

The influence of powder liquid ratio on the flexural strength of fibre reinforced acrylic resin material

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ABSTRACT

Introduction: Often the powder liquid (P/L) ratio of polymethyl methacrylate (PMMA) resins is changed to modify the handling properties of the material. While it is known that this may influence the mechanical properties of unreinforced PMMA resin, little is known about its effect on fibre reinforced resin.

Purpose: The purpose of this study was to determine how different P/L ratios influence the flexural strength (FS) of a glass fibre reinforced autopolymerizing PMMA resin used for fabricating fixed partial dentures.

Methods: Two main groups of PMMA resin, 1 unreinforced and 1 reinforced with glass fibre, had 3 subgroups (n=21) each representing a different P/L ratio. The manufacturer's recommended ratio served as control. The specimens were prepared for a 3-point bending test. Using a universal testing machine, maximum force was recorded and the FS was calculated. Median FS values were compared by means of non-parametric analysis of variance (Kruskal-Wallis). A p-value of less than 0.05 was considered significant.

Results: FS values of all reinforced subgroups were significantly higher than the values of the unreinforced subgroups ($p < 0.05$). Among the 3 unreinforced subgroups the difference in FS was insignificant ($p > 0.05$). Within the reinforced group there was a significant difference between the control group, which had a higher median FS value than the two other subgroups ($p < 0.05$).

Conclusion: When reinforcing PMMA resin with glass fibre, it is important to use the recommended P/L ratio. For unreinforced PMMA resin the P/L ratio can be changed within limits without adverse effects on the FS.

Key words: polymethyl methacrylate resin, glass fibre, powder liquid ratio.

INTRODUCTION

Autopolymerizing polymethyl methacrylate (PMMA) resins are often used for provisional fixed partial dentures (FPD). These restorations are subjected to masticatory load, especially in the case of long span FPDs, during long-term use or in patients with parafunctional habits. According to Hamza *et al.* (2004) a provi-

sional restoration should provide both pulpal and periodontal protection, have good marginal integrity, good aesthetics and sufficient durability to withstand forces of mastication.¹

Different techniques and materials for reinforcing PMMA resin have been suggested. Metal wires or mesh are popular methods of reinforcing. The disadvantages of using metal include the lack of adhesion between metal and resin, colour and bulk. The introduction of fibres addressed these problems. A chemical bond is established between fibre and PMMA matrix² that does not exist between metal and PMMA matrix.³ The modern fibres also have a neutral colour, are flexible and easily adapted to different shapes before polymerization. Different materials are used for fibres. The most popular materials in dentistry are the polyethylene and glass fibres. Both have been demonstrated to improve the physical properties of materials used for removable and FPDs.^{1,4} Fibres are available in unidirectional or multidirectional configurations. Woven fibres are thicker, and provide better strengthening characteristics because of their multidirectional configuration.⁵ The strength of the reinforced structure is also dependant on the volume of the fibres embedded in the PMMA matrix and the degree of adhesion between the fibre and the polymer. The higher the fibre content and the better the adhesion, the better are the strengthening characteristics.⁶

Clinicians often change the powder liquid (P/L) ratio of PMMA resins to modify handling properties. Williams *et al* (2001) found that this habit may have deleterious effects on the properties of the polymerized material.⁷ According to Syme *et al* (2001), it was found that while the stiffness of autopolymerizing resins was unaffected by variations in mixing ratio, extension to failure was greater with lower P/L ratios.⁸ These studies demonstrated that the mixing ratio has an effect on the strength of unreinforced polymerized material. However, little is known about its effect on glass fibre reinforced PMMA resin.

The purpose of this study was to analyze the effect of different P/L ratios on the flexural strength (FS) of reinforced PMMA resin compared to unreinforced PMMA resin.

The hypothesis to be tested was that a lower or a higher than the recommended P/L ratio decreases the FS of glass fibre reinforced PMMA resin used for provisional FPDs.

MATERIALS AND METHODS

Two groups of a PMMA resin (SR Ivocron®, Ivoclar Vivadent AG, Schaan, Liechtenstein) used for provisional FPDs were prepared: 1) group A consisted of 3 subgroups with different P/L ratios, 2) group B had the same P/L subgroups as group A but was reinforced with glass fibre (EverStick crown and bridge fibre®, StickTech, Turku, Finland). Each subgroup consisted of 21 specimens. One of the subgroups in each group used the manufacturer's recommended P/L ratio and acted as the control for the group. The manufacturer's recommended P/L ratio is 1/1 in volume and 1/0.83 in gram. A pilot study was conducted to confirm the handling properties of a 50 % higher and 50 % lower mixing ratio (1/0.498 and 1/1.245 by weight respectively).

Each specimen was prepared for the 3-point bending test. A custom-made stainless steel template was fabricated for this purpose. The rectangular specimens were 3mm wide, 6mm high and 25mm long. (Figure 1) The PMMA resin was mixed using accurate ratios as determined by the pilot study. The polymer and monomer were weighed using an analytical laboratory balance (Denver Instrument Co., Göttingen, Germany) with an accuracy of 0.0001g. The monomer and polymer were weighed in a glass beaker using the tearing option on the scale, which subtracted the glass beaker's weight to get the correct weight of the material. A 2ml Pasteur pipette (Vacutest KIMA, Arzergrande, Italy) was used to ensure the correct amount of monomer fluid was added.

For group A, the monomer and polymer were mixed in a glass beaker. The template was slightly overfilled. The surface was covered with a plastic matrix strip (Odus Universal Strip, Odus Dental S.A., Vevey, Switzerland) and a thick glass plate. A pressure of 5 kg was applied during the initial first 8 minutes to squeeze out excess material and to minimize porosity. Then, the template was placed, without the weight, in a pressure pot (Palamat practice Kulzer, Homburg, Germany) following the manufacturer's requirements for the PMMA, at 2 bar for 15 min submerged in 40-50°C water. After polymerization, the template was disassembled and the specimen removed. After removal, the edges of the specimen were finished with carbide paper (Wetordry P1200 grit, 3M, St. Paul, USA) and the width and height of each specimen was recorded at three different points using a digital height gauge (Mitutoyo Corporation, Higashi-Hiroshima, Japan) with an accuracy of 0.01mm. The averages of these dimensions were used in the FS formula.

For each specimen in group B the glass fibre was prepared before mixing the monomer and polymer. The fibre was removed from its silicone casing. It was cut with a surgical blade to a length of 27 mm. The fibre was polymerized in a light-polymerizing unit (Megalight MINI, Radeburg, Germany) for 2 min. The template was partially filled with unpolymerized PMMA to the height of the lateral stops, with a depth of 2mm. The location of the lateral stops ensured that the fibre was positioned on the tensile side of the specimen. It has been shown that the positioning of the fibre in this part of the specimen or restoration has the most efficient reinforcement potential.^{10,11} The polymerized fibre was placed parallel to the long axis of the specimen into the unpolymerized PMMA resin. The ends of the fibre rested in the lateral stops of the template. More resin was added and the template was slightly overfilled. The rest of the procedure was the same as for group A.

After polymerization, all the specimens were examined for voids on the surface. Discarded specimens were replaced with

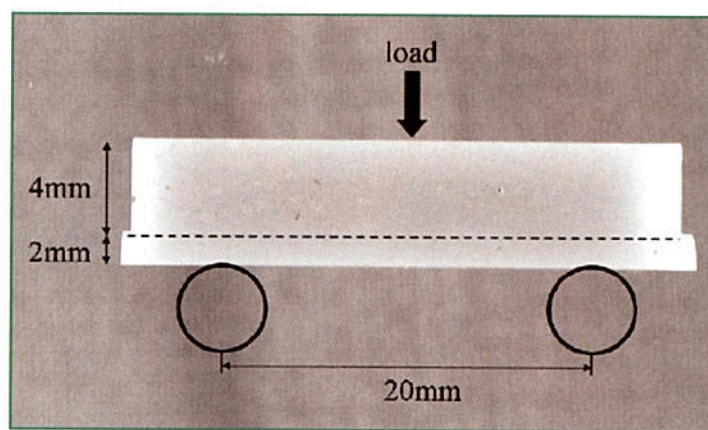


Figure 1: A specimen and its dimensions. The broken line indicates the position of the fibre.

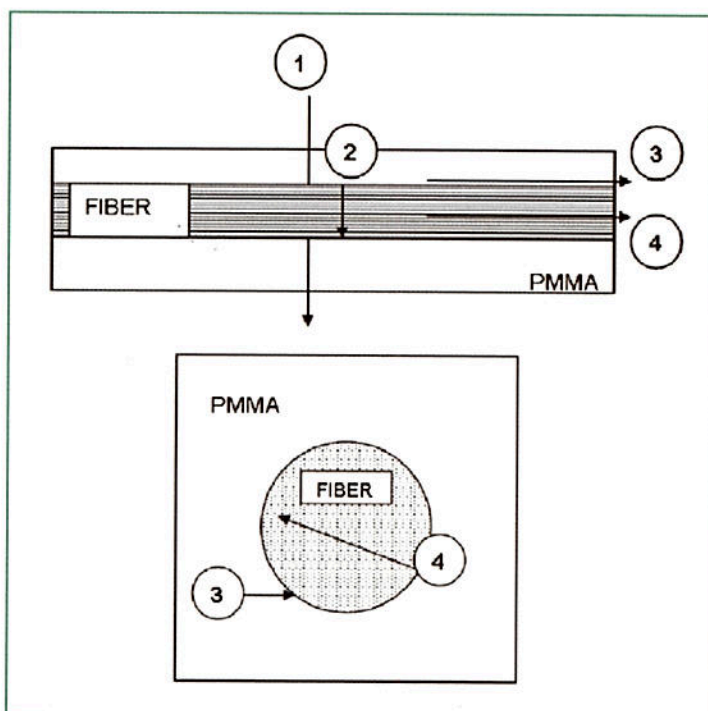


Figure 2: Diagram of the different fracture types: 1 = type 1 fracture through the PMMA matrix, 2 = type 2 fracture of the fibres, 3 = type 3 fracture between the PMMA matrix and the fibre bundle, 4 = type 4 fracture between the impregnation material and individual fibres.

new specimens. The specimens were stored dry before testing using a universal testing machine (Model 1446, Zwick, Ulm, Germany), connected to a computer equipped with the program TestXpert®. The specimens were positioned on the supports of the 3-point bending apparatus with a fixed span width of 20mm. A load was applied on the centre of the specimen at 90 degrees to the specimen axis through a stainless steel rod. By movement of the crosshead speed of 6mm/min, using a loading cell of 5kN, the load was increased and maximum force (F_{max}) in newton before specimen failure was recorded. The FS in MPa for each specimen was calculated using the following equation:

$$FS = 3F_{max} l / 2bh^2$$
 where F_{max} = maximum load before fracture in Newton, l = distance between supports in mm, b = width of specimen in mm and h = height of specimen in mm.

The means, medians and standard deviations for each group were determined. Multiple comparisons according to the Tukey-Kramer method were performed. The medians of the values were compared by means of non-parametric analysis of variance (Kruskal-Wallis). A p-value of less than 0.05 was considered significant.

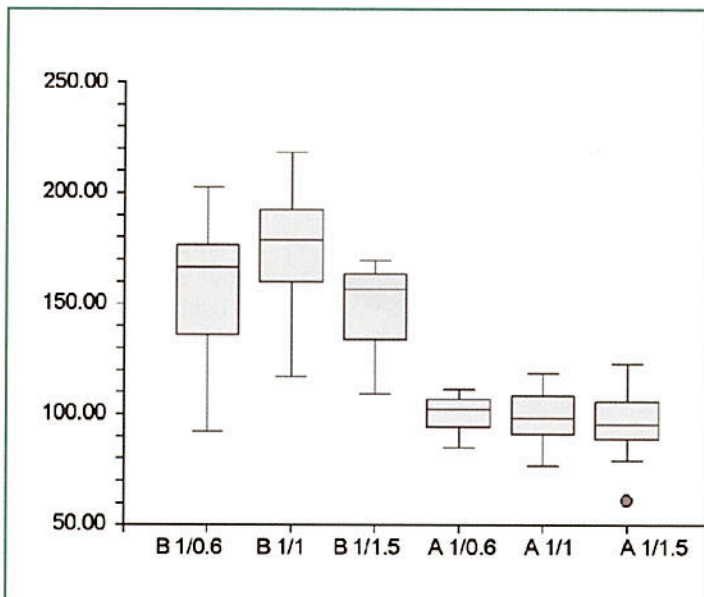


Figure 3: Side-by-side box-and-whisker plot

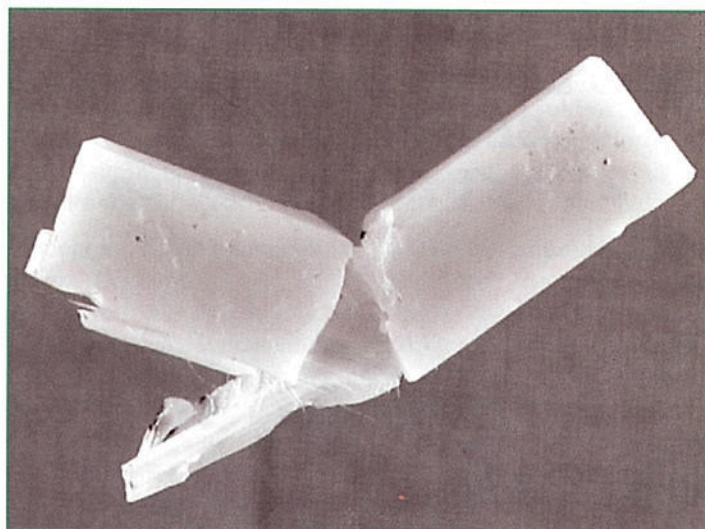


Figure 4: Specimen representing group B 1/1 showing a combination of fracture types 1, 3 and 4

The fracture patterns of the specimens were macroscopically analyzed according to the diagram in Figure 2. The fracture surfaces of representative specimens of each subgroup from group B were examined by means of a scanning electron microanalyzer (SEM) (model X-650, Hitachi, Tokyo, Japan).

RESULTS

One specimen from group A ratio 1/1 and one specimen from group B ratio 1/0.6 were not included in the analysis of the results, because of test failure: movement of the specimens on the supports was observed during loading before fracturing.

The descriptive statistics for the 3 subgroups in groups A and B are summarized in Table 1. The median FS for all the subgroups of group B (with fibres) was higher than the median FS of the subgroups of group A (without fibres). The strongest subgroup of group A, with a ratio of 1/0.6 and a median FS value of 102.7 MPa was lower than the weakest subgroup of group B, being ratio 1/1.5 with a median FS of 156.9 MPa. There was no overlapping of the medians between groups A and B.

Within group A, there was no significant difference between the median FS of the 3 subgroups ($p < 0.05$). For group B, there was no significant difference between the drier mixture of 1/0.6

Table 1: Descriptive statistics of the FS in MPa for the 3 subgroups of groups A and B

	Group A			Group B		
	1/0.6	1/1	1/1.5	1/0.6	1/1	1/1.5
Ratio	1/0.6	1/1	1/1.5	1/0.6	1/1	1/1.5
n	21	20	21	20	21	21
Mean	100.63	98.56	96.99	156.34	174.35	149.10
Median	102.7	98.8	96.1	166.9	179.0	156.9
SD	7.34	12.11	14.16	32.67	24.13	17.90
Min	84.96	76.92	61.34	92.29	116.88	109.13
Max	111.42	118.52	122.92	202.51	218.23	169.44

n = number of specimens per group, SD = standard deviation, min = minimum flexural strength, max = maximum flexural strength.

Table 2: Summary diagram of the Tukey-Kramer Multiple-Comparison test

	Group A			Group B		
	1/1.5	1/1	1/0.6	1/1.5	1/0.6	1/1
Ratio	1/1.5	1/1	1/0.6	1/1.5	1/0.6	1/1
Median	96.1	98.8	102.7	156.9	166.9	179.0

and the higher liquid content mixture of 1/1.5 ($p > 0.05$). However, there was a significant difference between the FS of these 2 ratios and the recommended ratio of 1/1 ($p < 0.05$). The multiple comparisons findings according to the Tukey-Kramer method can be summarised in Table 2. The groups linked by a black line do not differ statistically ($p > 0.05$).

In Figure 3, the box-and-whisker plot demonstrates the minimum, first quartile, median, third quartile and the maximum FS values. One outlier is present in group A 1/1.5. The value of this outlier is 61.34 MPa. It is clearly visible that the 3 subgroups in group B have a higher FS than the 3 subgroups in group A. The 3 mixes within group A have approximate symmetrical distributions. The distributions of the 3 subgroups in group B are less symmetrical than the 3 subgroups of group A.

Comparing the same ratios between groups A and B, the B group ratio always had the higher FS value. For ratio 1/1.5, the FS strength for the B group was 54% higher (A: 96.99; B: 149.10), for ratio 1/1 it was 77% higher (A: 98.56; B: 174.35) and for ratio 1/0.6 it was 55% higher (A: 100.63; B: 156.34).

All specimens from group A fractured according to the type 1 pattern. Specimens from subgroups B 1/1 and B 1/0.6 demonstrated type 1 in combination with predominantly type 3, but also type 4 fractures were observed (Figure 4). For subgroup B 1/1.5 the dominant fracture pattern combination was type 1 together with type 2 (Figure 5).

Figure 6 shows fragments of the pre-impregnation resin on the surface of the fibre bundle as well as tags of the pre-impregnation resin remaining in between the individual fibres. In between the tags and fibres are voids that indicate the loss of pre-impregnation resin. The surface of the fibre is smooth and shows no sign of damage in the process of the pre-impregnation resin being torn from the fibres. This type of fracture was a common occurrence for the specimens of subgroups B 1/0.6 and B 1/1. For subgroup B 1/1.5 it was observed that the fibres themselves fractured (Figure 7).

DISCUSSION

The hypothesis to be tested was that a lower or a higher than the recommended P/L ratio decreases the flexural strength of glass fibre reinforced PMMA resin used for provisional FPDs. This hypothesis can be accepted.

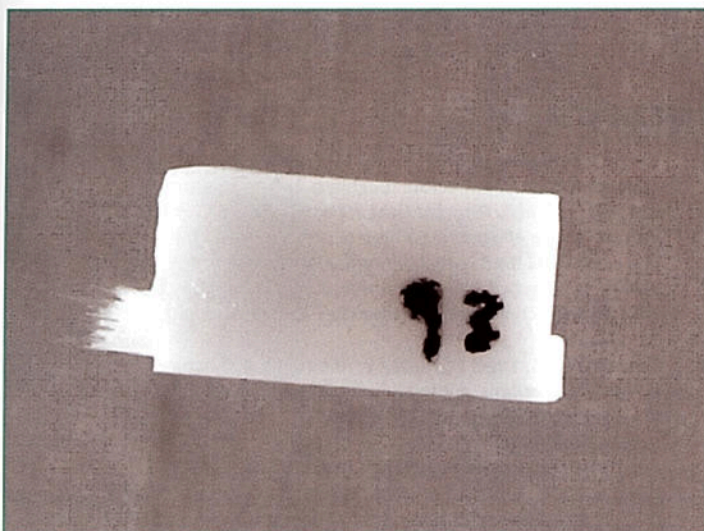


Figure 5: Specimen representing group B 1/1.5 with a combination of fracture types 1 and 2.

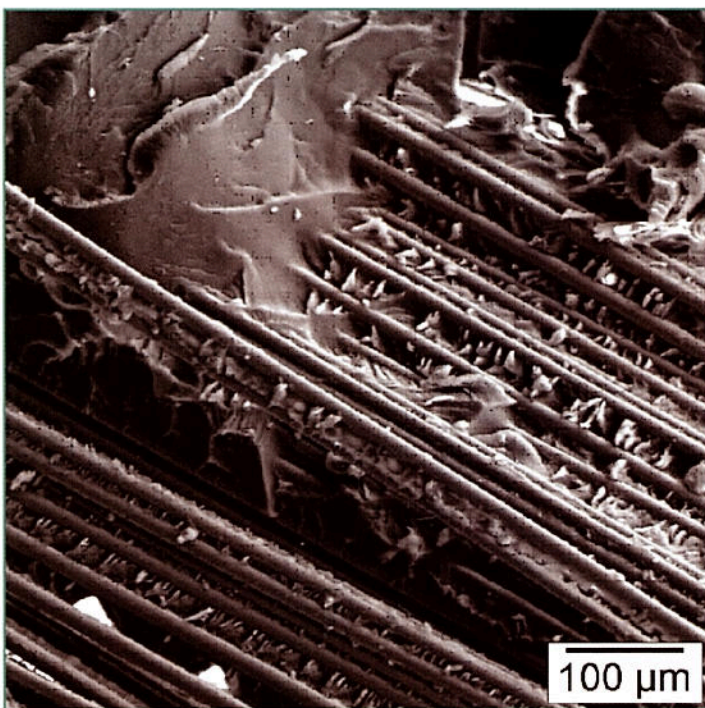


Figure 6: SEM image of the fracture surface of a specimen from subgroup B 1/1 (x169 magnification). The impregnation material is torn from the individual fibres (fracture type 4). Tags of the impregnation material remain in between the individual fibres.

PMMA resins are popular materials for fabricating provisional FPDs. Fibres can be incorporated in these resins in an effort to enhance the mechanical properties.¹ This statement is supported by the results of the present study: all the glass fibre reinforced groups had significantly higher FS than the unreinforced groups. These results indicate that the use of fibres is recommended if the FS of PMMA needs to be enhanced.

Practitioners often change the P/L ratio of PMMA resins to make handling easier for certain procedures or because of personal preferences. While it is known that this influences the mechanical properties of unreinforced resin materials,⁷ little is known about its effect on glass fibre reinforced PMMA resin. In this study the effect of 3 different mixing ratios on the FS of an unreinforced and a glass fibre reinforced PMMA was studied. One P/L ratio in each group was the manufacturer's recommended ratio. Although the median values of the 3 different subgroups within group A did not differ significantly, ratio 1/0.6 had the highest median value. This means that the stiffer mixture resulted in

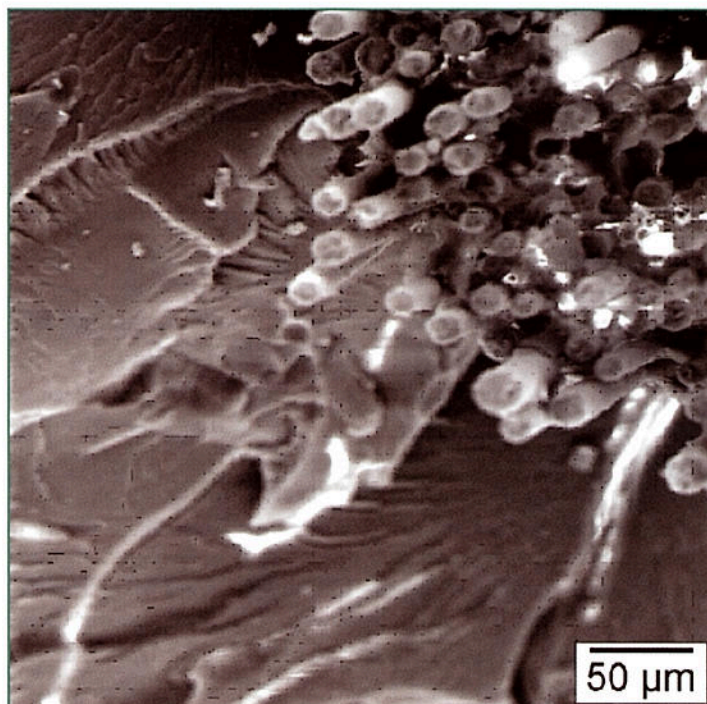


Figure 7: SEM image of the fracture surface of a specimen from subgroup B 1/1.5 (x 300 magnification). Parts of the fibre bundle fractured with fibres remaining adhered to the resin matrix (fracture type 2).

a slightly, but not significantly, stronger specimen. These results indicate that the P/L ratio does not have a significant effect on the FS of this unreinforced PMMA resin. Within the limits of the ratios used for this study, the consistency of a mixture can be modified by adding more powder or liquid to suit the clinical situation without detrimental effect to the FS of the unreinforced PMMA.

For group B, the scenario is different. The recommended ratio of 1/1 had the highest FS. There was a significant difference between this subgroup and the two other subgroups with ratios 1/1.5 (runny mix) and 1/0.6 (stiff mixture), with a lower FS. There was no significant difference between the ratios of 1/1.5, and 1/0.6. This means that it is important to follow the manufacturer's recommended ratio if glass fibre is incorporated into the mixture.

According to Vallittu (1994) a higher liquid content in the mixture increases polymerization shrinkage. Vallittu is of the opinion that this polymerization shrinkage might cause a slit between the fibre and the polymer matrix, reducing the amount of adhesion between the two components and reducing the strengthening effect.¹²

Syme and co-workers have shown that a mixture with higher liquid content produces a weaker specimen.⁸ However, higher powder content is not addressed in the literature. This study showed that a 50% higher than recommended powder content produced a higher FS, although not significantly, for unreinforced PMMA. With glass fibre reinforcement, a 50% higher powder content in the mixture produced a significantly lower FS compared to the recommended mixing ratio.

In vitro static load tests may not reflect the dynamic intra-oral conditions. Cyclic loading can be incorporated in the testing method to simulate the clinical environment. Microcracks and defects that grow inherently during thermal and mechanical processes can significantly reduce strength measurement.¹³ No cyclic loading in a moist environment was performed in the present study and should be considered a study limitation. The effect of different ratios on the strength of fibre reinforced specimens af-

ter cyclic loading could be investigated in the future. Because the physical properties of a resin are affected by changing the mixing ratios, this might also have an effect on the fatigue behaviour of the adhesion of the matrix to the fibres.

Significant differences in strength are reported among different brands of polymethyl methacrylate resin materials used for provisional FPDs.¹⁴ Therefore, comparing studies using different brands within one generic group of materials should be done with caution.

It is interesting to compare the different fracture patterns with the FS values for the fibre reinforced groups. The recommended ratio 1/1 and the stiffer ratio of 1/0.6 had the same dominant fracture pattern: the fibre bundle was torn from the specimen. This suggests that the weakest link in the reinforced structure was adhesion between the fibre bundle and the PMMA matrix. For the runnier ratio of 1/1.5 the fracture pattern was different: the fibres remained inside the PMMA matrix and broke off at the fracture interface. This suggests that the adhesion between fibres and PMMA is stronger than the fibre itself and a good transfer of forces from the PMMA to the fibres took place. However, the FS of this mixture compared to the FS of the other ratios was lower and significantly lower than the FS of the 1/1 ratio. This is difficult to explain in terms of the findings of Nohrstom *et al* (2000).¹⁰ They said that the transfer of stress takes place from the weaker polymer matrix to the fibre with a higher tensile strength. Therefore, the better the adhesion between the fibres and the matrix, the better the strengthening effect. It is also difficult to explain in terms of Vallittu (1999).⁵ He suggested that increased polymerization shrinkage might cause voids along the fibre bundle impacting negatively on the adhesion of the PMMA to the fibre bundle. These suggestions need to be investigated further.

A limited amount of studies have been performed on the significance of changing P/L ratios of fibre reinforced PMMA resins. The results of this study show that more information is needed to fully understand and explain the effect of different ratios on the adhesion between PMMA and the glass fibre bundle.

CONCLUSION

Within the limitations of this study, it was concluded that:

1. Reinforcement with glass fibre significantly increases the FS of a PMMA.
2. The use of different P/L ratios of unreinforced PMMA resin does not have a significant effect on the FS of the material.
3. The use of different P/L ratios of a glass fibre reinforced PMMA resin has a significant effect on the FS. A lower and a higher than the recommended P/L ratio decreases the FS.

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