Flexural properties of crosslinked and oligomer-modified glass-fibre reinforced acrylic bone cement

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The flexural properties of oligomer-modified bone cement with various quantities of crosslinking monomer with or without glass fibre reinforcement were studied. The flexural strength and modulus of acrylic bone cement-based test specimens ($N=6$), including crosslinked and oligomer-modified structures with or without glass fibres, were measured in dry conditions and after immersion in simulated body fluid (SBF) for seven days (analysis with ANOVA). One test specimen from the acrylic bone cement group containing 30 wt % crosslinking monomer of its total monomer content was examined with scanning electron microscope (SEM) to evaluate signs of the semi-interpenetrating polymer network (semi-IPN). The highest dry mean flexural strength (130 MPa) was achieved with the bone cement/crosslinking monomer/glass fibre combination containing 5 wt % crosslinking monomer of its monomer content. The highest flexural modulus (11.5 GPa) was achieved with the bone cement/crosslinking monomer/glass fibre combination containing 30 wt % crosslinking monomer of its monomer content. SBF storage decreased the flexural properties of the test specimens, as did the addition of the oligomer filler. Nevertheless, the addition of crosslinking monomer and chopped glass fibres improves considerably the mechanical properties of oligomer-modified (i.e. porosity-producing filler containing) acrylic bone cement. In addition, some signs of the semi-IPN structure were observed by SEM examination.

Introduction

Currently available bone cements are mostly acrylic polymers made of poly(methylmethacrylate) (PMMA) powder and methylmethacrylate (MMA) liquid. Using a small quantity of crosslinking monomer with PMMA–MMA powder liquid systems, it is possible to produce a multiphase acrylic polymer structure, which contains crosslinked, partially crosslinked and linear phases. The structure is similar to that used in denture base polymers [1, 2]. More precisely, the multiphase structure is called a semi-interpenetrating polymer network (semi-IPN) structure (Fig. 1). The semi-IPN differs from a typical copolymer in that there are two independent polymer networks, the crosslinked and linear, that are not bonded chemically together to form a single network polymer [3].

One shortcoming that has been reported in the use of acrylic bone cements consists of the poor flexural properties [4, 5]. Many efforts have been made to improve the flexural properties of bone cement, for example, by changing the mixing method of commercial bone cements, or by adding the reinforcing fibres to the cement [6–13]. The fibre-reinforced composites (FRC) can be combinations of homopolymer, copolymer or polymer networks that consist of reinforcing fibre fillers. Especially in dental applications, the use of FRCs has emerged in recent years [14, 15]. It is well known that many factors affect the flexural properties of FRC, that is, the composition of fibres and polymer matrix, the orientation and quantity of fibres, the adhesion between fibres and polymer matrix, and the impregnation of the fibres by the resin matrix [14, 16–19].

We have previously shown that the addition of hydrophilic oligomer, polyamide of trans-4-hydroxy-L-proline, to acrylic bone cement creates porosity in the set bone cement in an aqueous environment [20]. However, it was also noticed that the mechanical strength of oligomer-modified bone cement has reduced after porosity formation, and that the weakening could only partially be compensated for by reinforcing the cement with chopped glass fibres [21]. The semi-IPN structure, together with fibre reinforcement, might further improve

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TABLE I The materials used in the study

Brand	Manufacturer	Lot no.	Type of material
EGDMA	Fluka Chemie GmbH, Buchs, Swizerland	421734/1 40302	Crosslinking monomer
Oligomer filler	Biomaterial Research, University of Turku, Turku, Finland	A221mp1	Oligomer
Palacos® R powder	Schering-Plough, Labo n.v. Heist-op-den-Berg, Belgium	8-BHAA-8/9033	Polymer
Palacos® liquid	Schering-Plough, Labo n.v. Heist-op-den-Berg, Belgium	8-RDCA-27/2969	Monomer
E-glass fibers	Stick Tech Ltd., Turku, Finland	1010321-R-0058	Fibres

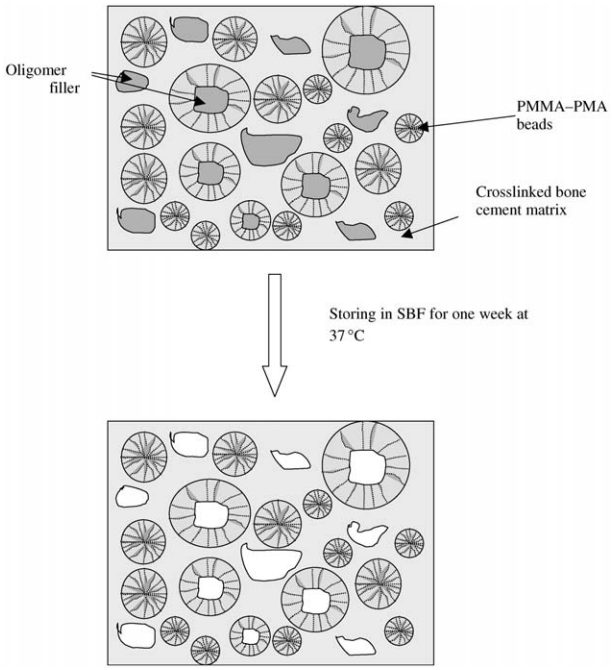

Figure 1 The schematic illustration of semi-IPN structure before and after immersion in SBF, the composite contains the oligomer filler.

the flexural properties of the acrylic bone cement. Therefore, we set out to study the mechanical properties of glass fibre containing porous acrylic bone cement reinforced by crosslinking monomers, that is, with a semi-IPN structure.

Materials and methods

Materials

Materials used in the study are listed in Table I. The commercial autopolymerising bone cement (Palacos® R) was used. Each packet contained 40 g of prepolymerised polymethylmethacrylate–polymethylacrylate (PMMA–PMA) copolymer powder and 18.8 g of MMA monomer liquid. Table II shows the detailed composition of the powder and liquid components as reported by the manufacturer.

Ethylene glycol dimethacrylate (EGDMA) was used as a crosslinking monomer. EGDMA (Fluka Chemie GmbH, Buchs, Swizerland) was the reagent grade (Lot: 421734/1 40302, purum: $\geq 97\%$ using gas chromatography) and was used as received without removing the inhibitor.

Commercial glass fibres (Stick Tech. Ltd., Turku, Finland) were used in this study. The reinforcing fibres consisted of continuous unidirectional silanised E-glass fibres, which had been preimpregnated with porous PMMA (Mw 220.000) [14]. The composition of the E-glass fibre is shown in Table III.

The oligomer filler was based on an amino acid of trans-4-hydroxy-L-proline, which can be polymerised to the corresponding polyester or polyamide [22]. In short, trans-4-hydroxy-L-proline was first converted into an ester by esterfication. The isolated and purified ester monomers were then subjected to melt-polycondensation at elevated temperatures *in vacuo*. The polyamide of trans-4-hydroxy-L-proline is a brittle and hydrophilic oligomer. The molecular weight of the oligomer varied, but the mean weight was ca. 5000. The brittle oligomer was crushed by hand in a mortar. The mean particle size of oligomer powders varied between 10 and 500 μm.

TABLE II Chemical composition of the commercial bone cement (Palacos® R) as reported by the manufacturer

Powder	40 g
Methyl methacrylate–methylacrylate copolymer	33.8 g
Benzoyl peroxide	0.2 g
Zirconium dioxide	6.0 g
Chlorophyll	0.001 g
Liquid	18.8 g
MMA (stabilized with ca. 60 ppm hydroquinone)	18.4 g
N,N-Dimethyl-*p*-toluidine	0.4 g
Chlorophyll-Cu-complex	0.0004 g

Test specimens

Four groups of test specimens were prepared (Table IV) of the acrylic bone cement which contained: (a) crosslinking monomer (Group 1), (b) crosslinking monomer and oligomer filler (Group 2), (c) crosslinking monomer and fibre reinforcement (Group 3), and (d) crosslinking monomer, fibre reinforcement and oligomer filler (Group 4). The crosslinking monomer replaced a weight fraction of the MMA monomer in Palacos® R cement (i.e. 5, 10, 20, and 30 wt %). The bone cement resin was polymerised by benzoylperoxide initiated and N,N-dimethyl-p-toluidine catalysed autopolymerisation at room temperature. Bar-shaped specimens (3.3 mm × 10 mm × 65 mm) were prepared for flexural testing under 15 min hydraulic press (Model Perkin Elmer IR Accessory Hydraulic Press, Germany).

The four test specimen groups were further divided into two subgroups, each containing six test specimens

TABLE III The composition of E-glass fibres (wt %)

Oxide	E-glass
SiO_2	54.5
CaO	22.9
Al_2O_3	14.2
Na_2O	0.1
MgO	0.7
K_2O	0.7
B_2O_3	6.3

TABLE IV The classification of bone cement composites in the study

Palacos R bone cement with	Abbr.	Dry (N) Subgroup1	SBF (N) Subgroup1	wt %[1]
(a) Crosslinking monomer	Group 1	6	6	0, 5, 10, 20, 30
(b) Crosslinking monomer and oligomer filler	Group 2	6	6	0, 5, 10, 20, 30
(c) Crosslinking monomer and fibre reinforcement	Group 3	6	6	0, 5, 10, 20, 30
(d) Crosslinking monomer, fibre reinforcement and oligomer filler	Group 4	6	6	0, 5, 10, 20, 30

[1]The amount of crosslinking monomer of its total monomer content.

$(=N)$. The test specimens in Subgroup 1 were tested dry at room temperature (23 °C). The test specimens in Subgroup 2 were individually immersed in 50 ml simulated body fluid (SBF) for one week at 37 °C, and tested in distilled water at 37 °C. The test specimens under SBF immersion were stored in a temperature-controlled water bath fitted with a vibrator (Model Grant OLS-200, England) for the immersion period of seven days before the flexural test. Kokubo's SBF was prepared by dissolving reagent chemicals of NaCl, $NaHCO_3$, KCl, $K_2HPO_4 \cdot 3H_2O$, $MgCl_2 \cdot 6H_2O$, $CaCl_2 \cdot 2H_2O$, and Na_2SO_4 in deionised and distilled water. The fluid was buffered at physiological pH 7.40, at 37 °C, with tris(hydroxymethyl)aminomethane (50 mM) and hydrochloric acid (HCl). The composition of SBF is shown in Table V [23].

In Group 1, the polymer powder (PMMA–PMA copolymer) was mixed with the monomer containing 0, 5, 10, 20, or 30 wt % of crosslinking monomer of its total monomer content. The specimens in Group 2 contained 20 wt % of oligomer-filler and 0, 5, 10, 20, or 30 wt % of crosslinking monomer, respectively. The oligomer filler was used to replace a weight fraction of the copolymer. In Group 3, the chopped $(l = 2\,\text{mm})$ continuous glass fibres were laid into the crosslinking monomer containing bone cement to completely fill the volume of the test specimens' mould. The samples in Group 4 were made with the same method except that the oligomer filler was first mixed with copolymer (PMMA–PMA) powder. The quantity of chopped glass fibres of the test specimens in Groups 3 and 4 was ca. 6.2 wt %. The quantity of glass fibres was determined by combustion analysis [14].

Scanning electron microscopy (SEM)

Linear polymer phases (i.e. not crosslinked PMMA–PMA beads) were dissolved from the surface of the polished specimen with tetrahydrofuran (THF) at room temperature for 15 min. The remaining crosslinking matrix in the region of the beads was expected to be a semi-IPN structure. The semi-IPN structure was examined from one of the test specimens of the Group 1 containing 30 wt % of crosslinking monomer of its total monomer content. The plain bone cement was also examined as a control. The polished control specimen surface was dissolved with THF at room temperature for 10 min. The surfaces of the specimens were evaluated with SEM (Model JSM 35 CF, JEOL, Japan) after treatment with THF. Before the evaluation, the surfaces of the specimens were coated with gold (thickness = 17 nm) using a sputter coater (Model BAL-TEC SCD 050 Sputter Coater, Liechtenstein).

TABLE V The ion concentrations of SBF, when the pH value was 7.4 at 37 °C

Ion	Concentration/mM
Na^+	142.0
K^+	5.0
Mg^{2+}	1.5
Ca^{2+}	2.5
Cl^-	147.8
HCO_3^-	4.2
HPO_4^{2-}	1.0
SO_4^{2-}	0.5

Flexural properties

The flexural properties of acrylic bone cement composites (i.e. with or without fibre reinforcement or oligomer filler) were measured by static test to establish the influence of crosslinking monomer quantities from 5 up to 30 wt %. The test was carried out using the three-point bending method according to the recommendation in ISO 1567 for determining flexural strength and modulus [24]. The crosshead speed of the material testing machine (model LRX, Lloyd Instruments, Fareham, United Kingdom) was 1.0 mm/min. The flexural strength and modulus were calculated using the NEXYGEN 2.0 software (model LRX, Lloyd Instruments, Fareham, UK). Flexural modulus (E) and strength (TS) were measured.

Statistical analysis

The statistical analysis was performed using SPSS (Statistical Package for Social Science, SPSS Inc., Chicago, IL) software for Windows. Mean values for the flexural properties were analysed with ANOVA, followed by Scheffes' *post hoc* analysis. The fixed factors were the quantity of crosslinking monomer, the type of filler (oligomer vs. fibres), and the environmental conditions. The dependent variables were the flexural strength and modulus of the different groups.

Results

Flexural properties

The flexural properties of the test specimens with fibre reinforcement were considerably higher compared to the same specimens without fibre reinforcement. ANOVA showed that all the fixed factors had a significant effect on both strength and modulus $(p < 0.0001)$, but there were also interactions of some degree.

In dry conditions, the flexural strength of acrylic bone cement composite containing crosslinking monomer

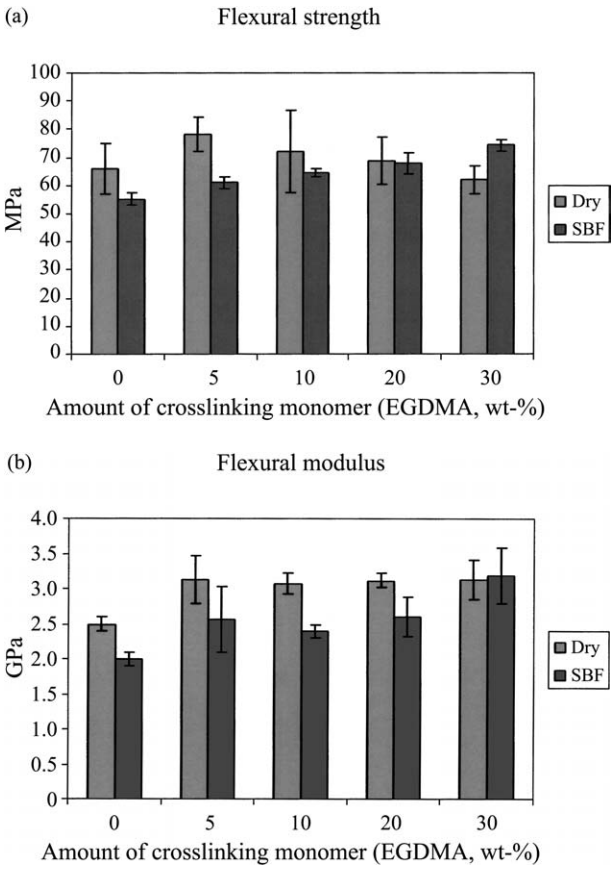

Figure 2 (a) The flexural strength of bone cement with various quantities of crosslinking monomer (Group 1), the test specimens were tested dry and after immersion in SBF solution for seven days. (b) The flexural modulus of bone cement with various quantities of crosslinking monomer (Group 1), the test specimens were tested dry and after immersion in SBF solution for seven days.

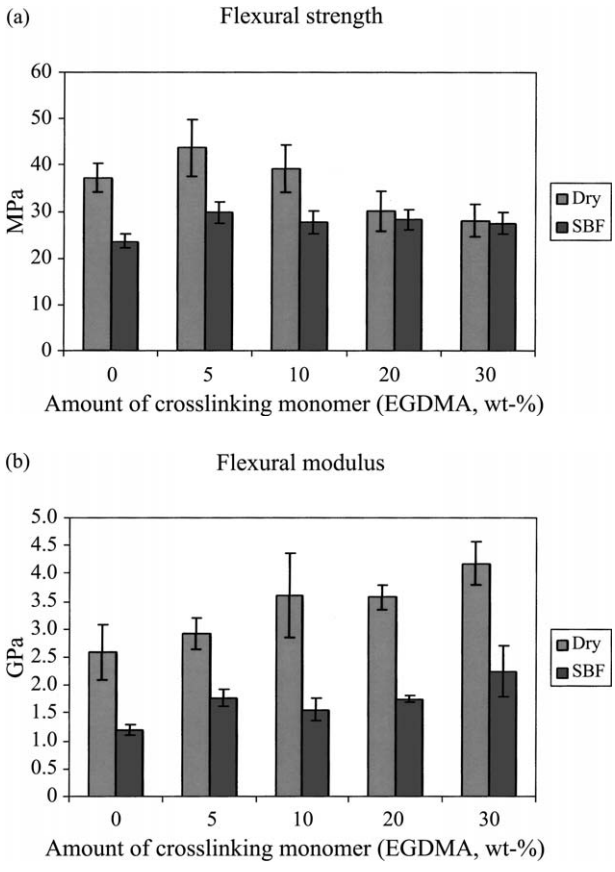

Figure 3 (a) The flexural strength of bone cement contained 20 wt % of Syncol-oligomer filler and various quantities of crosslinking monomer (Group 2); the test specimens were tested dry and after immersion in SBF solution for seven days. (b) The flexural modulus of bone cement contained 20 wt % of Syncol-oligomer filler and with various quantities of crosslinking monomer (Group 2); the test specimens were tested dry and after immersion in SBF solution for seven days.

(Group 1) varied from 62.1 to 78.4 MPa, while the flexural modulus was 3.1 GPa (Fig. 2(a) and (b)). The highest flexural strength (78.4 MPa) in this group was achieved when the composite contained 5 wt % crosslinking monomer (EGDMA) of its total monomer content. After one week immersion in SBF, the flexural strength varied from 61.2 to 74.4 MPa, and the modulus was between 2.4 and 3.2 GPa. In these conditions, the highest flexural strength (74.4 MPa) and modulus (3.2 GPa) were achieved when the composite contained 30 wt % of crosslinking monomer of its total monomer content. When the test specimens contained crosslinking monomer and 20 wt % of oligomer-filler (Group 2), the flexural properties were lower (Fig. 3(a) and (b)). In dry conditions, the flexural strength varied from 28.1 to 43.7 MPa, while the modulus varied between 2.9 and 4.2 GPa. After one week immersion in SBF, the highest flexural strength (29.8 MPa) in Group 2 was achieved when the composite contained 5 wt % of crosslinking monomer of its total monomer content. However, the highest flexural modulus (2.2 GPa) was achieved when the composite contained 30 wt % of crosslinking monomer of its total monomer content.

The chopped glass fibre reinforcement increased the flexural strength (Group 3): the highest flexural strength was 130.0 MPa when the test specimens contained 5 wt % crosslinking monomer of its total monomer content (Fig. 4(a)). After the one week immersion in the SBF, the flexural strength decreased to approximately 21% of its dry value. In dry conditions, the highest flexural modulus (11.5 GPa) was achieved when the test specimens contained 30 wt % of crosslinking monomer of its total monomer content (Fig. 4(b)). After one week's immersion in SBF, the variation in flexural modulus was not remarkable among the groups with glass fibre reinforcement and different quantities of crosslinking monomer. After one week's immersion in SBF, the highest flexural modulus (Group 3) was 8.4 GPa when the test specimens contained 20 wt % of crosslinking monomer of its total monomer content. Finally, when the test specimens contained crosslinking monomer, fibre reinforcement and oligomer filler (Group 4), the flexural strength varied from 59.3 to 76.5 MPa in dry conditions, and the modulus ranged between 6.7 and 7.6 GPa (Fig. 5(a) and (b)). After one week immersion in SBF, the flexural strength varied from 31.6 to 42.6 MPa and the modulus from 4.1 to 5.5 GPa.

SEM analysis

Surface topography of the specimen from Group 1 containing 30 wt % of crosslinking monomer of its total monomer content (Fig. 6(a)) shows different dissolve phases with the solvent of THF. However, the control group (Fig. 6(b)), the matrix had a greater tendency to be dissolved with the solvent of THF than the polymer beads.

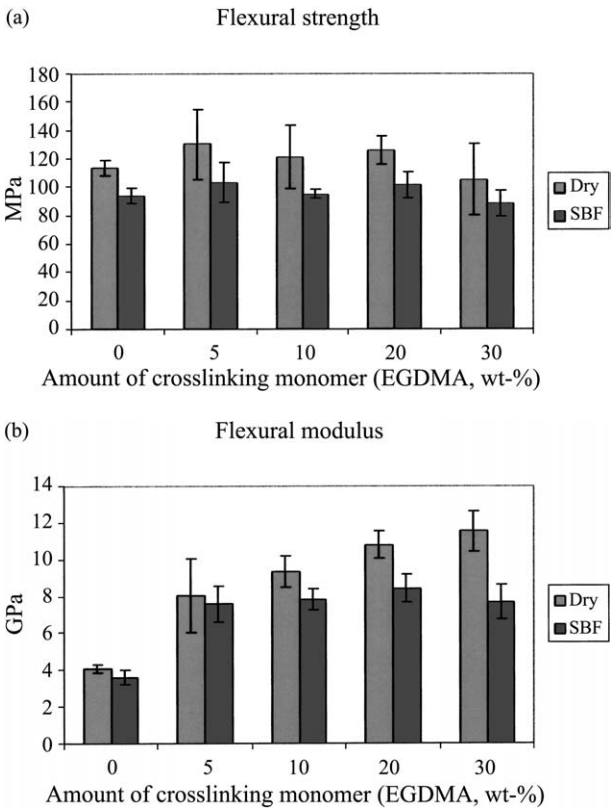

Figure 4 (a) The flexural strength of bone cement reinforced with chopped glass fibres and various quantities of crosslinking monomer (Group 3); the test specimens were tested dry and after immersion in SBF solution for seven days. (b) The flexural modulus of bone cement reinforced with chopped glass fibres and various quantities of crosslinking monomer (Group 3); the test specimens were tested dry and after immersion in SBF solution for seven days.

Discussion

A semi-IPN is defined as a network composed of two chemically independent polymers (i.e. crosslinked and linear ones). The semi-IPN structure differs from a typical polymer blend in that the properties are independently derived from each of the two polymers, and the phase separation occurs less frequently [25]. Frisch has reviewed the synthesis and properties of interpenetrating polymer networks [26]. The semi-IPN structures used in this study had a three-dimensionally crosslinked network with linear PMMA–PMA polymer

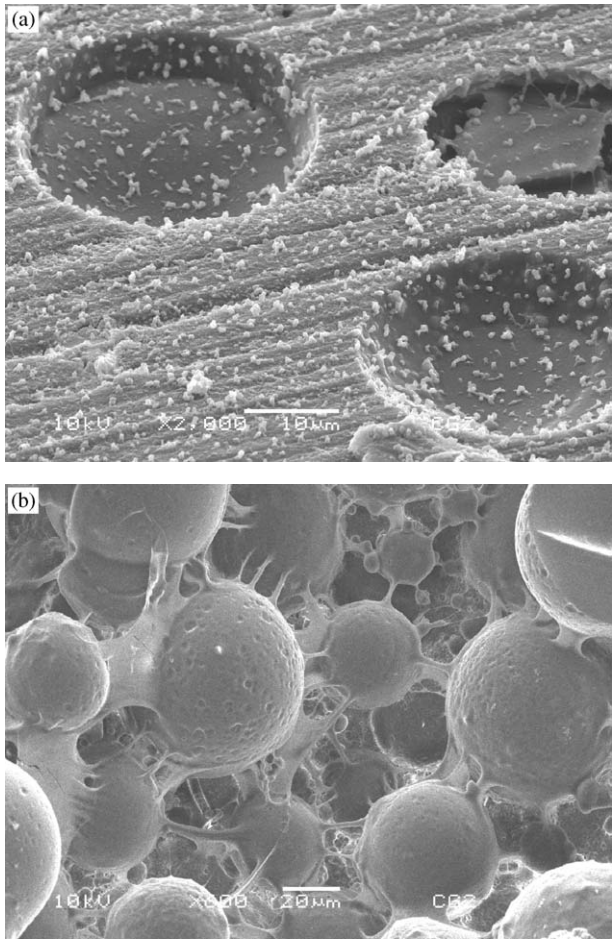

Figure 6 (a) SEM surface image illustrating, the semi-IPN structure was examined the test specimens from containing 30 wt % of crosslinking monomer of its total monomer content. Linear polymer phases (PMMA–PMA beads) were dissolved with THF. (b) SEM surface image illustrating, the surface of plain bone cement, the amorphous polymer matrix was dissolved with THF, whereas less THF soluble PMMA–PMA beads remained.

chains that were embedded in the composite with or without fibre reinforcement. In addition, in Groups 2 and 4, the hydrophilic oligomer-filler particles support the sites of porosity formation in the structure with or without fibre reinforcement.

The topography of a crosslinked specimen is shown (Fig. 6(a)), after removed of the linear PMMA–PMA

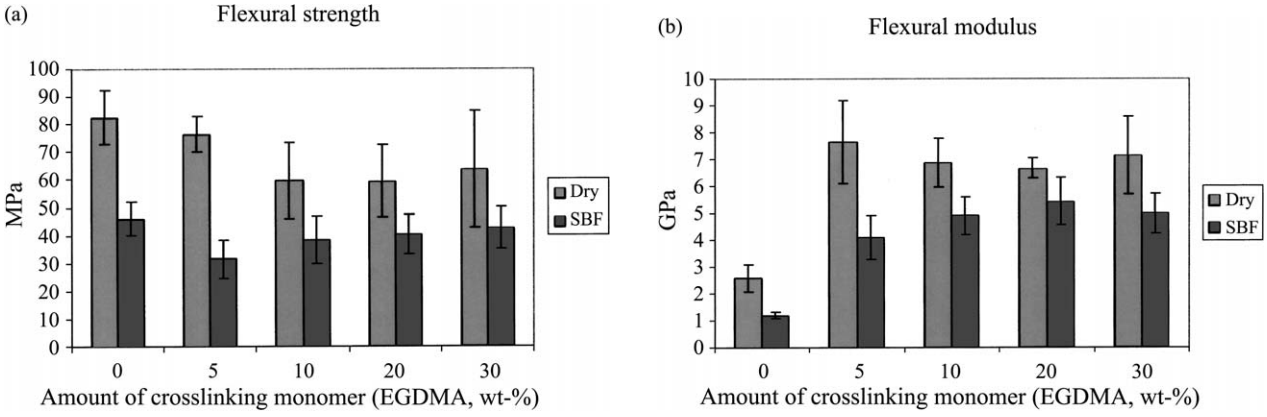

Figure 5 (a) The flexural strength of bone cement reinforced with chopped glass fibres; the test specimens contained 20 wt % of Syncol-oligomer filler and various quantities of crosslinking monomer (Group 4); they were tested dry and after immersion in SBF solution for seven days. (b) The flexural modulus of bone cement reinforced with chopped glass fibres; the test specimens contained 20 wt % of Syncol-oligomer filler and various quantities of crosslinking monomer (Group 4), they were tested dry and after immersion in SBF solution for seven days.

phases from its surface with THF. Fig. 6(b) shows the specimen of the control group, in which more amorphous polymer matrix has been dissolved with THF, whereas the corresponding PMMA–PMA beads with syndiotactic structure did not dissolve as rapidly. The surfaces between these two specimens show the formation of semi-IPN in the crosslinked structures.

Bone is a porous biological composite material, characterised by elastic, anisotropic, and heterogeneous structural properties [27]. The mechanical properties of ideal biomaterials for hard tissue applications would be as close as possible to the properties of bone. Therefore, the mechanical properties of biomaterials used in load-bearing applications are important, because these materials should be strong enough to withstand the physiological stresses of the body. At the same time, the synthetic biomaterials should encourage bone ingrowth to the structure. This study was a continuation of attempts to modify conventional dense bone cements to improve their structural properties towards those of living bone.

The purpose of acrylic bone cement is to anchor the prosthesis to the surrounding bone tissue. Traditional bone cements fill the space between the prosthesis and the bone in a purely mechanical manner. Theoretically, porous acrylic bone cements reinforced with fibres and a semi-IPN structure could enhance both the mechanical and biological connection between the bone and the prosthesis. Moreover, the porous structure facilitates bone ingrowth [28] and then strengthens the mechanical connection between these two different types of materials.

Our previous study showed that water diffuses through acrylic polymer, resulting in swelling and dissolution of oligomer filler and formation of interconnected porous structure. However, this reduced the mechanical properties of the cured bone cement [20]. The reduced mechanical properties in this kind of bone cement composites can be partially offset using fibre reinforcement, as has also been shown previously [21].

In this study, four groups of crosslinked polymer composites were used to evaluate their flexural properties. The crosslinked matrix increased the flexural modulus of the composite, even in porous structures, whereas the flexural strength did not increase considerably. Without fibre reinforcement and oligomer filler (Group 1), the dry flexural modulus was 3.1 GPa, whereas the flexural modulus was 2.5 GPa for unmodified acrylic bone cement [20]. However, by combining the fibre reinforcement with the addition of a crosslinking monomer (the semi-IPN structure), both the flexural strength and the modulus increased remarkably compared to the acrylic bone cement modified with oligomer filler. In Group 3, the mean dry flexural strength was approximately 1.7 times higher compared to the mean strength without fibres, whereas the mean modulus was 3.2 times higher. This could be due to better bonding of the crosslinked polymer matrix to the glass fibres compared to that of linear polymer alone. The glass fibres had been silane-treated to improve the adherence to the resin enhancing also mechanical properties.

This study showed that the combination of fibre reinforcement and crosslinked matrix significantly increases the flexural modulus of acrylic bone cement. From a practical perspective, the chopped fibres are quite easy to incorporate into the bone cement. Therefore, the combination of glass fibres and crosslinking monomer could be used in clinical practice. According to the Krenchel's factor, the chopped fibres have a homogeneously 20% isotropic reinforcing effect on the structure, which corresponds to the isotropic nature of bone [16].

Conclusion

The results of the study showed that the addition of increasing quantities of crosslinked monomer (up to 30%) increased the flexural modulus and strength of bone cement that had been modified with porosity-producing oligomer filler and glass fibres *in vitro*.

Acknowledgment

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