110.9 ± 4.5	2.6 ± 0.6	3.5 ± 0.1	1.5 ± 0.2
	Cloth	120.7 ± 7.7	81.7 ± 2.9	96.0 ± 4.8	0.9 ± 0.1	3.2 ± 0.2	1.5 ± 0.3
Kevlar fibre	Linear	141.1 ± 20.1	116.0 ± 7.3	87.6 ± 5.9	2.6 ± 0.6	4.4 ± 0.6	2.4 ± 0.1
Glass and Kevlar fibre	Combined (Linear)	123.5 ± 9.4	118.0 ± 6.2	103.6 ± 13.3	3.0 ± 0.6	4.4 ± 0.1	1.8 ± 0.1
Cont(Unfibred)	–	96.0 ± 3.1	88.2 ± 4.8	82.7 ± 7.1	0.5 ± 0.1	2.7 ± 0.1	0.3 ± 0.1

TABLE III Maximum strength and impact energy in fibre reinforced resins and unfibred resin after curing (base resin; visible LC resin). Sample size = 5

Reinforcing materials	Morphology	Maximum strength (MPa)	Impact energy (J)
Glass fibre	Linear	71.3 ± 7.8	1.2 ± 0.1
	Cloth	88.2 ± 18.5	1.8 ± 0.1
Kevlar fibre	Linear	78.4 ± 4.8	2.1 ± 0.3
Glass and Kevlar fibre	Combined (Linear)	71.7 ± 9.2	1.2 ± 0.1
Cont(Unfibred)	–	84.2 ± 2.0	0.5 ± 0.1

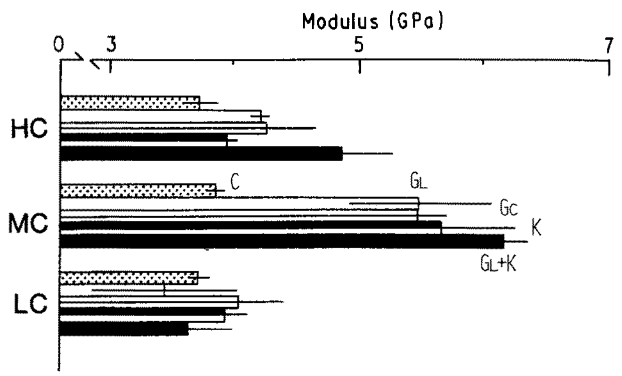

Figure 6 Elastic modulus obtained from the bending test of HC, MC and LC fibred resins after curing.

fibre reinforced specimens. As shown in Fig. 3 the LC fibre reinforced resin did not have increased values, and therefore immersion in distilled water was not done here. The changes of maximum strength and impact energy with the increase of immersion time in distilled water were not large. Especially, the increase in impact energy corresponded to that in bending deformation in Fig. 4. The effect of immersion time in distilled water on the deflection value at 1.5 to 3.5 kg load increment during a bending test was not found to be so large between HC and MC fibre reinforced resins. That is, the values at the 60 days immersion were almost the same as those of the 30 days immersion, showing that the average amount of deflection ranged from 1.1 to 1.5 mm as compared with the values (1.5 to 1.6 mm) in the plain base resins.

4. Discussion

The data from the static transverse deflection test indicate that the plain bases denoted as C had almost the same values, 1.6 (1.5 to 3.5 kg load increment) and 2.8 to 3.1 (1.5 to 5.0 kg), but that the fibre reinforced LC resins had the larger values than those in the reinforced HC and MC resins. The required values were below 2.0 (1.5 to 3.5 kg load increment) and 2.5 to 5.0 mm (1.5 to 5.0 kg) as specified by the Japan Industry Specification (JIS) for acrylic denture base resin in [11]. Almost the same requirements for the transverse deflection are given by other specifications for the same load increments [12]. The results in this study satisfied those increments. From these specifications, the latter resins had the rigid behaviour at a static transverse deflection test, and the values were within each required value of deflection. The result agreed with the tendency of increase in modulus as shown in Fig. 2.

Bending strength and deformation values for fibre reinforced LC resins had almost the same pattern as the control LC base resin (Fig. 3), so that the effect of reinforcing materials on their values was not so remarkable with LC resins. The LC base resin material has the same required values as the other resins when done according to ADA specification No. 12, described in [10], and both the values in transverse deflection and modulus are similar to those of HC base resin. As compared with HC and MC reinforced resins, a greater deflection LC resin was obtained (Figs 1, 2, and Table III). The difference of impact strength between a conventionally cured resin and MC resin was not significant, and some other properties have not been obtained.

In previous work, the base resin used was a HC resin, but similar patterns were observed between HC and MC fibre reinforced resins in comparison with those of LC resin. The results from bending test on the HC and MC fibred specimens which were immersed in distilled water for 30 and 60 days at 37 °C showed no significant differences between HC and MC fibred resins (Fig. 7), although significant difference between their resins was obtained for the deformation values after curing.

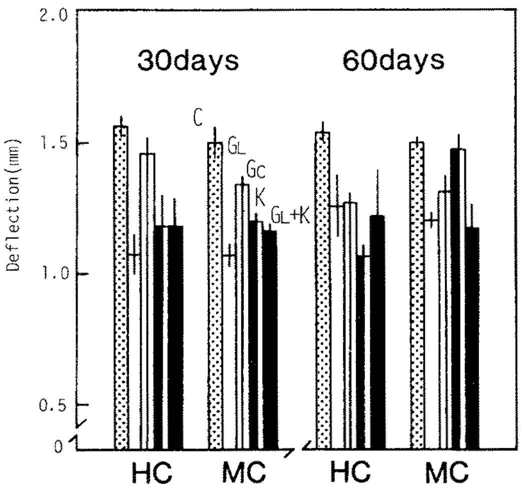

Figure 7 Transverse deflection values in HC and MC fibred resins after 30 and 60 days immersion test (1.5 to 3.5 kg load increment; distilled water, 37 °C).

The reinforcing materials were included in three types of base resins to clarify the differences of transverse deflection and bending properties and impact energy. The presence of the fibres in base resins had a role to prevent the propagation of cracks within the base resins as shown from the scanning electron microscopy about the interface between fibre and resin matrix [5], each reinforced resin indicating a rigid behaviour compared with those in control base resins. The increased values obtained by long term immersion in distilled water (30 and 60 days) had an importance, because the behaviour in MC fibred resin was similar to that in HC fibred resin. The MC resin matrix could be used as a base for the reinforcement with only a silane coupling treatment of fibre in dental prostheses. The reinforcement of the light-curing system was largely unsuccessful. Future work will be aimed at improving the LC resins.

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