



# DMTA method in determining strength parameters of acrylic cements

P. Postawa <sup>a,\*</sup>, A. Szarek <sup>b</sup>, J. Koszkuł <sup>a</sup>

<sup>a</sup> Department of Polymer Processing and Production Management, Czestochowa University of Technology, ul. Armii Krajowej 19C, 42-200 Czestochowa, Poland

<sup>b</sup> Quality Engineering and Bioengineering, Institute of Metal Working and Forming, Czestochowa University of Technology, ul. Armii Krajowej 21, 42-200 Czestochowa, Poland

\* Corresponding author: E-mail address: postawa@kpts.pcz.czyst.pl

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## ABSTRACT

**Purpose:** The paper presents the results of investigations of dynamic properties for bone cement with different fillers by means of DMTA method. Addition of any substance causes the change in mechanical properties. Pure PALAMED<sup>®</sup> material and material filled with four different fillers have been analysed.

**Design/methodology/approach:** One of the methods of thermal analysis for polymeric materials has been used for investigations. DMTA method is based on the analysis of the signal (reaction) from the deformed material under particular conditions, at the changeable ambient temperature as well as vibrations frequency and amplitude. DMTA thermograms give information on change in storage modulus  $E'$  and the mechanical loss factor  $tg\delta$ , which is responsible for dissipation of energy during deformation. Pure cement as well as the cement filled with: BiO-OSS and PORESORB bone graft substitute materials,  $Al_2O_3$  ceramic material and powdered animal bones. Method of specimen preparation and proportion in which the specimens were prepared are presented in the text.

**Practical implications:** As it results from the literature analysis, no investigations of such a wide group of fillers, both organic and inorganic have been carried out yet. The investigations enabled the storage modulus and  $tg\delta$  to be determined for each of the prepared materials, thus to indicate the material whose properties enable this material to be used in further alloplasty surgeries for hip joint. Practical application of the results of the investigations described in this paper will be possible after long and comprehensive clinical trials.

**Originality/value:** Original value of this paper are the results of tests since such an analysis has never been conducted by scientific environment working on this subject.

**Keywords:** Methodology of research; Biomaterials; Mechanical properties; DMTA method

## METHODOLOGY OF RESEARCH, ANALYSIS AND MODELLING

### 1. Introduction

Polymethylmethacrylate (PMMA) or related polymers (e.g. polymethylmethacrylate and PMMA-PMA/ PMMA) have been used almost 40 years as bone cements to fix orthopaedic implants to the bone [1]. Of the total hip joint replacements, the cemented fixation method was mostly adopted owing to offer the immediate stability from cement-stem and cement-bone bonding interface after implant surgery. However, clinical studies also reported that

the cemented hip prostheses failed to function properly due to the loosening of fixation after long-term use [2,3,4,5].

The clinical results for total cemented and uncemented hip joint replacement deteriorate in the course of time due to different reasons. Most of the failures are the results of the aseptic loosening i.e. slow, but progressive process, often coexisting with the bone defect. The fixation or attachment of artificial prostheses to hard tissue, continues to be a major area of interest in orthopaedic. The standard method of prosthesis fixation is to use an acrylic cement composed primarily of polymethyl

methacrylate (PMMA) [6,7]. Adhesion of bone cement to bone and the prosthesis may be critical to solving this problem [8-9]. The finite element stress analysis technique can be used to optimise both design and materials selection in many load-bearing components.

Three-dimensional finite element models for a femur implanted with a cemented prosthesis were constructed with a representative physiological loading condition. The effect of prosthesis Young's modulus and cement Young's modulus on stresses in the prosthesis stem and cement layer was studied [10].

Modification of bone cement in order to increase its strength, biocompatibility and making hypertrophy of PMMA by the bone is one of the major areas of research. [11, 12, 13,14,15,16,17]

## 2. Materials and methods

PALAMED<sup>®</sup> 40 acrylic cement (PMMA) used for fixation of the prosthesis in the bone have been selected for the investigation. Cubicoid samples with dimensions of 3x3x50 [mm] composed of pure bone cement with the chemical constitution given by the manufacturer with addition of fillers. As a filling substances, the following materials were used:

- BiO-OSS with granularity of 0,25-1 [mm],
- Poresorb with granularity of 0,16-0,3 [mm],
- Al<sub>2</sub>O<sub>3</sub> ceramic material with granularity of 10-20 [μm],
- powdered animal bone with granularity of 10-30 [μm].

PMMA were manually formed in special-purpose mould after previous application of anti-adhesion agent, with the same polymerisation conditions for all the specimens. For the modified cement the concentration of fillers did not exceed 5%.

PALAMED<sup>®</sup> 40 cement was modified by addition of abovementioned fillers. The purpose of addition of bone graft substitute materials was to simulate the structure of cement to which bone filings got as a result of incomplete washing of the marrow cavity during alloplasty. Ceramic material was added, however, in order to increase the strength and biocompatibility of the applied PMMA.

Dynamic investigations of mechanical properties by means of DMTA method (Dynamic Mechanical Thermal Analysis) are conducted in order to determine the mechanical characteristics of the bone cement as a material by means of various fillers. A wide range of temperatures was assumed for investigations from -50°C to 120°C. This will enable to assess the changes in dynamic properties of investigated materials not only in narrow range of temperatures (37-41°C), but it will enable to track the differences in morphological structure, which could be revealed only outside the range of usable temperatures.

### 2.1. Equipment and conditions of measurements

The tests were performed by means of *DMA 242* device by NETZSCH<sup>®</sup> (Fig. 1a) with the holder for three-point free bending in the form of a beam. The measured part with a sample has been presented in Fig. 1b.

Fig. 1b presents a measurement device with a specimen, mounted and loaded. For the specimen in the sample the

vibrations with different frequency and fixed amplitude were applied while heating the specimen. On the basis of the value of force and deformation (read by means of extensometric sensors in-built in the device), with consideration of the specimen dimensions, the value of the preservation modulus  $E'$  and loss modulus  $E''$  as well as  $\tan\delta$  are calculated. Next, the obtained results are presented in the form of chart of changes in the abovementioned values as a temperature function.

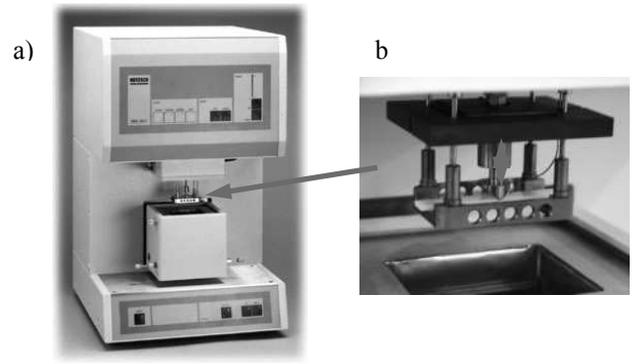


Fig. 1. DMA 242 device by NETZSCH, a) general overview; b) specimen overview

## 3. Results of DMTA methods research

The results of DMTA tests have been presented in the form of two charts. First chart (Fig. 2) presents a change in storage modulus  $E'$  as a function of temperature change, second chart presents changes in mechanical loss factor  $\tan\delta$  as a function of temperature change (Fig. 3). In the whole temperature range from -50°C to 120°C, the highest value of  $E'$ , thus best mechanical properties (strength) are presented by the cement filled with powdered animal bone. The mechanical characteristics for other bone graft substitute materials were slightly below this value. All the specimens filled with bone graft substitute materials caused improvement in mechanical properties in relation to the pure, not modified PALAMED 40 material.

Deterioration of mechanical properties by addition of filler in the form of Al<sub>2</sub>O<sub>3</sub> powder (ceramics). Storage modulus  $E'$  for the cement with this filler is below the value for pure cement. Under clinic conditions, where the temperature is within 37-41°C, these results are similar to those described in the text above. Mean change in  $E'$  modulus for all the investigated specimens amounts ca. 250MPa, whereas the highest difference has been observed for the cement with ceramic filler, ca. 300MPa.

After exceeding of 80°C all the values of storage modulus  $E'$  decrease sharply reaching the value around 1000MPa – here the plastic deformation of the investigated specimens take place.

Damping properties of the investigated materials prove gradual increase with the increase of the temperature, up to 70°C. Above this temperature,  $\tan\delta$  shows sharp increase reaching maximal value at 100°C, mean value ca. 0.45. This proves very good damping properties, however, after correlation with the

results of changes in  $E'$  modulus the specimens show high plasticity at this temperature.

In the usable range under clinic conditions, good damping properties are presented by cements with the following fillers: BiO-OSS bone graft substitute,  $Al_2O_3$  ceramic material and powdered animal bones. Tgd for these specimens changes within 0.15. For other materials (Porosorb and pure PMMA) tgd is within 0.12

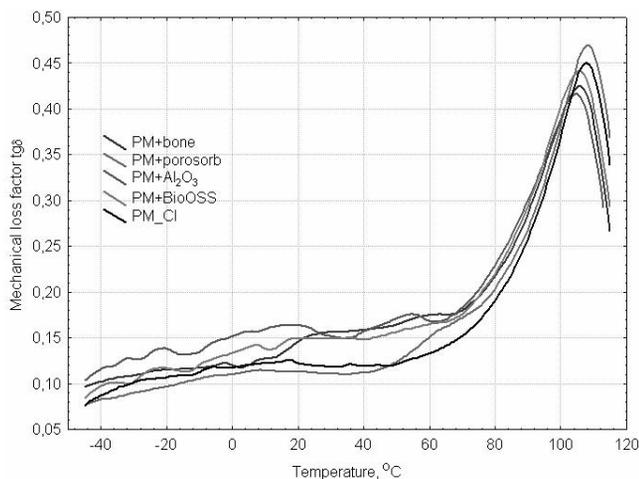


Fig. 2. Changes in storage modulus  $E'$  as a function of temperature PALAMED 40 acrylic cement

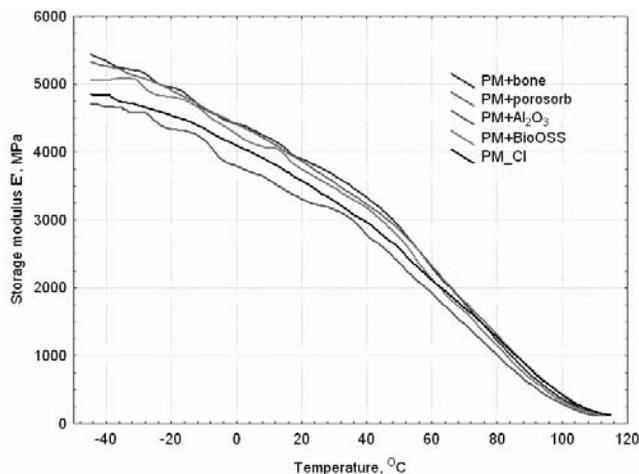


Fig. 3. Changes in mechanical loss factor  $tg\delta$  as a function of temperature PALAMED 40 acrylic cement

#### 4. Conclusions

Presented results of the investigations of dynamic properties of PALAMED 40 with bone graft substitute material and ceramic fillers gave the answer to the question of to which degree the addition of these materials causes changes in mechanical

properties. Strength properties, apart from damping properties are of major importance for a success of hip joint alloplasty and further exploitation of the implant. The most frequent reason for inflammations and loosening of stems is insufficient bone-cement interface.

The investigations have made it possible to prove that addition of bone graft substitute materials caused improvement in mechanical properties and most of dynamic properties. Only in one case, i.e. by using cement with Poresorb filler the vibration damping properties deteriorated.

#### References

- [1] J. Charnley, Anchorage of femoral head prosthesis to the shaft of the femur, *Journal of Bone and Joint Surgery* 42-B (1960) 28-30.
- [2] M. Jasty, W.J. Maloney, C.R. Bradgon, D. O'Conner, T. Harie, W.H. Harris, Histomorphological studies of the long-term skeletal responses to well fixed cemented femoral components, *Journal of Bone and Joint Surgery* 72A/8 (1990) 1220-1229.
- [3] M. Jasty, W.J. Maloney, C.R. Bradgon, D. O'Conner, T. Harie, W.H. Harris, The initiation of failure in cemented femoral components of hip arthroplasties, *Journal of Bone and Joint Surgery* 73B/4 (1991) 551-558.
- [4] L.D.T. Topoleski, A fractographic analysis of in vivo PMMA bone cement failure mechanism, *Journal of Biomedical Materials Research* 24/2 (1990) 135-154.
- [5] T.P. Culleton, P.J. Prendergast, D. Taylor, Fatigue failure in the cement mantle of an artificial hip joint, *Clinical Materials* 12/2 (1993) 95-102.
- [6] A. Gnatowski, O. Suberlak, P. Postawa, Functional materials based on PA6/PVP blends, *Journal of Achievements in Materials and Manufacturing Engineering* 18 (2006) 91-94.
- [7] K. Ishihara, Hard tissue compatible polymers, Biomedical applications of polymeric materials, Boca Raton: CRC (1993) 143-62.
- [8] M. Sadao, F. Kohtaro, I. Kazuhiko, N. Nobuo, Performance of adhesive bone cement containing hydroxyapatite particles. *Biomaterials* 19 (1998) 1601-1606.
- [9] N. Nakabayashi, E. Masuhara, E. Mochida, I. Ohmori, Development of adhesive pit and fissure sealants using a MMA resin initiated by a tri-n-butyl borane derivative, *Journal of Bone and Joint Surgery* 12 (1978) 149-65.
- [10] H.F. El-Sheikh, B.J. MacDonald, M.S.J. Hashmi, Material selection in the design of the femoral component of cemented total hip replacement. *Journal of Materials Processing Technology* 122 (2002) 309-317.
- [11] G. Lewis, Relative roles of cement molecular weight and mixing method on the fatigue performance of acrylic bone cement, simplex P versus osteopal. *Journal of Biomedical Materials Research* 53 (2000) 119-30.
- [12] K. Kawagoe, M. Saito, T. Shibuya, T. Nakashima, K. Hino, H. Yoshikawa, Augmentation of cancellous screw fixation with hydroxyapatite composite resin (CAP) in vivo. *Journal of Biomedical Materials Research* 53 (2000) 678-84.

- [13] S.Y. Kwon, Y.S. Kim, Y.K. Woo, S.S. Kim, J.B. Park, Hydroxyapatite impregnated bone cement: in vitro and in vivo studies. *Biomedical Material Engineering* 7 (1997) 129-40.
- [14] A. Szarek, J. Szyrowski, Strength differences of some acrylic cements. *Engineering of biomaterials* 45 (2005) 24-29.
- [15] A. Mervi, A. Puska, Anne K. Kokkari, Timo O. Narhi, Pekka K. Vallittu, Mechanical properties of oligomer-modified acrylic bone cement, *Biomaterials* 24 (2003) 417-425.
- [16] P. Postawa, D. Kwiatkowski, Residual stress distribution in injection molded parts. *Journal of Achievements in Materials and Manufacturing Engineering* 18 (2006) 171-174.
- [17] E. Bociaga, T. Jaruga, Visualization of melt flow lines in injection moulding, *Journal of Achievements in Materials and Manufacturing Engineering* 17 (2006) 331-334.