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Effects of preparation techniques on the porosity of acrylic cements

B. Willem Schreurs, Pieter T. J. Spierings, Rik Huiskes and Tom J. J. H. Slooff

We investigated four acrylic cement preparation techniques for their effects on cement porosity: hand mixing, pressurization in a pneumatic pistol, centrifugation, and vacuum mixing. All the techniques were tested on three types of cement with different viscosity characteristics. The best results were obtained with vacuum mixing using a newly designed experimental system, yielding porosity reductions of 60-80 percent relative to hand mixing. Vacuum mixing with a commercial system was also effective, but to a somewhat lesser extent.

Pressurization and centrifugation had no substantial effect on the overall porosity. Centrifugation led to considerable nonuniformity in the distribution of pores and additives.

The porosity of acrylic cement is important for its fatigue strength and depends, among other things, on the preparation technique. We have determined the effects of four different techniques on the porosity of three cements with different viscosity characteristics in the curing phase.

Material and methods

Cylindric test bars

Three different cement types were used: a low viscosity type (Zimmer LVC, Zimmer Inc., Warsaw, ID, U.S.A.), a medium one (Palacos EMD 42 521-Y), and a high viscosity type (Palacos R, Merck, Darmstadt, FRG.). In order to document the differences between these cements, their viscosity developments were measured in a Weissenberg Rheogoniometer, type R19 (Sangamo Controls Ltd., U.K.). Using a rotating plate-cone configuration (Walters 1975), the simple shear

viscosity as a function of time after mixing was determined (shear rate 0.36 s^{-1} and a mean room temperature of $22 \text{ }^\circ\text{C}$). Although these viscosity vs. time curves are extremely sensitive to both the shear rate and the environmental temperature, objective comparisons could be made because all the test conditions were kept equal.

In the porosity tests, 26-mm-diameter cylindric cement bars were prepared. Means and standard deviations (SD) of the porosity values for the three cements, prepared in four different ways, were determined from the measured apparent density values, relative to the densities of the standard test bars. The reproducibility of the apparent density determination was 0.0015 g/cm^3 (approx. 0.12 percent), as determined from duplicate measurements, which is small relative to the variety found between test samples. During the tests the laboratory temperature was $24 \pm 1 \text{ }^\circ\text{C}$ and the air humidity 30 ± 5 percent.

The different methods of cement preparation are illustrated in Figure 1. Hand mixing was carried out in a plastic bowl with a spatula, stirring frequency 1-2 rounds/s in accordance with the instructions of the manufacturer. After mixing, the cement dough was poured (LVC and Palacos E) or finger packed (Palacos R) into a cylindric mold.

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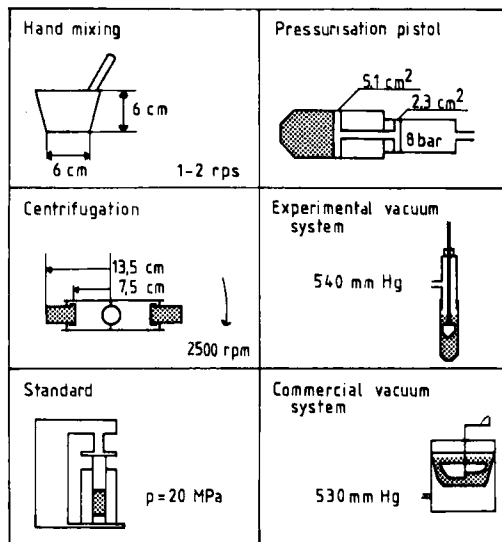


Figure 1. Specifications for the hand-mixing method (upper left), pressurization pistol (upper right), centrifuge (middle left), experimental vacuum system (middle right), pressure curing for the reference bars (lower left), and the commercial vacuum system (lower right).

In testing the pressurization pistol (Scientific Developments, Munich, FRG), mixing took place as in hand mixing, after which the cement dough was poured or finger packed (Palacos R) into the plastic pistol syringe and pressurized for 1 minute (high viscosity cement) to 3 minutes (low viscosity cement) at 8 bars air pressure in accordance with the instructions of the manufacturer. At the end of the pressurization period, the cement was left to polymerize.

Centrifugation was carried out, starting 80 seconds after normal mixing of the cement, in a Cemtrifuge® (Biodynamic Technologies Inc., Pompano Beach, U.S.A.), at an angular velocity of $2,500 \pm 50$ rpm (strobe controlled), for 2 minutes, including 20 seconds' drag. The radius of the centrifuge was between 7.5 cm (top) and 13.5 cm (bottom). Cement mixing and syringe insertion were as in pressurization.

Vacuum mixing was performed first in a self-made apparatus. The design of this device was based on four requirements: 1) Powder and fluid are combined after a vacuum is applied; 2) stirring takes place in a vacuum; 3) the stirring rotor is removed in a vacuum; and 4) the cement dough is injected directly from the apparatus into the

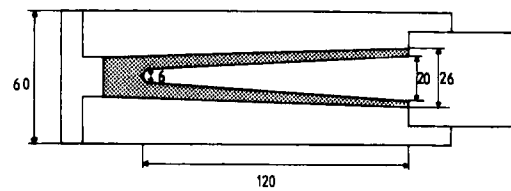


Figure 2. The femur/stem mold for the simulation of stem fixation. The femur mold is made out of PTFE, the stem out of metal.

bone or, in this case, in the test mold. The powder was put into a 26 mm plastic syringe, which was a part of the small Howmedica cement pistol. The fluid was poured into a glass bowl. Then, a vacuum of $540 (\pm 10)$ mmHg was applied and the glass bowl was rotated to pour the liquid into the powder. Stirring was carried out with the rotor at 220 rpm for 45 seconds. Then, the rotor was pulled out, the vacuum was released 2 minutes after mixing started, and the cement was left to cure.

For the purpose of producing zero-porosity reference samples, standard test bars were made for each cement under pressure of $20 (\pm 2)$ MPa in a hydraulic press in the curing phase (15 min).

Stem fixation model

Tests were carried out with a mold simulating the curing conditions of cement around a femoral prosthesis (Figure 2). The purpose of this test was to determine whether porosity reductions, obtained during preparation, would be permanent after application of the cement in artificial-joint fixation. In this case only the Palacos E cement was used, prepared by hand or by vacuum mixing.

In addition to the experimental vacuum device, the commercial Mixevac II® (Stryker, U.S.A.) was used (Figure 1) according to the instructions of the manufacturer. In this case a vacuum of 508–559 mmHg was applied after adding powder to the liquid, but before the mixture was stirred in a closed plastic stirring mill. This system was used both with and without the application of a vacuum in order to have a control for the use of the stirring mill alone. After stirring, the vacuum was released, the stirrer removed, and the cement poured into a syringe.

In all the cases the Howmedica syringe was used to fill the femur mold in the antegrade fashion.

Table 1. Density values of the standard reference samples cured under 20 MPa pressure, as measured pycnometrically

Cement type	Density (g/cm ³)	
	Sample I	Sample II
Zimmer LVC	1.242	1.243
Palacos E	1.293	1.289
Palacos R	1.290	1.290

The metal stem was inserted 4 minutes after starting the mixing. The stem and the cement mantle were removed 15 minutes after insertion.

Porosity evaluation

All the test bars and cement mantles, for each technique and each cement, were produced five times except for the standard bars, which were produced twice. All of them were dehydrated for 10 (\pm 1) days in an exsiccator containing silica gel. After this period, the apparent density of each sample was determined using pycnometric methods according to ASTM 792-66 (Table 1). All the measurements were carried out in duplicate; ultrasound was used for removing air bubbles.

In addition, the test bars were radiographed. Then, two out of every five bars were sectioned using water cooling; the centrifugal specimens in four equal parts, and the others in three. The sections were polished, macroscopically checked, and evaluated for area porosity by quantitative microscopy (automatic gray discrimination of video image).

Results

Cylindric test bars

Due to its high viscosity, vacuum mixing of Palacos R cement proved impossible because of the stirring methods, both in the experimental and in the commercial system (Figure 3).

Centrifugation of LVC cement created a thin liquid layer on top of the test bar, curing eventually into a foamlike matter. The centrifuged Palacos E samples showed a white layer on the other side, close to the bottom, which was on the far side of the centrifugation axis. This material turned out to be the radiopaque filler zirconium oxide.

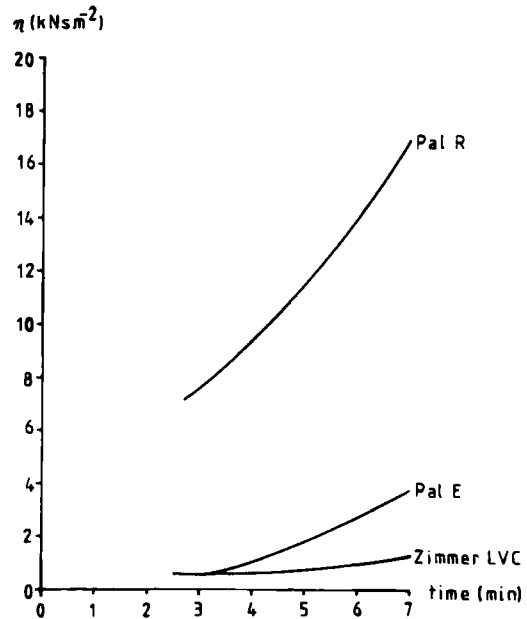


Figure 3. Viscosity developments as function of time after start (hand) mixing for Palacos R, Palacos E-Y, and Zimmer LVC. Average of five tests in a plate/cone configuration of a rotating rheogoniometer (room temperature 21.6-22.7 °C; shear rate 0.36 s⁻¹).

Microscopic evaluation of the standard reference test bars, produced under high pressure, showed a porosity of less than 0.1 percent in all the cases, indicating that the requirement of zero porosity was nearly fulfilled (Table 1).

Porosity reductions relative to hand mixing were only obtained with the vacuum method (Table 2). However, the centrifuge seemed to have some effect with Palacos E cement and the pressurization pistol with Palacos R cement. In all the cases, the Palacos E cement had lower porosity than LVC and Palacos R.

The radiographs displayed macroscopically visible pores for all three cements prepared by hand mixing and by the pressurization pistol technique. In centrifuged samples of LVC and Palacos E, no pores were visible in contrast to Palacos R. The centrifuged LVC samples showed a cloudy density near the bottom, on the far side of the centrifugation axis (Figure 4); and the centrifuged Palacos E samples showed a dense layer at the far end. The radiographs of the vacuum mixed LVC and Palacos E samples showed macroscopic pores only occasionally near the top.

Table 2. Volume percentages porosity (mean [SD]) in the cylindrical specimens, calculated from the apparent density measurements of the test samples* as determined with microscopic porosity measurements

Cement type	Porosity (%). Preparation Method				Experimental vacuum system
	Hand-mixed	Pressurization pistol	Centrifuge		
			Bottom	Top	
Zimmer LVC	7.7 (0.7)	6.8 (1.2)	9.3 (1.4)		1.2 (0.1)
Palacos E	4.2 (0.4)	4.3 (0.3)	3.2 (0.4)		0.9 (0.2)
*Palacos E	5.1 (3.3)	5.5 (3.5)	3.4 (1.2)	4.4 (1.8)	0.3 (0.5)
Palacos R	8.4 (1.1)	6.6 (1.1)	6.9 (0.6)		-

Table 3. Average pore size for pores > 60 μm in the cross sections of the test samples, as estimated microscopically

Cement type	Average pore size (mm). Preparation Method				Experimental vacuum system
	Hand-mixed	Pressurization pistol	Centrifuge		
			Bottom	Top	
Zimmer LVC	0.22	0.20	0.16	0.30	0.17
Palacos E	0.18	0.21	0.14	0.18	0.10
Palacos R	0.25	0.25	0.25	0.31	-

Table 4. Volume percentages porosity (mean [SD]), as calculated from the apparent density measurements, in the test samples and in the cement mantle, simulating femoral stem fixation (Palacos E)

Test configuration	Porosity % Preparation Method			
	Hand-mixed	Experimental vacuum system	Commercial vacuum system	
			with vacuum	without vacuum
Cylindric bars	4.2 (0.4)	0.9 (0.2)	-	-
Stem fixation model	2.6 (0.6)	0.7 (0.1)	1.3 (0.3)	3.0 (0.5)

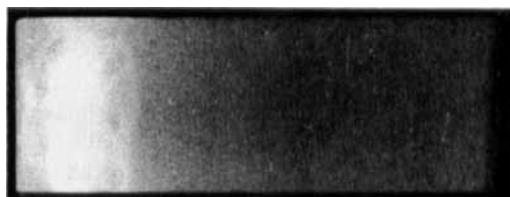


Figure 4. Radiograph of a centrifuged LVC cylinder. Bottom to the left.

Macroscopic inspection and measurement of the polished cross sections of the test bars revealed pores of 0.5 to 6.0 mm diameter for all three cement types after hand mixing and pressurization with the pistol. The largest pores, up to 6 mm, were found in the hand-mixed Palacos R. The centrifuged samples of the LVC and Palacos E cement showed no pores larger than 0.5 mm; some were visible, however, in the Palacos R samples (up to 1 mm). Macroscopic pores in the vacuum-mixed samples were seen only incidentally.

Under the microscope, pores were seen in all cross sections of all the samples except for the standard bars (Figure 5). These results for Palacos E confirm those of the apparent density determinations, but in this case the standard deviations were much higher (Table 2).

A notable finding was that in the centrifuged samples of all three cements the porosity in the bottom section (on the far side of the centrifugation axis) was the smallest, and in the top section the largest (Figure 5). The LVC samples showed the highest gradient in this respect (bottom 3 percent, top 16 percent). The average diameter of all the pores larger than 60 μm in the cross sections was estimated microscopically (Table 3).

Stem fixation

The porosity reduction of vacuum mixing had a permanent character also after application of the cement in prosthetic fixation (Table 4). Some

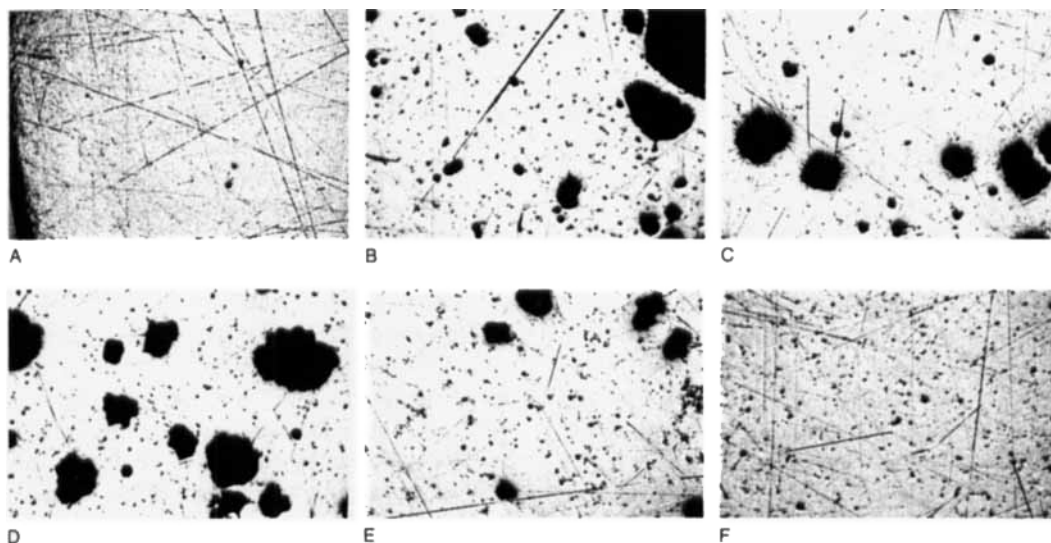


Figure 5. Photomicrographs of the cross sections of the Palacos E test samples (X 57). A. Standard reference. B. Hand-mixed. C. Pistol. D. Centrifuged (top). E. Centrifuged (bottom). F. Vacuum-mixed.

general reduction in porosity occurred when comparing the cement mantle with the test bars. This may be an effect of superior heat conduction from the cement mantle, with less monomer boiling as a result or of porosity reductions during flow through the nozzle of the syringe. The commercial vacuum system, although to a somewhat lesser extent, had an effect similar to the experimental system on cement porosity.

Discussion

According to de Wijn (1982), pores in acrylic cement can be caused by either of three mechanisms: by air solutions in the monomer liquid, by entrapment of air in the cement dough during stirring, and by boiling of monomer. Debrunner (1976) showed that microscopically visible pores smaller than $60 \mu\text{m}$ in diameter are abundant, but do not contribute substantially to the overall porosity. Bayne et al. (1975) achieved a reduction in the porosity of cement when using high pressure in curing. The pressures did not affect the polymer chain length, so it may be assumed that our standard test bars, also cured under pressure, are valid references for porosity.

The porosity values found by us in the hand-mixed samples are close to earlier results reported in the literature; Keller and Lautenschlager (1983) found a 6.4 percent average porosity in

LVC cement, and Jasty et al. (1985) found a 9 percent average porosity in Palacos R cement. Gates et al. (1983) introduced the centrifugation technique for reducing the porosity and increasing the strength of acrylic cements. Burke et al. (1984), from the same group, found an increase in static and fatigue strength in Simplex P specimens after centrifugation. Jasty et al. (1985) found definite effects of centrifugation on the porosity in AKZ and Simplex P cements, but not in Palacos R and Zimmer LVC cements. The latter findings were confirmed by Rimnac and Wright (1985), who reported that centrifugation had no effect on the fracture toughness of Palacos R and Zimmer LVC cements.

A general problem when comparing results of cement centrifugation is that angular velocities and centrifugation time are variable, and that centrifuge radii are seldom reported. It also appears from the literature that the effects of preparation techniques vary among cements, predominantly depending on the viscosity vs. time characteristics. Hence, documentation of these characteristics is of importance (Figure 3). The three cements tested by us are believed to reasonably represent the variation in cement types that are available.

Our findings relative to the centrifugation of Palacos R cement are in general agreement with those of Jasty et al. (1985). They did not, how-

ever, report the increased porosity of Zimmer LVC cement that we found, probably because of the less dependable cross-sectional microscopic evaluation technique that was applied in their measurements. This increase in our tests was mainly an effect of local monomer boiling and foaming.

Skinner and Murray (1985) reported the effects of centrifugation on the apparent density distribution in the cement, whereby the radiopaque fillers in particular are forced away from the centrifugation axis, through the cement dough, and the pores remain concentrated on the other side. We found separation of radiopaque fillers with Zimmer LVC and Palacos E.

Because pores of macroscopic size were not found in the radiographs of the centrifuged samples, in contrast to the hand-mixed samples, and because the overall porosity of the centrifuged samples was not reduced, it must be assumed that this technique divided the larger pores into smaller ones. The gradient in the average pore size over the length of the cylindrical bars can be explained by the nonuniform distribution of the centrifugation forces. It is probably due to the higher viscosity of Palacos R that this mechanism occurs to a lesser extent in this case.

The pressurization pistol is assumed to reduce the sizes of pores in the cement. Because the viscosity of the cement increases during the pressurization period, the reduced pore sizes would be permanent (Draenert 1986). It is important to note that due to an area increase of 225 percent, the real pressure on the cement is only 3.5 bars when the air pressure is 8 bars.

Keller and Lautenschlager (1983) and Eyerer and Jin (1986) reported that an increased cement porosity resulted in lower tensile strength. The former authors found an increase of strength when using the cement pistol without prepressurization. These results may be explained by the effects of the small nozzle of the pistol (3.5 mm diameter), which may cause a redistribution of the pores while the cement is flowing out.

We found here that the use of the pistol had no effect on the pore sizes in either of the three cements, whereas the overall porosity did not change in Palacos E and Zimmer LVC, and was reduced only slightly in Palacos R.

Vacuum mixing resulted in substantial reductions of the overall porosity and average pore sizes in both Palacos E and Zimmer LVC cements. Unfortunately, it could not be applied to Palacos R, due to its high viscosity.

Demarest et al. (1983) mixed Simplex P cement under 730 mmHg vacuum and obtained an average porosity reduction from 5 percent to 1 percent, and a clear increase in strength. Lidgren et al. (1984) compared the effects of 570 mmHg vacuum mixing on a high and a low viscosity cement. They found that the results of the high viscosity cement were the most dramatic, amounting to a strength increase of 15-20 percent. Later, Lidgren et al. (1987) compared the fracture strength, stiffness, hardness, fatigue life, and porosity of hand- and vacuum-mixed Palacos cement kept at 4 °C. They found substantial improvements of mechanical properties and a reduction of the porosity for the vacuum method. Wixson et al. (1985) obtained a porosity of 0.1 percent in vacuum-mixed Simplex P, as measured microscopically in cross sections.

Simulating the application of cement in hip-prosthetic fixation, we found that the porosity reduction obtained in vacuum mixing was permanent. The overall porosity in the actual cement mantle was even less than in the test samples, probably as an effect of the lower curing temperature, or due to flow in the nozzle of the syringe (Keller and Lautenschlager 1983).

The use of both the experimental and the commercial vacuum system resulted in porosity reductions, but to a much higher degree in the experimental system. This was caused by creating a vacuum before the powder and the fluid were combined, and by pressing the dough directly out of the cartridge after mixing.

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