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CHARACTERIZATION OF BONE CEMENTS

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For the past twenty years bone cements, based on polymethylmethacrylate, have been used in orthopaedic surgery for the fixation of endoprostheses especially in cases of total hip replacement. During this period a large number of papers have been published on the use of acrylic cement.

From the beginning it has been clear that the cement itself and the method of intramedullary implantation have a distinct influence on the surrounding tissues and on various physiological functions of the body during or shortly after operation.

As a result several clinical and experimental studies have been carried out regarding the unfavourable side effects of the acrylic cement, such as the development of allergy to the monomer, and to the inhibitors and catalysts, as well as their cytotoxic effects and the histopathological reactions in the surrounding cortical bone.

In addition, much attention has been paid to the causes of physiological disorders such as a drop in blood pressure during implantation of the acrylic cement and fat embolism.

Clinical follow-up studies of total hip replacements have been published dealing with postoperative complications such as infection, loosening of the prosthesis and loss of function. Biomechanical investigations have been carried out with respect to the strength of the construction bone-prosthesis with and without fixation by acrylic cement and to the techniques and possibilities for obtaining an optimal bond between cement and bone.

A final group of investigations to be mentioned here deals with measurements of the temperature rise and intramedullary pressure during implantation and hardening of the cement and with methods to keep this temperature down to an acceptable level.

Generally there is agreement that all these untoward side effects are due to the monomer in the acrylic cement, to the metabolites, to the large temperature rise during polymerization and to the method of implantation in the medullary cavity. Up to this moment, however, an exact analysis of the possible causes of the various unfavourable side effects has not been made.

Until now, little attention has been paid to the physical and mechanical properties of the various cements themselves, or to the meaning of these properties with respect to the functioning of the construction as a whole. The purpose of this investigation is an attempt to fill in this gap in the information.

For three of the most commonly used bone cements* we determined curing time and consistency as working properties of the uncured cement mixture; water resorption, solubility and disintegration were selected to yield an impression of the physicochemical stability of these materials. For comparison of mechanical properties, flexural strength and impact strength of the cured cements were measured. Additionally we determined the influence of additives such as an antibiotic and radiopacifiers on these properties and the influence of porosity on flexural strength and impact strength.

MATERIALS AND METHODS

Bone cements

Simplex P: acrylic bone cement; radiopacifier added by manufacturer ($BaSO_4$).

Palacos R: acrylic bone cement; radiopacifier (ZrO_2) and pigments added by manufacturer.

Palacos K: acrylic bone cement; no radiopacifier but with pigments.

CMW : acrylic bone cement; no heterogeneous additives in powder as supplied. Two packages $BaSO_4$ are delivered separately to be mixed with the cement by the operator.

Our notation: CMW – 0, no additives.
CMW – 1, with 4 per cent $BaSO_4$ added.
CMW – 2, with 8 per cent $BaSO_4$ added.

Antibiotic: Erythromycine lactobionate (Erythrocyne i.v. Abbott S. A. Belgium) added amount: 4.2 per cent by weight.

Curing time

A cylindrical cell made from heat-insulating polyurethane foam was fitted with an Fe-Co-thermocouple and filled with 0.5 ml of the cement mix. Ambient

* Surgical Simplex (North Hill Plastics Ltd., London, England).
Palakos (Kulzer & Co., Bad Homburg, Germany).
CMW (CMW Laboratories Ltd., Blackpool, England).

temperature: 22° C. The thermocouple was coupled to a recording instrument, the chart transport of which was activated when powder and liquid components of the cement were brought together. The time from the start of mixing to the moment the temperature in the cell reached a maximum was taken as the curing time.

5-minute consistency

A fixed amount of the cement was mixed at an ambient temperature of 22° C; 4 min after the mixing was started 0.5 ml of the cement was placed on the centre of a glass plate (50×50×5 mm) by means of a gauged glass tube and covered with a second glass plate; 5 min after the mixing was started a weight was put on the centre of the top glass plate forcing the cement to slump out to a nearly round disc. After the cement was cured under this load and between the glass plates, the mean diameter of the disc was measured. The weight that was necessary to produce a cement disc with a diameter of 25 ± 1 mm was taken as a measure of the consistency.

Water resorption; solubility and disintegration in water

This test is a modification of a test prescribed by the American Dental Association for denture base polymers (A.D.A. Specification no. 12; Guide to Dental Materials and Devices, American Dental Association 1970–1971).

By means of a stainless steel mould, non-porous discs with a diameter of 50 mm and a thickness of 0.5 mm were processed from the cement. Immediately after hardening of the cement the discs were weighed (disc weight a mg) with a precision of 0.2 mg and immersed in distilled water of 37° C in preweighed flasks for 24 h (flask weight b mg). After this time the discs were removed from the water, wiped with a dry handtowel and weighed again (final disc weight c mg).

The water in which the cement had been immersed was carefully evaporated and the flasks were dried at 50° C to constant weight (final flask weight d mg). The weight increase of the cement disc (c–a mg) minus the weight of the flasks (d–b mg) gave the amount of water that had been absorbed by the cement. The weight increase of the flasks after evaporation of the water measured the non-volatile ingredients of the cement which were dissolved or disintegrated. Finally the discs were dried to constant weight in a desiccator containing dry anhydrous calcium sulphate at 50° C (dried disc weight e mg).

The weight decrease caused by this treatment (c–e mg) is a measure of the total amount of volatile ingredients of the cement after water resorption.

Flexural modulus of elasticity

For this test we used an apparatus as described by the American Dental Association for transverse deflection tests of denture base polymers (A.D.A. Specification no. 12). By means of a stainless steel mould the cement was processed to a non-porous test specimen of 65×10×2.5 mm. The specimen was conditioned in distilled water at 37° C for 24 h. The deflection of the specimen caused by a load of 1000 g and, to check for linearity, also one of 2000 g was measured 5 seconds after loading. The flexural modulus was calculated by means of the following expression

$$E_b = \frac{P}{F} \frac{L^3}{4BD^3}$$

E_b = flexural modulus of elasticity in $kgmm^{-2}$; P = load in kg; F = deflection in mm; L = the width of the span; B = width of the specimen and D = thickness of the specimen.

Compressive strength and proportional limit

Cylindrical test specimens were made by filling precision glass tubing having an inside diameter of 6 mm with the cement mix. Non-porous rods were obtained by curing the cement at 2 atm. in a high-pressure vessel. From these rods specimens of 12 mm in length were sawn and the ends of the cylinders were ground flat and parallel to each other with 600 mesh grinding paper. The test specimens were conditioned for 24 h in distilled water at 37° C and tested in compression at 22° C by means of an Instron testing machine.

Compressive strength and proportional limit were determined at three different initial loading rates: 1.5 kg cm^{-2} s^{-1}; 15 kg cm^{-2} s^{-1} and 150 kg cm^{-2} s^{-1}.

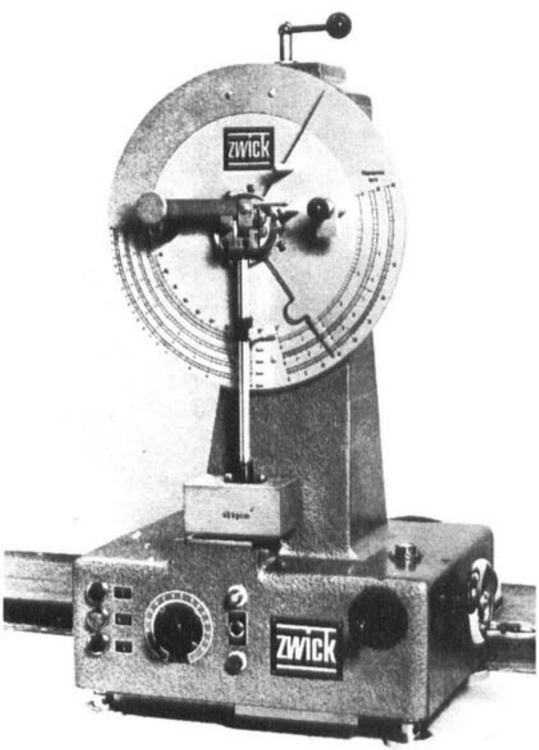

Figure 1. The Dynstat-apparatus for determination of flexural strength and impact strength.

Flexural strength and impact strength

These measurements were made with a Dynstat apparatus (Zwick & Co., Eisingen, Germany) according to DIN 51230, by means of which a four-point flexural test (DIN 53452) as well as an impact test (DIN 53453) can be performed (Figure 1).

The dimensions of the test specimens were 15×10×2 mm. Specimens were conditioned for 24 h in distilled water at 37° C and all tests were carried out at 22° C ambient temperature.

RESULTS

Curing time

The tested cements appeared to differ slightly in curing time as defined and measured in the manner described. Simplex cement hardened in 12–13 min, Palacos R in 10–11 min and CMW cement in 6–8 min. Radiopacifiers did not influence the curing time of CMW cement.

Consistency

As it is obviously not possible to determine the viscosity of a curing cement mix, it is common practice to take the "consistency" after a certain time as a measure for the workability of such a mix. In this study the consistency is defined as the force necessary to slump out 0.5 ml cement mix between two glass plates to a disc with a mean diameter of 25 ± 1 mm. The force was applied 5 min after the mixing was started; this time is an estimate of the time necessary for mixing of the components, waiting for dough formation, filling of a cement syringe (Slooff 1969), filling of the medullary cavity and introduction of the prosthesis.

The results indicated that Simplex cement had the most fluid consistency after 5 min, the relevant force being about 200 gf. Palacos R required 400 gf and CMW cement with 1200 to 1500 gf appeared to have the thickest consistency after 5 min. Of course these results are closely related to the curing times of the various cements tested.

Water resorption—solubility—disintegration

The tested cements showed no difference or only slight differences with respect to these properties. Water resorption percentages were 0.9 to 1.3 per cent for Simplex, Palacos R and CMW without radiopacifiers. The addition of radiopacifier (8 per cent $BaSO_4$) resulted in a slight increase of the absorption of water (1.2–1.5 per cent), while adding an

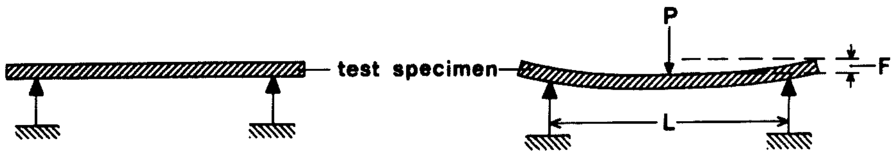

Figure 2. Principle of bending test; P = *load,* F = *resulting deflection;* L = *width of span.*

antibiotic (4.2 per cent erythrocine-lactobionate) seemed to enhance water resorption in CMW cement (1.6–1.8 per cent).

The amount of volatile contents appeared for all cements to lie between 1.9 and 2.3 per cent, while addition of an antibiotic raised this value to about 3 per cent.

Solubility and disintegration were immesurably low for all cements tested except for the cements to which erythrocine was added. In this case a solubility of 0.2 per cent per 24 h in distilled water of 37° C was found, which reflects, of course, the desired solubility of the antibiotic.

Flexural modulus of elasticity

The principle of this bending test is shown in Figure 2. When the deflection of the specimen resulting from a load P is determined, a measure for the stiffness of the material can be calculated from

$$E_b = \frac{P}{F} \frac{BD^3}{L^3}$$

E_b being the modulus of elasticity (kg/mm^2); P the applied load (kg); F the deflection (mm); B and D the width and thickness of the specimen, respectively (mm). This test did not reveal significant differences between the cements tested; all measurements were between 240 and 290 kg/mm^2.

Compressive strength; proportional limit

The compressive strength of a material is defined as the compressive stress (force per unit area) causing the material to break. In the case of high molecular weight materials, such as bone cements, most mechanical properties are strongly dependent on the rate with which the material is loaded. Under loading in compression it is likely that the polymeric material will not break (Figure 3 a) but instead will flow continuously beyond a certain maximum load (yield stress)

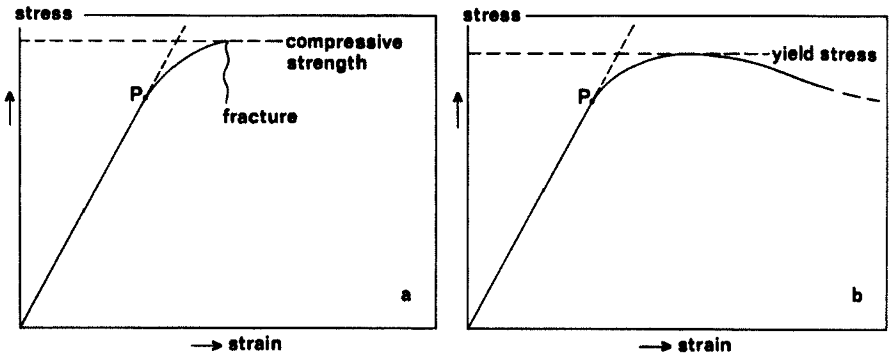

Figure 3 a. Stress-strain curve if (brittle) fracture occurs.

Figure 3 b. Stress-strain curve with plastic flow instead of fracture.
P = proportional limit.

causing the stress-strain curve to pass through a maximum (Figure 3 b).

The linear part of the stress strain curve (low stresses, low strains) represents the elastic area, where the deformation resulting from the applied load is largely reversible. The non-linear part of the curve, beyond the point P in Figure 3 b (the proportional limit), represents the plastic deformation area, where deformation is largely irreversible. Thus when a material is loaded beyond its proportional limit, a permanent deformation will remain when the load is removed.

Consequently, if a material has a load-bearing function, both the proportional limit and the strength are important properties. Because of the dependence of compressive strength (yield stress) and proportional limit on the loading rate it is necessary, when comparing different materials, to keep loading rate constant in all measurements

Table 1. Yield stress ($kg\ cm^{-2}$) at different loading rates.*

Cement	Loading rate $kg\ cm^{-2}\ s^{-1}$ 1.5	15	150
Simplex	575 (± 25)	750 (± 20)	825 (± 35)
Palacos R	560 (± 25)	700 (± 32)	840 (± 51)
CMW–0	650 (± 20)	830 (± 41)	1020 (± 42)
CMW–2	640 (± 21)	790 (± 50)	925 (± 31)
CMW–0+4.2 % erythrocyne	640 (± 15)	810 (± 10)	1020 (± 25)

* Values in parentheses represent the 95 per cent confidence interval of the mean.

or to carry out the determinations of mechanical properties over a certain range of loading rates. In this presentation the last-mentioned possibility has been chosen. The values for proportional limit did not show significant differences for the cements tested. The values were 350 ($\pm$ 50) kg/cm², 440 ($\pm$ 50) kg/cm² and 570 ($\pm$ 50) kg/cm² for loading rates of 1.5, 15 and 150 kg cm^{-2} s^{-1}, respectively. Table 1 shows the yield stresses found for the various cements.

Flexural strength; impact strength

For the determination of flexural strength and impact strength methods were chosen according to DIN 53452 and DIN 53453, respectively, using a Dynstat apparatus according to DIN 51230. This method has been developed especially for the testing of polymeric materials. Relatively small test specimens (15×10×2 or 3 mm) are used with this method, which is important for planned investigations concerning the testing of bone cements, which have been used in patients as fixation material. The principles of these tests are drawn schematically in Figures 4 a and 4 b. Specimens were obtained from plates which were processed from the cement under pressure and thus exhibited no porosity. The results are shown in Table 2. Bone cement without any additives (CMW-0) has the highest values for both flexural strength and impact strength. The availability of this pure form of acrylic cement facilitates the determination of the influence of heterogeneous additives such as $BaSO_4$ and antibiotics on these proper-

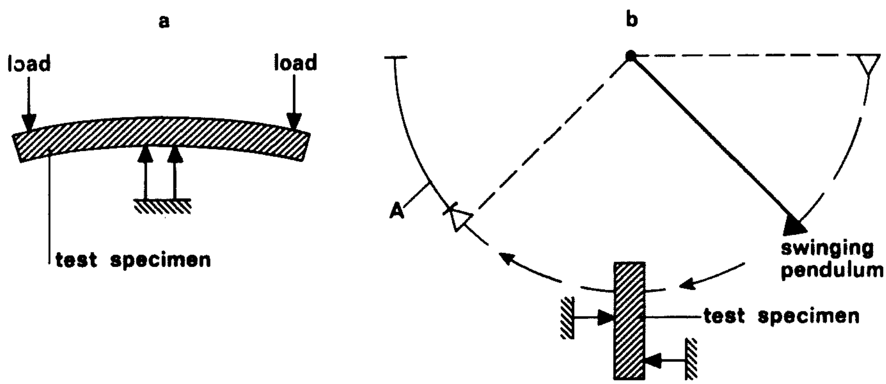

Figure 4 a. Principle of flexural strength test; the specimen is loaded until fracture occurs.

Figure 4 b. Principle of impact strength test; arc-length A is proportional to energy absorbed by breaking of the specimen.

Table 2. Flexural strength and impact strength.*

Cement	Flexural strength ($kg\ cm^{-2}$)	Impact strength (kg cm)
Simplex	839 (± 15)	3.4 (± 0.2)
Palacos R	863 (± 15)	4.1 (± 0.3)
Palacos K	913 (± 25)	3.9 (± 0.3)
CMW-0	1069 (± 39)	5.9 (± 0.6)
CMW-1	819 (± 30)	3.3 (± 0.5)
CMW-2	786 (± 24)	2.8 (± 0.3)
CMW-0 + 4.2 % erythrocyne	860 (± 22)	4.7 (± 0.3)
CMW-2 + 4.2 % erythrocyne	765 (± 17)	3.8 (± 0.3)

* Values in parentheses represent the 95 per cent confidence interval of the mean.

ties (resp. CMW-1; CMW-2; CMW-0 + erythrocyne and CMW-2 + erythrocyne). From the results in Table 2 it follows that these additives decrease the values of flexural strength and impact strength to the level of the other cements which are premixed with radiopacifiers, such as $BaSO_4$ or ZrO_2 (Simplex, Palacos R) or even pigments only (Palacos K), by the manufacturer. These results concern, as already mentioned, non-porous specimens; however, in clinical practice, the cement in the femur or acetabular cavity will be highly porous, due to the large temperature rise during hardening and to the clinical technique of mixing. Therefore it is important to determine the influence of this porosity on mechanical properties.

The processing of plate material (as used in the other experiments to obtain specimens) with a porosity that resembles the clinical situation appeared to be impossible. In order to obtain a representative porosity, glass tubes with a square cross section (2×2 cm) were filled with the cement mix and immersed in water at 37° C until curing had taken place. After curing, the cement rod was removed from the glass tube and test specimens were obtained by sawing the rod on a circular saw. Microscopic examination showed the porosity pattern to be comparable with the porosity in a piece of cement that was obtained from material removed out of the femur of a patient who needed revision of a loosened hip posthesis Figures 5 a and 5 b, respectively). As these specimens could not be compared with the specimen obtained from plate material because of the different processing technique, a similar glass tube was filled with carefully mixed cement but in this case the cement was cured under two atmospheres air pressure in a

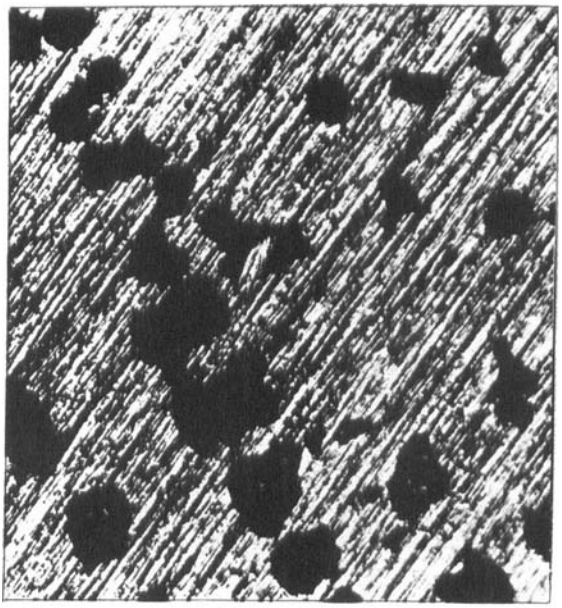

Figure 5 a. Porosity pattern in bone cement (Palacos) cured under laboratory conditions at normal pressure. (Magnification 80 ×; incident light.)

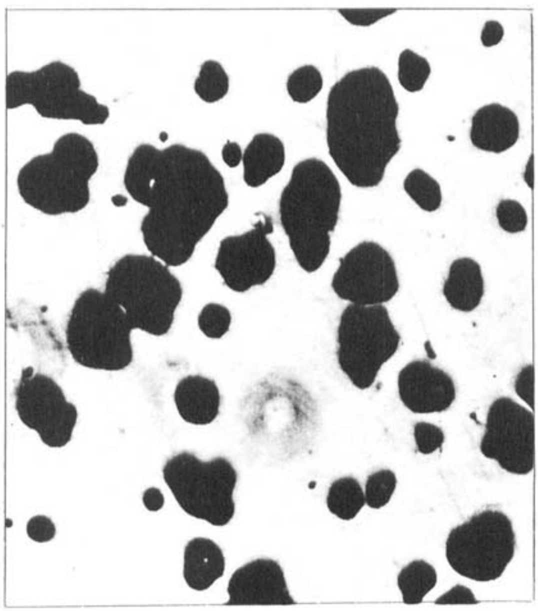

Figure 5 b. Porosity pattern in bone cement (Palacos) as obtained from revision operation. (Magnification 80 ×; incident light.)

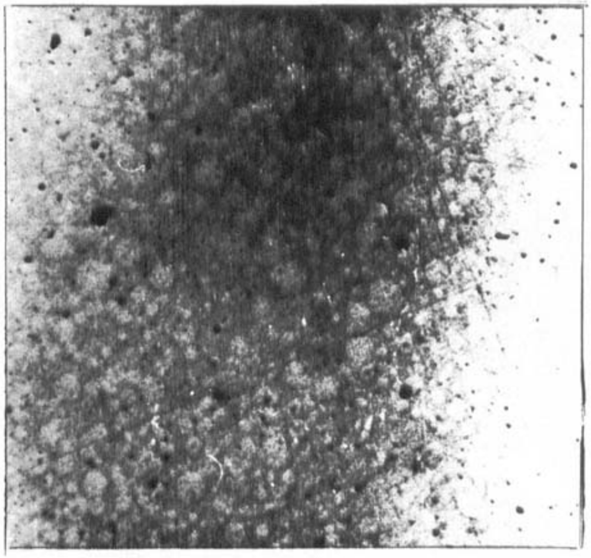

Figure 5 c. Bone cement cured under 2 atm. pressure in laboratory. (Magnification 80 ×; incident light.)

high pressure vessel. The high pressure diminishes the volatility and, in fact, increases the boiling point of the monomer (100° C at normal pressure) so that the temperature rise during curing causes far less porosity (Figure 5 c). Specimens were obtained from this "non-porous" rod in a similar way as in the case of the porous specimens. Flexural strength and impact strength could now be compared and the results for some cements are presented in Table 3. These results clearly show the not unexpected negative influence of porosity on these mechanical properties to which the already noticed disadvantageous effect of heterogeneous additives is added.

Table 3. Influence of porosity on flexural strength and impact strength.*

Cement	Flexural strength ($kg\ cm^{-2}$)	Impact strength (kg cm)
CMW–0 (non-porous)	810 (± 65)	3.9 (± 0.4)
CMW–0 (porous)	579 (± 39)	1.8 (± 0.2)
CMW–2+4.2 % erythrocyne (porous)	356 (± 44)	1.7 (± 0.3)
Palacos R (non-porous)	751 (± 38)	3.5 (± 0.3)
Palacos R (porous)	442 (±100)	2.5 (± 0.4)

* Values in parentheses are 95 per cent confidence intervals.

DISCUSSION

In the non-cured state, i.e. immediately after mixing of powder and liquid, the three bone cements tested showed specific differences in the values for curing time and consistency. As mentioned before these handling properties are closely related and depend *inter alia* on the concentrations of initiators, accelerators and stabilizers. These concentrations have been chosen by the manufacturer. Other factors which will influence curing time, and thus consistency, are volume of the cement mix, powder to liquid ratio and ambient temperature, but these factors are, within practical limits, unlikely to even out the differences between the brands as revealed by our more standardized experiments. Accordingly, each operator can determine which curing time and consistency suits him best. It is likely, however, that too short a curing time and a too rapidly rising consistency will not be favourable for homogeneous filling of the femoral cavity. The mechanical properties of the various cured bone cements do not appear to differ very much, at least not enough to base a preference for a specific brand thereon. Besides that, the lack of information concerning the required minimum or optimum levels of mechanical properties of bone cements could only lead to a "the-stronger-the-better" philosophy, if there had been essential differences between the mechanical properties of the tested cements. A marked phenomenon is the disadvantageous influence of heterogeneous additives such as radiopacifiers and antibiotics particularly on flexural strength and impact strength (Table 2). This could be an argument in the dispute about whether these additives are strictly necessary or not, if it were not for the deleterious effect of porosity on mechanical properties (Table 3). The overall effect of this porosity—which is inevitable with current cements and operating techniques—in combination with heterogeneous additives is a decrease of flexural strength and impact strength of about 50 per cent as compared to non-porous cement without additives (CMW-0). Other reports (Lautenschlager 1973) show the same adverse effect of porosity on compressive strength and diametral tensile strength. Porosity in bone cements is caused mainly by two factors: the enclosure of air during mixing of powder and liquid and the volatility of the monomer and of water from body fluids with which the cement is contaminated during operation. When the temperature in the cement rises during curing, these low boiling point components will form more or less finely dispersed gas bubbles within the cement mass. Due to the formation of gas bubbles and the thermal expansion of enclosed air the cement will expand 3–5 per cent by volume (Charnley 1970). Here we have a dilemma: this "foaming" effect will undoubtedly favour a good adaptation of the cement to the cavity wall, which is necessary for a stable fixation of the prosthesis. On the other hand we see a dramatic decrease of mechanical properties due to the resultant porosity. Within the framework of current clinical techniques it seems rather difficult to optimize for these tendencies which affect the quality of the arthroplasty in opposite directions. It seems to be necessary for clinical techniques to be developed in which the porosity of the cement can be controlled.

Again the need for information about the required level of relevant mechanical properties is felt. The extensive literature concerning follow-up cases of total hip replacements does not reveal the extent or the frequency of mechanical failure of the cement in relation to, e.g. loosening of the prosthesis.

It is likely that the strength of the cement will play a role, but also that the proportional limit, flow properties and modulus of elasticity will have a definite influence on the stability of artificial joint constructions.

SUMMARY

Properties of acrylic bone cements during and after curing were determined for three brands of bone cement. Curing time and consistency were chosen for the characterization of the handling and working behaviour of these materials. The performance of bone cements after curing may be related amongst other things to the following properties: water resorption, solubility/disintegration, flexural modulus of elasticity, yield stress, proportional limit, flexural strength and impact strength. Methods to determine these handling and material properties are described.

The influence of radiopacifying and antibiotic additives on these properties is evaluated as well as the influence of porosity on flexural strength and impact strength.

The results indicate that considerable differences in the handling properties occur. The material properties of the three brands tested do not show marked differences. Radiopacifying and antibiotic additives appear to have a negative effect on material properties; the effect of porosity as it develops during curing under simulated clinical conditions is more pronounced.

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REFERENCES

Charnley, J. (1970) *Acrylic cement in orthopaedic surgery.* E. S. Livingstone Ed. London.

Lautenschlager, E. P. (1973) Physical characteristics of setting of acrylic bone cement. Paper given at 5th International Biomaterials Symposium, Clemson, U.S.A.

Slooff, T. J. J. H. (1969) A cement syringe. *Acta orthop. belg.* **35**, 1012.

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