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Effect of iodixanol particle size on the mechanical properties of a PMMA based bone cement

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Abstract

Iodixanol is a water soluble opacifier widely used in radiographical examinations of blood vessels and neural tissue, and it has been suggested as a potential contrast media in acrylic bone cement. The effect of the iodixanol particle size on the polymerisation process of the bone cement, the molecular weight, and the quasi-static mechanical properties have been investigated in this paper. The investigation was performed using radiolucent Palacos powder mixed with 8 wt% of iodixanol with particle sizes ranging from 3 to 20 µm MMD, compared with commercial Palacos R (15 wt% ZrO_2) as control. Tensile, compressive and flexural tests showed that smaller particles (groups with 3, 4, and 5 µm particles) resulted in significantly lower mechanical properties than the larger particles (groups with 15, 16, and 20 µm particles). There was no difference in molecular weight between the groups. The thermographical investigation showed that the IDX cements exhibit substantially lower maximum temperatures than Palacos R, with the 4 µm IDX group having the lowest maximum temperature. The isothermal and the constant rate differential scanning calorimetry (DSC) did not show any difference in polymerisation heat (ΔH) or glass transition temperature (T_g) between radiolucent cement, or cement containing either IDX, or ZrO_2. Based on our findings the particle size for a bone cement containing iodixanol should be above 8 µm MMD.

Introduction

Acrylic bone cement is used in joint replacement by acting as a grouting agent and a stress distributor between the implant and the bone. The cement mantle is investigated regularly using X-rays post operatively to check for implant position and cement filling, and later for the presence of cracks and bone resorption around the cement mantle. However, acrylic bone cements based on poly-(methyl methacrylate) (PMMA) and related polymers produce a low X-ray attenuation. Contrast media are therefore added to the cement to increase the radiographic attenuation, thereby allowing visualisation of the cement especially in relation to bone. Current commercial bone cements contain either barium sulphate ($BaSO_4$) or zirconium dioxide (ZrO_2) as contrast media, and these are incorporated as hard particles, which are typically 20 µm in diameter. The use of these substances has some disadvantages, including reduced mechanical properties for the bulk material and, after the production of particles, third body wear [1-3], tissue inflammation [4] and increased osteclast activity and bone resorption [5,6].

Other types of contrast media have previously been investigated to find contrast media which are as effective at providing X-ray attenuation [7] without reducing the mechanical properties or producing abrasive or inflammatory particles. Iohexol and Iodixanol are non-ionic water-soluble contrast media that are used extensively throughout the world, as solutions that are injected into the body for radiographic imaging [8]. The powder-based opac-

ifier iodixanol (IDX) has previously been investigated in PMMA bone cement as an X-ray contrast media. The radiographic contrast study showed that equal concentrations, by weight, of IDX and ZrO_2 produce similar attenuation of X-rays under clinical X-ray measurement situations [7]. The *in vitro* and *in vivo* inflammation response was investigated [9] and it was found that the inflammatory response to cement containing IDX is lower than those for cements containing ZrO_2. The tissue response was more favourable for cement containing IDX than for cement with $BaSO_4$. The release of contrast media from bone cement has been investigated and the water uptake profile studied [10], limited opacifier release from the IDX cement was detected. The tensile properties of cement containing IDX have been previously reported [11] for a few particle sizes of contrast media, and it was hypothesized that the mechanical strength of cement with IDX varies depending on the particle size, and that the use of smaller contrast media particles reduces the tensile strength of the bone cement. It was argued that the smaller particles inhibit, to some extent, the polymerisation process, due to the large number of contrast media particles on each individual polymer bead; and that the tensile strength of the bone cement increased with particle size up to a threshold where the strength again goes down, due to stress concentrations.

This study investigates the quasi-static mechanical properties (tensile, compressive and flexural) of cement containing IDX contrast media of various particle sizes. In addition, in order to determine if the various sizes of the IDX particles interfere with the polymerisation process of the bone cement, a molecular weight analysis has been carried out on cured bone cement containing different sizes of particles. The polymerisation reaction was also studied by investigating the polymerisation heat (ΔH), glass transition temperature T_g and the polymerisation exotherm for one particle size in order to better understand the process of the polymerisation reaction.

Materials and methods

Materials

The IDX cement was supplied by Bicema AB (Karlstad, Sweden) as radiolucent Palacos R (Biomet Europe, Sjöbo, Sweden) cement mixed with 8 wt% (based on the powder content) IDX contrast media powder in the various particle sizes. Prior to mixing, the particle size distribution of all the IDX batches was measured by laser diffraction using a Malvern – Mastersizer 10 (Malvern, UK).

The material was prepared by mixing 40 g powder (including opacifier) with 20 mL of Palacos monomer. The cements were prechilled according to the manufacturer's guidelines, and were mixed under vacuum using the Optivac mixing system (Biomet Europe, Sjöbo, Sweden). The cements were injected into the appropriate moulds and allowed to polymerize under pressure. After curing, the specimens were removed from the mould and stored in saline solution at 37±1°C for a minimum of two weeks as a simulated ageing procedure.

Tensile investigation

Five different batches of IDX cements were prepared. The batches had Mass Median Diameters (MMDs) of 3, 5, 8, 15 and 20 µm. Also included are two batches from a previous study [11] where 8 wt% IDX was used in two particle sizes, 4 and 15 µm. The data from the 15 µm batch from the previous study has been combined with the 15 µm batch from this study. Palacos R, which contains 15 wt% ZrO_2, and radiolucent Palacos R (Palacos without ZrO_2) were used as controls. Three minutes after mixing, the cement was injected into moulds in order to produce half-size ISO 527 specimens. Samples were aged as described above, and prior to testing, the cross sectional area of the gauge section was measured. Testing was performed on an Instron 8511 load frame (High Wycombe, UK) with an MTS TestStar II controller (Minneapolis, USA). Strain was measured using an Instron 2620-602 extensometer (High Wycombe, UK), the specimens were loaded under displacement control at 2 mm min^{-1}. The fracture surfaces were inspected and those specimens that contained pores with a diameter of more than 1 mm were excluded. Means and standard deviations were calculated and one-way ANOVA was used for statistical analysis of the ultimate tensile stress (UTS), strain at failure and Young's modulus.

Compressive investigation

Cements with IDX of MMDs 3, 4, 8 and 16 µm were prepared. Again Palacos R and radiolucent Palacos were used as controls. Three minutes after mixing, the cements were injected into cylindrical compressive test moulds according to ISO 5833 (6 mm in diameter, 12 mm in height) and the samples were aged as described above. Testing was again performed on an Instron 8511 load frame with an MTS TestStar II controller, and the specimens were loaded under displacement control at 20 mm min^{-1} according to ISO 5833. Means and standard deviations were calculated with extreme points excluded, one-way ANOVA was used for statistical analysis of the compressive strength.

Flexural investigation by four-point bending

IDX cement with MMDs 4 µm, 8µm, 16 µm and 20 µm was prepared, and Palacos R was used as control. Three minutes after mixing, the cements were injected into rectangular test moulds in order to produce flexural specimens according to ISO 5833 (75 mm in length, 10 mm in width and 3.3 mm thick). The specimens were aged as described above. The four-point bending test followed ISO 5833 and was again performed on an Instron 8511 load frame with an MTS TestStar II controller, the specimens were loaded under displacement control at 5 mm min^{-1}. The deflections at the centre of the specimens were recorded at applied loads of 15 N and 50 N, in order to calculate the flexural modulus according to ISO 5833. The fracture surfaces were inspected and those specimens that contained pores with a diameter of more than 1 mm were excluded. Only two specimens were available in the 4µm MMD group due to lack of material. Means and standard deviations were calculated and one-way ANOVA was used for statistical analysis of the maximum flexural strength and flexural modulus.

Molecular weight analysis by GPC

Six samples, each weighing approximately 0.5 g, were used in the molecular weight analysis (carried out by Rapra Technologies Ltd., Shropshire, UK). The samples included four IDX cements; radiolucent Palacos cement mixed with 8 wt% IDX (per powder) in the particle sizes 4, 8, 16 and 20 µm MMD, Palacos R and also radiolucent Palacos cement. The procedure was done by dissolving approximately 20 mg of the specimen in approximately 10 ml of tetrahydrofuran. The solutions were left for four hours to dissolve and then warmed to 60 °C for 30 minutes. Each sample was thoroughly mixed and then filtered through a 0.2 micron polyamide membrane before transferred to sample vials, which were then placed in a near-ambient temperature Gel Permeation Chromatography (GPC) autosampler. The GPC system was calibrated using poly(methyl methacrylate) calibrants. The column used was 2 x mixed bed-B, 300 mm, 10 µm, and the flow rate was 1.0 mL min^{-1} (nominal) at 30 °C. Data capture and subsequent data handling was carried out using Viscotek 'Trisec 3.0' software.

Thermographical measurements

A total of seven batches of prepared cement were used: radiolucent Palacos, pre-chilled Palacos R, and Palacos cement with 8wt% of either 4, 8, 16 or 20 µm in MMD. Each cement was injected into a round Teflon mould according to ISO 5833, and a tight fitting plunger was pressed down (superfluous cement could escape through drilled holes) into the mould, until a fit was achieved, creating a disc of cement with a diameter of 60 mm and a height of 6 mm. The soldered tip of a thermocouple was placed in the centre of the disc thus measuring the temperatures in the middle of the specimen. The thermocouple was connected to the terminal block (TBX-68T, National Instruments Corporation, Austin, Texas, USA) and the data was logged to the computer via an PCI-card instrument NI PCI-4351 (National Instruments Corporation, Austin, Texas, USA). The temperature was monitored until steady-state was reached. Setting times were calculated according to ISO 5833, by estimating the time when the temperature is half the difference between the maximum temperature and the ambient temperature, as $\frac{1}{2}(T_{max} - T_{room})$.

Isothermal differential scanning calorimetry (DSC)

Each sample was prepared by mixing 5 mL of monomer with 10 g powder (Optivac, Biomet Europe, Sjöbo, Sweden). The powders were: radiolucent Palacos powder mixed with 8 wt% IDX with a MMD particle size of 6 µm, radiolucent Palacos and Palacos R. The mixed cement was immedi-

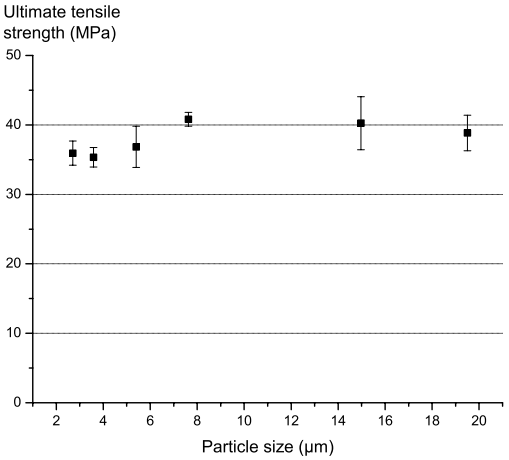

Figure 1. Ultimate tensile strengths of radiolucent cement containing 8 wt% iodixanol with the particle sizes 3, 4, 5, 8, 15 and 20 µm MMD.

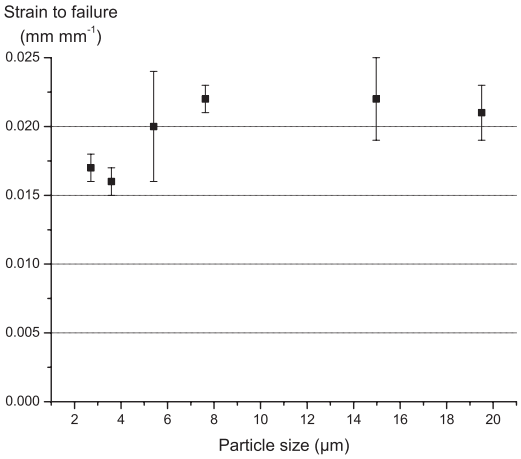

Figure 2. Strains to failure of radiolucent cement containing 8 wt% iodixanol with the particle sizes 3, 4, 5, 8, 15 and 20 µm MMD.

ately transferred into a 2.5 mL syringe which was then used to inject approximately 15–25 mg into a pre-weighed DSC pan. The pan was closed and rapidly transferred into the DSC (DSC Q1000, TA Instruments, New Castle, USA), time from start of mixing to start of the DSC run was approximately 3 minutes. The specimen was kept at 20 °C for a minimum of 40 minutes. After each run the filled pan was weighed in order to measure the sample weight. The data was analysed using Universal Analysis 2000 (TA Instruments, New Castle, USA) and the polymerisation heat (ΔH) calculated using the sigmoidal tangent setting.

Glass transition temperature by constant rate differential scanning calorimetry

Specimens were prepared by mixing 10 mL of monomer with 20 g powder. The powders were: radiolucent Palacos powder mixed with 8 wt% IDX with a MMD particle size of 6 µm, radiolucent Palacos, as well as Palacos R. The mixing was done in Optivac mixing systems, and the paste was then extracted into Optivac syringes, which was then placed in a 37 °C incubator for 18 hours prior to analysis. Approximately 10 mg of material, in pieces, cut from the centre of each rod was extracted and inserted into the DSC pans. The closed pan was weighed and then inserted into the DSC (DSC Q1000, TA Instruments, New Castle, USA), respectively. The analysis consisted of the cycles heating – cooling – heating; these were done at 10 °C min^{-1} between 10 °C and 150 °C following [12-14]. The data was analysed using Universal Analysis 2000 (TA Instruments, New Castle, USA), and the glass transition temperature T_g calculated by the software on the second heating cycle.

Results

Tensile investigation

The size of the contrast media particles has a significant effect, the 15 µm batch had an ultimate tensile stress significantly higher ($p<0.05$) than that of the 3 µm, 4 µm and 5 µm batches (figure 1). Likewise, the 8 µm batch shows (table 1) significantly higher ($p<0.02$) values in UTS and strain to failure than the 3 µm batch (figure 2). The 20 µm batch shows significantly higher ($p<0.03$) strain to failure than the 3µm and the 4 µm batches. The strain to failure of the 15 µm batch is significantly higher ($p<0.01$) than that of the 3 and 4 µm batches. The control groups had ultimate stresses of 43.4 ± 1.6 MPa (Palacos R) and 48.3 ± 3.9 MPa (radiolucent Palacos) and strains to failure of 2.5 ± 0.3 % (Palacos R) and 2.6 ± 0.4 % (radiolucent Palacos).

Compressive investigation

The compressive strength of the Iodixanol cement varies with the particle size of the contrast media

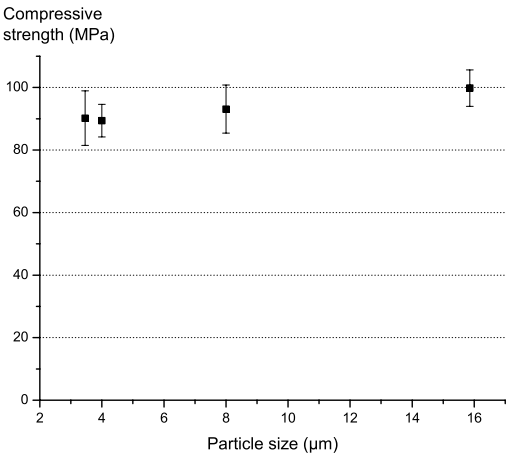

Figure 3. Compressive strengths of radiolucent cement containing 8 wt% iodixanol with the particle sizes 3, 4, 8, and 16 µm MMD.

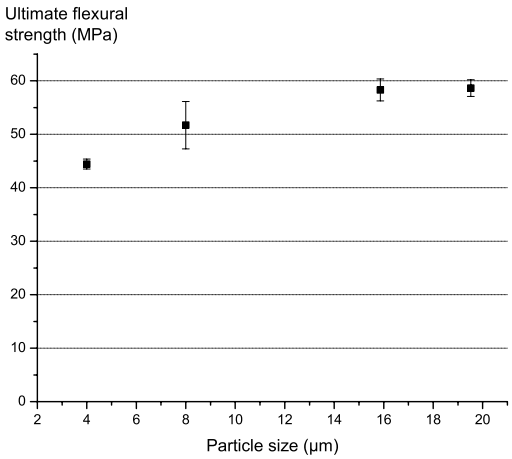

Figure 4. Ultimate flexural strengths of radiolucent cement containing 8 wt% iodixanol with the particle sizes 4, 8, 16 and 20 µm MMD. Only two specimen are included in the 4µm group due to lack of material.

Table 1. Tensile properties for cements containing IDX with particle sizes 3, 4, 5, 8, 15 and 20 µm, as well as for Palacos R and radiolucent Palacos

No. of specimen	Particle size / material (µm)	Ultimate tensile strength (MPa) mean	S.D.	Strain (%) mean	S.D.	Young's modulus (GPa) mean	S.D.
7	3	35.9	1.7	1.7	0.1	2.87	0.16
3	4	35.3	1.4	1.6	0.1	2.83	0.06
7	5	36.8	3.0	2.0	0.4	2.77	0.04
4	8	40.8	1.0	2.2	0.3	2.96	0.08
6	15	40.2	3.8	2.2	0.3	2.95	0.13
6	20	38.8	2.6	2.1	0.2	2.83	0.15
8	Palacos R	43,4	1,6	2.5	0.3	3.01	0.12
9	Radiolucent Palacos	48.3	3.9	2.6	0.4	2.93	0.10

Table 2. Compressive strength for cements containing IDX with particle sizes 3, 4, 8 and 16 µm, as well as for Palacos R and radiolucent Palacos.

No. of specimen	Particle size / material (µm)	Compressive strength (MPa) mean	S.D.
20	3	90.2	8.7
17	4	89.4	5.2
18	8	93.1	7.7
10	16	99.8	5.8
14	Palacos R	97.7	6.6
18	Radiolucent Palacos	101.4	3.1

(Figure 3), with the larger particles showing a significantly ($p<0.006$) higher compressive strength than the smaller particles (table 2).

Flexural investigation by four-point bending

The maximum flexural strength of the Iodixanol cement increases significantly ($p<0.02$) with the particle size of the contrast media (table 3); with the exception of the 16 µm and 20 µm cements which show similar flexural strengths (figure 4). The 16 µm and the 20 µm groups have similar flexural strengths as Palacos R (59.0 ± 1.6 MPa). There is no significant difference in modulus between the cements.

Table 3. Flexural properties for cements containing IDX with particle sizes 4, 8, 16 and 20 µm, as well as for Palacos R.

No. of specimen	Particle size / material (µm)	Flexural strength (MPa) mean	S.D.	Flexural modulus (GPa) mean	S.D.
2	4	44.4	0.9	5.31	0.14
5	8	51.7	4.4	5.33	0.22
5	16	58.3	2.1	5.34	0.18
4	20	58.6	1.6	5.50	0.15
4	Palacos R	59.0	3.4	5.54	0.14

Table 4. Molecular weight M_w and M_n and polydispersity for cements containing IDX with particle sizes 4, 8, 16 and 20 µm, as well as for radiolucent Palacos and Palacos R.

Material	M_w	M_n	Polydispersity
4 µm	722,000	154,000	4.7
	709,000	154,000	4.6
8 µm	690,000	145,000	4.8
	694,000	145,000	4.8
16 µm	717,000	143,000	5.0
	710,000	142,000	5.0
20 µm	719,000	140,000	5.1
	705,000	138,000	5.1
Radiolucent Palacos	702,000	150,000	4.7
	717,000	153,000	4.7
Palacos R	693,000	162,000	4.3
	700,000	161,000	4.3

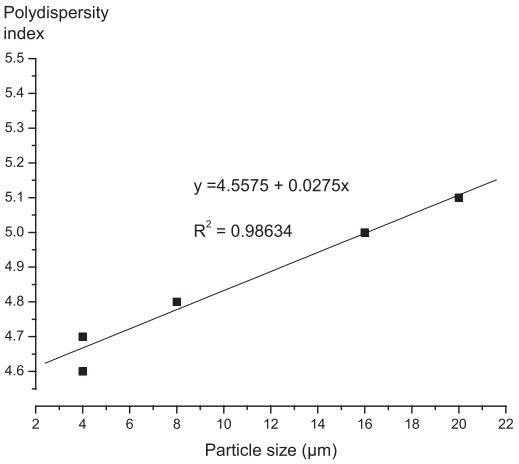

Figure 5. Polydispersity index from the molecular weight analysis for radiolucent cement containing 8 wt% iodixanol with the particle sizes 4, 8, 16 and 20 µm MMD. A linear fit is included.

Table 5. Setting times and maximum temperatures for cements containing IDX with particle sizes 4, 8, 16 and 20 µm, as well as for radiolucent Palacos and Palacos R.

Cements	Maximum temperature (°C)	Time to reach maximum temp. (min)	Setting time (min)
Palacos R	64.78	9:23	8:45
Radiolucent Palacos	54.16	8:14	7:37
IDX 4 µm (1-test only)	41.35	9:48	7:38
IDX 8 µm	53.72	10:52	10:05
IDX 16 µm	49.51	10:04	9:12
IDX 20 µm	49.47	8:49	7:56

Molecular weight analysis by GPC

The size of the particulate IDX does not seem to impact the mean molecular weights of the cement (table 4), neither by weight of molecules (*Mw*) nor by number of molecules (*Mn*). However, the polydispersity is directly related to the size of particles (figure 5), with the lowest polydispersity for the cement containing IDX with a particle size of 4 µm, and the highest polydispersity for the 20 µm IDX cement, in the IDX cement group. The difference in polydispersity is small but real. Polydispersity was low for both radiolucent Palacos and Palacos R.

Thermographical measurements

The results from the thermographical measurements are seen in table 5. The setting times are comparable with no substantial difference. The radiolucent Palacos showed a setting temperature of 54.2 °C, Palacos R showed an increased maximum temperature of 64.8 °C, and the IDX cements demonstrate a lower maximum temperature of 41–50 °C. However, within the IDX group there are large differences, the 4 µm group show a maximum temperature of only 41.4 and the other three groups (8, 16 and 20 µm) show a maximum temperature of 49–54 °C. Typical curves can be seen in figure 6.

Isothermal differential scanning calorimetry (DSC)

Curves for isothermal differential scanning calo-

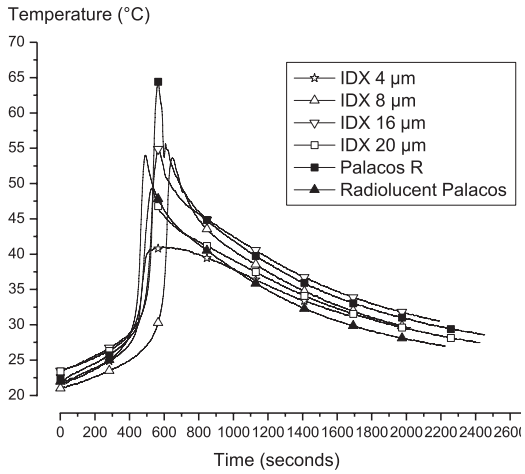

Figure 6. Temperature graph from the thermographical measurement for radiolucent cement containing 8 wt% iodixanol with the particle sizes 4, 8, 16 and 20 µm MMD. Palacos R and radiolucent Palacos are also included as controls.

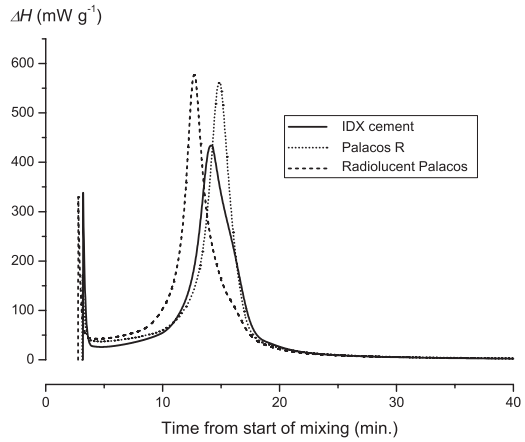

Figure 7. Exotherm graph from the isothermal differential scanning calorimetry for radiolucent cement containing 8 wt% iodixanol with a particle size of 6 µm MMD. Palacos R and radiolucent Palacos are also included as controls.

rimetry are shown in figure 7. The polymerisation heat ΔH for radiolucent Palacos is 105.9 J g^{-1} with the peak occurring at 12.7 min. The IDX cement behaved similarly with a ΔH of 105.2 J g^{-1} with the peak occurring at 14.2 min, and the Palacos R showed a lower polymerisation heat of 101.3 J g^{-1} with the peak occurring at 14.8 min.

Table 6. Glass transition temperatures (T_g) calculated on the second heating cycle for bone cement containing 8 wt% IDX, radiolucent Palacos and Palacos R.

Material	Amount (mg)	Tg (°C)
Palacos with IDX	11.147	103.74
Pure Palacos	11.322	104.14
Palacos R	12.951	101.99

Glass transition temperature by constant rate differential scanning calorimetry

The glass transition temperatures extracted from the constant rate DSC are shown in table 6. It does not appear that the addition of IDX to the formulation has affected the T_g in any manner.

Discussion

The results demonstrate that the quasi-static mechanical properties are related to the particle size of the contrast media in the bone cement. Bone cement containing large particles demonstrate better mechanical properties than bone cement containing smaller contrast media particles, up to a particle size of about 15 µm where the mechanical properties seem to plateaux.

The results indicate differences between the various test methods; the smallest difference in mechanical strength is seen in the compression study, where the difference in compressive strength was only 10.4 % between the maximum and minimum values. The differences between the maximum and minimum values in flexural strength and in tensile strength were 24 and 13.5 % respectively. Also, the main shift in mechanical strength comes into effect at a lower particle size in tension than in flexion and compression. The largest shift in UTS takes place between the 5 µm group and the 8 µm group, the change in either compressive strength or maximum flexural strength is more gradual and takes place at a larger particle size. The results from the flexural test and the tensile test indicate that the material undergoing tensile stress is more sensitive to the filler than a material undergoing compressive stress, in accordance with the results of [15] and [16]. Under tension the failure will occur

at the filler–cement interface, whereas compressive failure will occur in the cement matrix, that is the composite matrix which is the same for all the materials tested. Large stress concentrators are detrimental to the strength of polymers; however, we failed to observe such a relationship in this study. Most likely, our difficulty in seeing such a relationship clearly, lies in the selection of particle sizes. A larger particle size, up in the region of 30–50 μm would possibly have detected the upper transition (reduction) in mechanical properties. However, it was of interest in the current study to investigate the lower transition (reduction) in mechanical properties.

The mechanical properties to some extent follow what was found when the addition of triphenyl bismuth (TPB) as an opacifier to a bone cement was investigated [17,18]. They found that the ultimate tensile strength of bone cement which contained 10 wt% TPB powder, stored in distilled water for two weeks in 37 °C prior to testing, was 42.95 MPa, similar to our findings. Further more, the strain to failure reported (1.7 %) is lower than what we found; the 15 μm IDX cement had a strain to failure of 2.2 %. However, since it is not the same base material the results are not directly related, but it can give some indication. Further more, the compressive strengths was similar. However, the flexural strength in our study was lower than what was reported for the TPB cement, we found the maximum flexural strength to be 58.64 MPa, this can be compared to 83.77 MPa in the other study. A comparison between four cements [19], including, radiolucent cement, cement containing $BaSO_4$, cement containing ZrO_2, and cement containing a radiopacifying monomer, IHQM, was done. The findings correlate to some extent to our results, similar compressive and tensile strengths were found for the ZrO_2-cement, but the radiolucent cement showed a substantially lower value than what we found. However, the base cement (cement without opacifier) differs and this can, most likely, explain the difference.

The reason for the difference in mechanical strength between the cements that contain small particles and the cement that contain larger particles can possibly be explained by two processes; firstly, small particles tend to agglomerate, as was shown by Liu *et al.* [16] who reported that $BaSO_4$ is prone to form agglomerates within the polymer matrix, thereby acting both as stress concentrators degrading the mechanical properties of the bone cement, and, by being agglomerates, they are themselves mechanically weak, thus intra agglomerate failure can occur. Secondly, the small size of the powders can result in them covering the individual polymer beads, and that there are a large number of these particles and thus they affect the polymerisation between the co-polymer and the monomer. During mixing, the particles could remain as a layer over the individual cement beads, rather than becoming thoroughly mixed with the monomer liquid and being fully incorporated during polymerisation [11]. Since the polymer beads are usually in the particle size ~150 μm and the contrast media particle size down to <1 μm, the surface of the individual polymer beads are covered by the small contrast media particles to such a high degree that they can interfere with the polymerisation process leading to shorter interconnecting polymer chains between the polymer beads, which would again decrease the mechanical properties of the bulk material [20].

Molecular weight analysis did not show a noticeable difference of molecular weight between the different groups, although within the IDX cements it was found that increasing the particle size increased the polydispersity index, which describes the width of the molecular weight distribution. Since reduced molecular weight of acrylic bone cement is associated with decreased resistance to fracture and also to reduced fatigue properties [20,21], the materials that have lower molecular weight might not be suitable for load-bearing applications. However, the results do not support the hypothesis stated earlier that the large number of small particles which covers each individual polymer bead acts as a hindrance during the formation of the polymeric chains.

Analysis of the maximum polymerisation temperatures of the materials did reveal that the smallest IDX particle size group had a substantially lower maximum polymerisation temperature than the other IDX cements. The 4 μm IDX cement had a maximum polymerisation temperature of 41.4 °C, which is lower than those found in the other IDX cements, ranging from 49–54 °C. Since the maximum polymerisation temperature for this particle size is so different from the maximum poly-

merisation temperatures of the other IDX cements, it is conceivable that the polymerisation reaction has been affected more at this lowest particle size. The IDX containing cements show overall lower polymerisation temperatures than radiolucent Palacos or Palacos R, indicating a reduced risk for thermal necrosis to the bone [22], which is associated with heat released to the bone bed by the bone cement as it polymerises *in situ*. Temperatures *in situ* are known to be lower than *in vitro* [23]. The maximum polymerisation temperatures for these materials are nevertheless similar to those reported for commercial cements [24,25].

The opacifier particles that are present in the material can be affecting the performance of the monomer, due to the large total surface area of the particles. The bulk powder, including the particulate opacifier, is wetted by the monomer, thereby the amount of monomer that is available during the dissolution of the surface of the polymer beads are reduced. The reduction in available monomer may reduce the polymerisation rate and thereby the maximum temperature. At higher polymerisation rates, larger numbers of polymer chains are active, creating a material that contain many shorter polymer chains [26]. Further more, higher temperatures increases the disproportionation chain termination (where two active chains meet and are terminated into two separate chains) [26], leading to a large variation of the molecular weight, i.e. higher polydispersity. By this explanation the radiolucent Palacos show a lower polydispersity index than what is expected, however, the monomer to polymer ratio is lower in this cement than in the experimental cements [24], which have a direct impact on the polymerisation process. The low polydispersity index of Palacos R can partly be explained by the higher amount of radiopacifier present (15 wt% ZrO_2) in the cement, which in turn alter the powder to liquid ratio.

The polydispersity index could help to explain the low polymerisation temperature of the 4 µm IDX cement and the low peak of the polymerisation heat for the 6 µm IDX cement. However, it seems that the reaction is merely slowed, since the DSC analysis did not detect any difference in the total polymerisation heat, as it was similar between the radiolucent palacos and the IDX cement. Instead it was the Palacos R that demonstrated the lowest polymerisation heat, but the difference was minimal. All values were, however, higher than those (72–88 J g^{-1}) reported by Yang [27], whereas our values were in the range 101–106 J g^{-1}, however, we used Palacos R as the base material and they used Simplex P. Further more, the isothermal temperature differed, we used 20 °C and they used 25 °C. As for the glass transition temperature, which is related to the thermal expansion coefficient, the flexural modulus, and the mechanical absorption [24], no difference could be observed between the three materials. The addition of 8 wt % IDX to the PMMA does not seem to alter the glass transition temperature of the material. Our results are higher than reported by Kühn [24], however, the methodology used in the two studies differs to a large extent.

Conclusions

The quasi-static mechanical properties of the cement with the larger IDX particles are similar to that of Palacos R; it is therefore proposed that IDX is a viable option as a contrast media in bone cement. The investigation show that the mechanical properties depend on the particle size of the IDX, and that the optimum particle size is above 8 µm. The addition of IDX to bone cement does not change the molecular weight of the final polymer, neither does it change the glass transition temperature. However, the addition of IDX does lower the maximum polymerisation temperature of the cement.

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References

1. L. CARAVIA, D. DOWSON, J. FISHER and B. JOBBINS, *Proc. Inst. Mech. Eng [H.]* **204** (1990) 65.

2. J. R. COOPER, D. DOWSON, J. FISHER and B. JOBBINS, *J. Med. Eng Technol.* **15** (1991) 63.
3. G. H. ISAAC, B. M. WROBLEWSKI, J. R. ATKINSON and D. DOWSON, *Clin. Orthop. Relat. Res.* **276** (1992) 115.
4. P. ASPENBERG and H. VAN DER VIS, *Clin. Orthop. Relat. Res.* **352** (1998) 75.
5. J. A. WIMHURST, R. A. BROOKS and N. RUSHTON, *J. Bone. Joint. Surg. Br.* **83B** (2001) 278.
6. J. A. WIMHURST, R. A. BROOKS and N. RUSHTON, *J. Bone. Joint. Surg. Br.* **83B** (2001) 588.
7. F. KJELLSON, T. ALMEN, K. E. TANNER, I. D. MCCARTHY and L. LIDGREN, *J. Biomed. Mater. Res.* **70B** (2004) 354.
8. T. ALMÉN, *Acta Radiol. Suppl* **399** (1995) 2.
9. J. S. WANG, J. DIAZ, A. SABOKBAR, N. A. ATHANASOU, F. KJELLSON, K. E. TANNER, I. D. MCCARTHY and L. LIDGREN, *J Roy Soc Interface* **2** (2005) 71.
10. F. KJELLSON, B. BRUDELI, I. D. MCCARTHY and L. LIDGREN, *J. Biomed. Mater. Res.* **71A** (2004) 292.
11. F. KJELLSON, J. S. WANG, T. ALMEN, A. MATTSSON, J. KLAVENESS, K. E. TANNER and L. LIDGREN, *J. Mater. Sci Mater. Med.* **12** (2001) 889.
12. A. BORZACCHIELLO, L. AMBROSIO, L. NICOLAIS, E. J. HARPER, K. E. TANNER and W. BONFIELD, *J. Mater. Sci Mater. Med.* **9** (1998) 835.
13. A. BORZACCHIELLO, L. AMBROSIO, L. NICOLAIS, E. J. HARPER, K. E. TANNER and W. BONFIELD, *J. Mater. Sci Mater. Med.* **9** (1998) 317.
14. S. N. ABDULGHANI, S. N. NAZHAT, J. C. BEHIRI and S. DEB, *J. Biomater. Sci Polym. Ed.* **14** (2003) 1229.
15. K. FRIEDRICH and U. A. KARSCH, *J. Mater. Sci* **16** (1981) 2167.
16. C. LIU, S. M. GREEN, N. D. WATKINS, P. J. GREGG and A. W. MCCASKIE, *Proc. Inst. Mech. Eng [H.]* **215** (2001) 359.
17. S. DEB, S. ABDULGHANI and J. C. BEHIRI, *Biomaterials* **23** (2002) 3387.
18. S. ABDULGHANI, Thesis title:*"An investigation into the mechanical and biological properties of acrylic bone cement containing triphenyl bismuth (TPB) as an alternative radiopacifier"* Department of Materials, Queen Mary, University of London (2003).
19. M. P. GINEBRA, L. ALBUIXECH, E. FERNANDEZ-BARRAGAN, C. APARICIO, F. J. GIL, R. J. SAN, B. VAZQUEZ and J. A. PLANELL, *Biomaterials* **23** (2002) 1873.
20. J. GRAHAM, L. PRUITT, M. RIES and N. GUNDIAH, *J. Arthroplasty* **15** (2000) 1028.
21. K. F. HUGHES, M. D. RIES and L. A. PRUITT, *J. Biomed. Mater. Res. A* **65** (2003) 126.
22. N. J. DUNNE and J. F. ORR, *J. Mater. Sci Mater. Med.* **13** (2002) 17.
23. S. TOKSVIG-LARSEN, H. FRANZEN and L. RYD, *Acta Orthop. Scand.* **62** (1991) 102.
24. K.-D. KÜHN, in "Bone Cements" (Springer-Verlag, Berlin Heidelberg, 2000)
25. G. BAROUD, M. SAMARA and T. STEFFEN, *J. Biomed. Mater. Res.* **68B** (2004) 112.
26. G. G. ODIEN, in "Principles of Polymerization" (John Wiley & Sons, Inc, New York, 1981)
27. J. M. YANG, *Biomaterials* **18** (1997) 1293.