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Effect of monomer composition of polymer matrix on flexural properties of glass fibre-reinforced orthodontic archwire

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SUMMARY To compare force levels obtained from glass fibre-reinforced composite (FRC) archwires. Specifically, FRC wires were compared with polymer matrices having different dimethacrylate monomer compositions. FRC material (E-glass provided by Stick Tech Ltd, Turku, Finland) with continuous unidirectional glass fibres and four different types of dimethacrylate monomer compositions for the resin matrix were tested. Cross-sectionally round FRC archwires fitting into the 0.3 mm slot of a bracket were divided into 16 groups with six specimens in each group. Glass fibres were impregnated by the manufacturer, and they were initially light-cured by hand light-curing unit or additionally post-cured in light-curing oven. The FRC archwire specimens were tested at 37°C according to a three-point bending test in dry and wet conditions using a span length of 10 mm and a crosshead speed of 1.0 mm/minute. The wires were loaded until final failure. The data were statistically analysed using analysis of variance (ANOVA). The dry FRC archwire specimens revealed higher load values than water stored ones, regardless of the polymer matrix. A majority of the FRC archwires showed higher load values after being post-cured. ANOVA revealed that the polymer matrix, curing method, and water storage had a significant effect ($P < 0.05$) on the flexural behaviour of the FRC archwire. Polymer matrix composition, curing method, and water storage affected the flexural properties and thus, force level and working range which could be obtained from the FRC archwire.

Introduction

Fibre-reinforced composites (FRCs) have been studied and developed for clinical use in dentistry and medicine for over 40 years (Smith, 1961). FRCs have been used in many dental applications: denture repairs (Vallittu and Sevelius, 2000), resin-bonded bridges to replace a missing tooth (Meiers and Freilich, 2006), resin-bonded retainers in orthodontics, periodontal splinting, and endodontic posts (Le Bell *et al.*, 2005).

Presently, the commonly used orthodontic wire alloys are stainless steel, cobalt–chromiun, betatitanium, and nickel–titanium (Kusy, 1997). Aesthetic and cosmetic values have become more important in dentistry in general and also in orthodontics. This has moved the development and use of orthodontic devices in to direction of tooth-coloured and transparent appliances. There have also been efforts to develop tooth-coloured orthodontic archwires. One alternative to this is to use coated metal wires.

Wires have been coated with tooth shaded teflon (polytetrafluoroethylene; Lim *et al.*, 1994), epoxy resin (Imai *et al.*, 1999b), and polyethylene (Neumann *et al.*, 2002). Also, FRCs have been started to be utilized in orthodontics (Jancar *et al.*, 1994; Kennedy *et al.*, 1998a,b; Imai *et al.*, 1998; Imai *et al.*, 1999b; Zufall and Kusy, 2000b). FRC archwires consist of unidirectional fibres embedded in polymer matrix. Their stiffness properties are in the same range as those of metallic archwires although the stiffness of FRC archwires can easily be changed by varying the polymer matrix composition and fibre reinforcement. Therefore, they can be used during the initial and intermediate stages of orthodontic treatment. There was a commercially available transparent composite wire (Optiflex®, Ormco company, Glendora, USA) which consisted of silicon dioxide (quartz) fibres, but the force level obtained from the wire, suggested it to be light for clinical use (Lim *et al.*, 1994). Recently, a new aesthetic composite orthodontic wire (BioMers®, Biomers Products, Naples, USA) has come into the market. There are no investigations available yet into this archwire. Glass-reinforced orthodontic archwires had not been clinically successful; however, further scientific work is needed to optimize a clinical use of FRC archwires.

The purpose of this study was to compare the force levels of different glass FRC archwires which were impregnated with four different dimethacrylate resin systems, providing different mechanical properties for the polymer matrix of the FRC archwire. The effects of polymerization process and water saturation were also investigated. The following null hypothesis was set up that FRC archwires force level can be optimize by changing polymer matrix of the FRC archwire, and water sorption may reduce the force levels.

Materials and methods

Sixteen groups of FRC archwires (continuous unidirectional E-glass, tex 300, Stick Tech Ltd, Turku, Finland) were tested with a three point bending test to determine the flexural properties of the wire and the level of force they can withstand for orthodontic use. Each group contained six FRC archwire specimens. Additionally, control group was used omega steel 0.016 inch wire (G&H®, Wire Company, USA). The cross-sectionally round FRC archwires were made from unidirectional, impregnated E-glass fibre measuring 0.3 mm in diameter, fitting into the conventional brackets slot.

Fibres were preimpregnated with four resin mixtures by the manufacturer to uncured form (Table 1). Group A1 was impregnated with a mixture, which consists of 50 per cent bisphenol-A-glycidyldimethacrylate (bis-GMA; Esschem ×950 0000) and 50 per cent dimer aciddiurethanedimethacrylate (DADD; Esschem ×725 000). Group A2 was impregnated with 30 per cent bis-GMA and 70 per cent DADD. Group B2 was impregnated with 50 per cent bis-GMA and 50 per cent ethoxylated bisphenol-A-dimetacrylate (bis-EMA; Esschem ×970 0000). Group B3 was impregnated with 30 per cent bis-GMA and 70 per cent bis-EMA. The mixtures consisted of 2-(dimethylamino) ethylmethacrylate (DMA) and camphorquinone as a photoinitiator system.

FRC archwires were initially light-cured in two different position with Optilux 501 (halogen-lamp, tip of diameter 11 mm, Kerr, Connecticut, USA; light intensity 550 mW/cm²) hand light-curing unit for 40 seconds per point or first post-cured 10 seconds with Optilux 501 and then post-cured with TargisPower (IVOCLAR, Shaan, Florida, USA) light-curing oven for 25 minutes. The temperature rose gradually to +95°C in the oven. The polymerized FRC archwire specimens were placed into the desiccator (+37°C) for a drying period. Dry FRC archwire specimens were tested to determine their flexural properties instantly after being taken out from the desiccator. The wet FRC wires, in turn, were tested after being stored in distilled water (20 ml) for 30 days at the temperature of +37°C.

Table 1 The monomer composition of the polymer matrices of the fibre-reinforced composite archwires according to the study groups. bis-GMA, bisphenol-A-glycidyldimethacrylate; DADD, dimer aciddiurethanedimethacrylate; bis-EMA; ethoksylatedbisphenol-A-dimetacrylate.

Group	Polymer matrix	
A1	BisGMA 50%, DADD 50%	E-glass fibre
A2	BisGMA 30%, DADD 70 %	E-glass fibre
B2	BisGMA 50%, BisEMA 50%	E-glass fibre
B3	BisGMA 30%, BisEMA 70%	E-glass fibre

The FRC archwires were tested according to a three-point bending test. Mesiodistal crown diameters of permanent first incisors by Morrees (1959) were in males group 8.78 (0.46) mm and in females group 8.48 (0.53) mm. Ranges were 7.0–10.00 mm (males) and 7.1.–9.8 mm (females). So the span length between the supports was chosen 10 mm and fibres were cut 15 mm length pieces. Dimensions of all specimens were measured before testing. The test speed was 1.0 mm/minute. The wires were tested until fracture (final failure). The preload of the device was 0.100 N. The test was done with a universal testing device (Lloyd LRX, Lloyd Instruments, Fareham, UK) and the results were entered into Nexygen-program for further calculations. The statistical analysis was carried out by using analysis of variance (ANOVA) and SPSS 16.0 (SPSS Inc, USA) factor analysis (using factors: polymer matrix, curing method, and water storage) followed with Tukey test ($P < 0.05$) *post-hoc*.

Results

The descriptive results for the maximum load values of the 16 groups are shown in Figure 1. Load values of FCR wire groups varied between 3.3 and 0.4 N, whereas control steel group was 11.4 (0.1) N. In dry conditions, all load values were higher than in wet conditions. The post-cured FRC archwire specimens had higher load values in both A1 and B2 groups and also in A2 water stored group. The hand light-cured specimens had higher load values in dry B3 and A2 groups. The load levels were lower with the hand light-cured specimens than with the post-cured specimens. Among all the FRC archwire groups, A1 oven post-cured group had the highest force level, the next one being B3 hand light-cured group, and the third one being A1 hand light-cured group. The deflection values at maximum load of the FRC archwires in the different groups are reported in Figure 2. ANOVA revealed that polymer matrix, curing method, and water storage had a significant ($P < 0.05$) effect on the force level and deflection values of FRC archwire. Examples of the load–deflection curves of groups A1 and B2 are given in Figure 3a and 3b.

Discussion

In orthodontics, the composition polymers and composites have varied considerably depending on their application areas (Jancar *et al.*, 1994; Lim *et al.*, 1994; Watari *et al.*, 1998; Imai *et al.*, 1999a; Zufall and Kusy, 2000a; Fallis and Kusy, 2000). There are some investigations in which different composite materials and FRCs have been compared (Watari *et al.*, 1998; Goldberg and Burstone, 1992; Goldberg *et al.*, 1994). Watari *et al.* (1998) compared two different

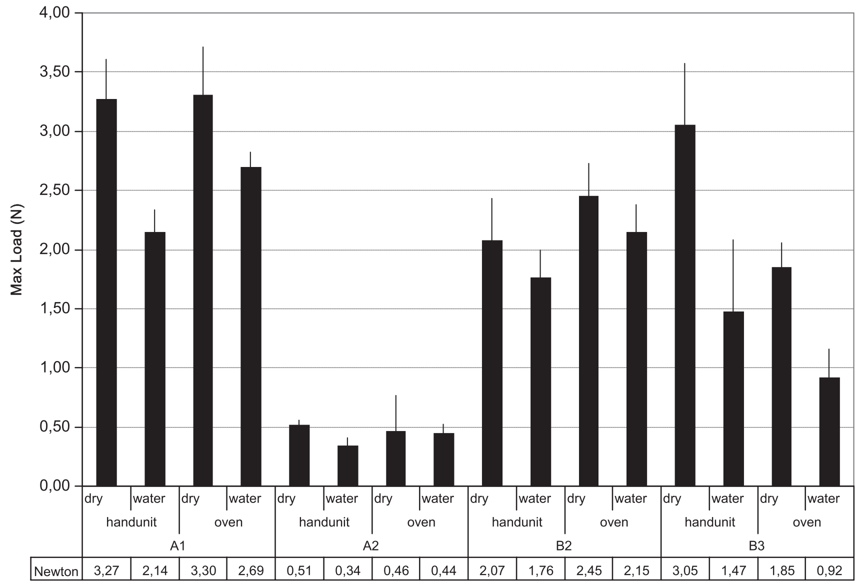

Figure 1 The maximum load values before failure of the fibre-reinforced composite (FRC) archwires with various monomer compositions (for group names, see Table 1) with standard deviations (vertical bars). ‘Handunit’ refers to polymerization by hand light-curing unit and ‘oven’ refers to post-curing by light and heat.

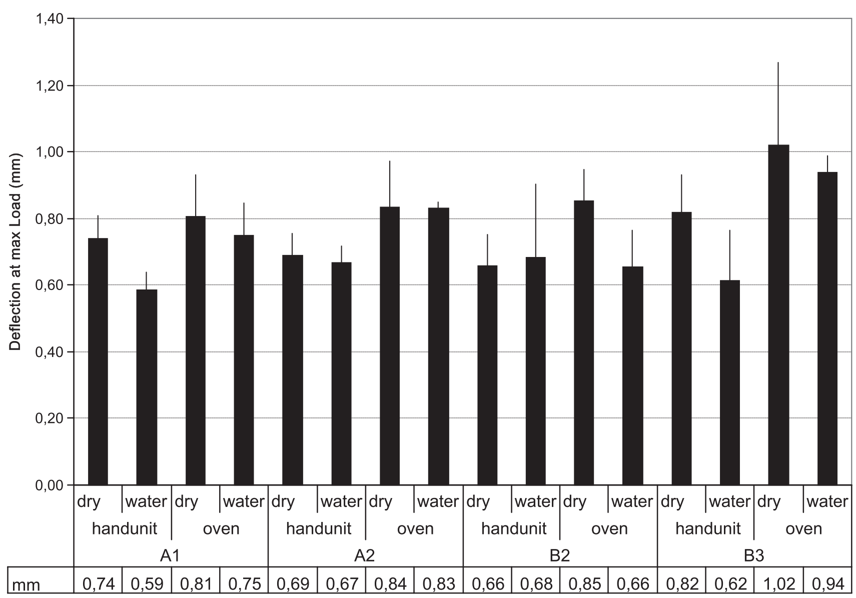

Figure 2 The deflection (millimetre) at maximum load (Newton) before failure of the fibre-reinforced composite (FRC) archwires with various monomer compositions (for group names, see Table 1) with standard deviations (vertical bars). ‘Handunit’ refers to polymerization by hand light-curing unit and ‘oven’ refers to post-curing by light and heat.

polymers, namely polymethylmethacrylate (PMMA) and epoxy resin by moulding and by hot drawing to fabricate FRC. The fibres were alumina ($Al_2 O_3$) or biocompatible glass made of CaO, P_2O_5, SiO_5, and Al_2O_3 (CPSA) glass fibres. The hot-drawn FRC archwires had better mechanical properties than those made by moulding process. The hot-drawn FRC archwires had enough flexural rigidity for orthodontic use. In terms of polymer matrix, the epoxy resin-based FRC archwires were able to produce higher load values than PMMA-based FRC archwires. However, if the emphasis is put on elastic properties in general, alumina/PMMA FRC archwire provided better elastic recovery properties than alumina/epoxy FRC archwire. PMMA is a non-cross linked polymer and has a lower modulus of

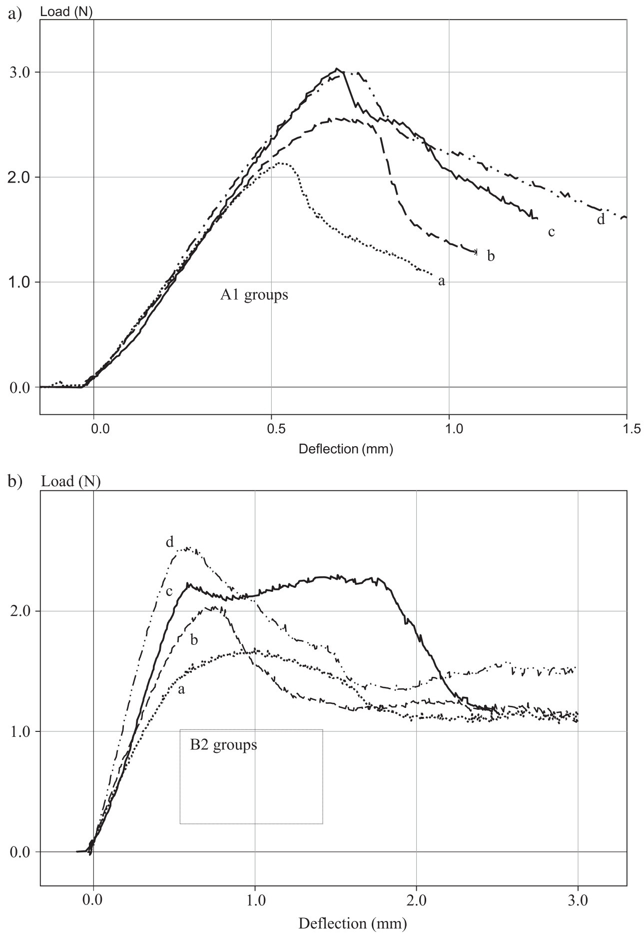

Figure 3 Examples of the load–deflection curves for (a) A1 groups from below to top; a, a dotted line/hand light-cured water group; b, a cutted line/oven post-cured water group, c, a straight line/oven post-cured dry group; and d, a segment dotted line/hand light-cured dry group. (b) B2 groups from bellow to top; a, a dotted line/hand light-cured water group; b, a cutted line/hand light-cured dry group; c, a straight line/oven post-cured water group; and d, a segment dotted line/oven post-curing dry group.

elasticity than a cross-linked epoxy polymer, which explained the better elastic recovery properties. This information supported the authors of the present study to add DADD monomer to the resin matrix because DADD monomer is able to plasticize the polymer matrix made on cross-linked dimetharylate monomers of bis-GMA and bis-EMA.

Goldberg and Burstone (1992) developed FRC wires which consisted of silane-treated S-2 glass-fibres and poly(ethylene terephtahlate glycol; PETG) or poly(1,4-cyclohexylene dimethylene terephthalate glycol; PCTG) polymer matrices. The FRC wires were made by using a pultrusion process. The mechanical properties of these two FRC wires were quite similar; the flexural modulus of PETG-FRC and PCTG-FRC was about 20 and 21 GPa, respectively. They also investigated E-glass, S-2 glass and Kevlar fibres, and PETG, polycarbonate (PC), nylon6, nylon 12, and 2 poly(sulfoxide-urethane; Pus) FRC archwires (Goldberg *et al.*, 1994). There was a significant difference between the resins as polymer matrix of FRC: PETG-FRC and PC-FRC showed the highest strength.

The present study utilized conventional bisGMA and bis-EMA monomers in various ratios in polymer matrix of the FRC archwires. An additional plasticizing DADD monomer was added to the polymer matrix. The monomers formed in a free radical polymerization cross-linked thermoset copolymer. Only minor differences in the maximal load values and stiffness values were found based on the additional DADD monomer. However, the plasticizing properties of DADD monomers may be greater in creep tests, where continuous static load is applied to the FRC archwire. Creep tests could better simulate the clinical condition of orthodontic treatment than conventional static loading tests.

It is known that after light-curing, only the monomer conversion of dimetharylate monomers remains at a relatively low level (55–65%). By post-curing, the polymer at increased temperature, up to the glass transition temperature of the polymer, the polymerization proceeds, and a higher degree of monomer conversions is reached. The degree of the monomer conversion has a relationship with the material's physical properties: a higher degree of conversion result in higher strength and stiffness of the material (Viljanen *et al.*, 2005). The monomer systems and polymerization conditions used in the present study confirm this by showing slightly higher maximal load values after post-curing except the specimens in group B3. That there were only minor differences between hand light-cured and post cured FRC archwires suggest a good initial polymerization of the monomers even by hand light-curing only. Technical problems in the fabrication of wires to the group B3 occurred: the FRC archwires were sticky and their handling was difficult, which may have caused variation in cross-sectional shape and diameter, resulting in misleadingly high maximal load values.

Water sorption to the polymer and polymer-based composites is known to reduce material properties by plasticizing the polymer matrix (reversible reduction) and by possible deterioration on the glass fibre surface, polymer matrix, or their interface (irreversible reduction) (Lassila *et al.*, 2002; Lastumäki *et al.*, 2001).

High quality E-glass with an appropriate sizing and silanation in combination to the polymer matrix retains its stability and mechanical properties even in longer term (up to 10 years) in simulated conditions (Vallittu *et al.* 1998; Vallittu, 2007). The present study showed a reduction of maximal load values and stiffness with all tested material combinations. The water storage period of 30 days was selected by knowing the mass increase behaviour by diffusion of water of the specimens slightly larger in size than those tested in this study (Lassila *et al.*, 2002). Some tendency for max-load values to reduce by water saturation was observed for the group A2 with a higher DADD monomer content. This suggested that the DADD may increase hydrophobic of polymer matrix.

Conclusions

- Different polymer matrix compositions and polymerization methods affect the force levels of experimental FRC orthodontic archwires. By changing the polymer composition, the force and stiffness levels of archwires are adjustable.
- Water sorption of the FRC archwires decreased force levels, and in general, post-curing at elevated temperature give higher force levels than those with hand light-cures only.
- In this study effect of different polymer matrix composition, curing methods and humidity on wire flexibility was evaluated; these issues need to be investigated more.

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