Influence of the Bone Cements Processing on the Mechanical Properties in Cranioplasty

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The aim of this study is to observe the time of mixing influence on the properties of commercially available PMMA bone cement, widely used in cranioplasty. The studied bone cement is provided in the form of a solid powder (the copolymer) and a liquid monomer. The increase of the mixing phase duration and the use of two mixing methods (manual and mechanical) effect on surface and mechanical characteristics were studied. The samples were prepared as if in the operation room. Surface characteristics were studied by means of contact angle measurements, morphology by scanning electron microscopy (SEM) and mechanical characteristics determined by flexural tests in a three point bending configuration. The conclusion of this study is that by using a mechanical mixing method and increasing mixing time higher flexural strength can be achieved by reducing pore content within bone cement.

Keywords: bone cement, PMMA, flexural strength, surface wetting

Current uses for poly(methyl methacrylate) or PMMA range from bone cements in orthopaedics (for implant fixation), in dentistry (base for dental prosthesis) and for cranial reconstruction. In clinical practice, many surgical interventions need the support given by the synthetic biomaterials. Also, the various aspects related to the interface between synthetic biomaterials and human tissue appear to be interesting not just for neurosurgery [1,2], but also for many clinical specializations like orthopedics [3-7], dentistry [8-10], abdominal surgery [11-14], gynecology [15-18], cardiovascular surgery [19,20], and ophthalmology [21].

Cranioplasty is the surgical repair procedure used to treat and repair acquired defects or congential deformities of the cranium [22]. Its main purpose is to reconstruct or to replace the damaged or missing bone tissue for brain protection and aesthetics.

However, cranioplasty can present different complications like excessive inflammation, infection, dislocation, bone resorbtion, convulsions, excessive bleeding quickly turning into haemorrhage and even intracranial hematomas. Many of these complications appear based on the dimension and localization of the defect more than on the type of material used for bone reconstruction [10, 23].

Cranial reconstruction can be performed using various materials: autografts, allografts and distinct biomaterials. Even though using bone grafts from the patient itself seems the best choice, bone resorbtion is one of the main disadvantages of cranioplasties in which autologous bone is used [1, 6, 24]. Therefore, the number of second surgical interventions can be twice as many compared to situations where biomaterials were used.

In clinical practice the synthetic biomaterials used in cranioplasties are titanium, polymethilmethacrylate (PMMA), polyethylene (PE), (PEEK), hydroxyapatite (HA). Of wide use is polymethylmethacrylate (PMMA) since it is inert, non-magnetic, and easy to model, relatively cheap and has adequate mechanical properties [1, 10, 22, 25].

The processing route of PMMA is straightforward: the kit is comprised of a powder and a liquid monomer which is mixed according to a manufacturer established protocol. The mixing can be performed by hand, by centrifugation, vacuum mixing or a mechanical mixing.

Most frequent is manual mixing, where de powder is added to the liquid monomer held in a polypropylene bowl. These components are then stirred, usually for 45-120 s, followed by the so called *waiting phase* where the polymerization proceeds.

The next stage, *the working stage* implies injecting the obtained mixture via a cement gun into a mould or adjusting it, by hand, over the bone flap. In the *hardening or setting stage* the polymerization ends and the cements become hard.

Specific procedures are described by PMMA bone cement manufacturers, but sometimes, in practice, given uncontrollable factors as operating room and patient temperature or even the cement thickness, the prescribed times might be altered: either delayed or sped up, depending on the situation [26].

Since the homogeneity of the cement is crucial for mechanical performance, the aim of this study was to observe mixing time influence over the mechanical and surface characteristics.

Experimental part

The polymer was received as a bi-component kit, a solid powder of poly(methylmethacrylate)(PMMA) and a liquid monomer. The powder contains a benzoyl peroxide (BPO), a radiopaque substance and antibiotics that are released after implantation.

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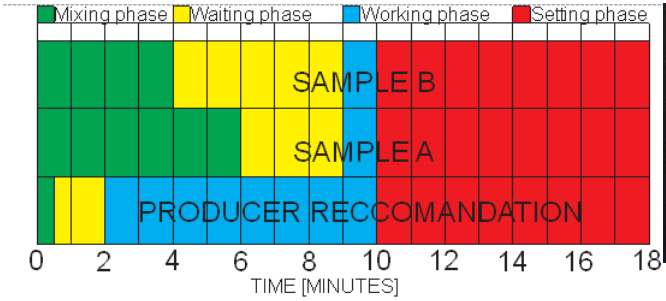

Fig.1. Stage duration for the experimental samples compared to producer recomandation

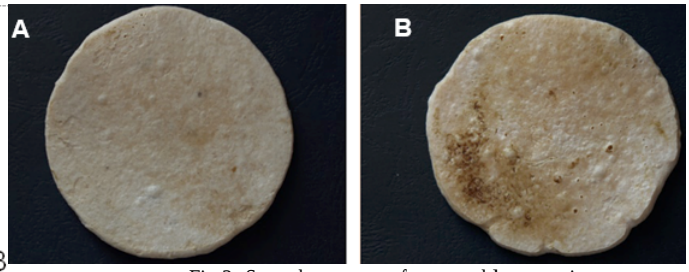

Fig.2. Sample aspect after mould extraction

The powder polymer was mixed with the liquid monomer and an exothermic reaction took place, some laboratory experiments showed that the temperature can reach up to 70°C [27].

These high temperatures can distroy the tissue up until the point where losing the implant becomes inevitable. However, the temperature reached during polymerization depends on the quantity.

As recommended by the producer, the mixing time should be 30 s, followed by a wait time up to 2 min then the polymer has entered the working phase. In the study the mixing phase and, subsequently, waiting phase, working phase were altered, as shown in figure 1.

Two procedures were used to obtain two samples, coded sample A and sample B. Sample A was obtained by mechanical mixing for 6 minutes and the obtained cement injected in a prefabricated mould. Sample B was prepared with a lower mixing time, 4 min, using a hand mixing method and placing the polymer, by hand, over a mould.

The samples, as extracted from the mould, are shown in figure 2. A first macroscopic analysis reveals air pockets and pores on both samples. Since sample B was less homogenized than sample A it shows a large number of air pockets and pores as well as a burned region caused by local overheating.

The samples are divided using a metalographic cutter with cooling into specimens with specific dimensions for each test to be performed.

Surface wetting characteristics were determined using the *DSA100 KRUESS/Germany* contact angle measuring system on 20x20x5mm samples which were then used for scanning electron microscopy (SEM) investigations on a *Quanta Inspect F Scanning Electron Microscope - FEI Company/U.S.A.*

The mechanical properties were determined on 70X7x4mm test samples obtained from sample A and 70x7x6mm test samples obtained from sample B. The testing was performed on a *Walter + Bai AG LFV300* universal testing machine using a three point bending configuration.

Results and discussions

SEM study

The scanning electron microscopy investigations were performed to obtain information on component morphology and defects, pores, in this specific example. Using the secondary electron and backscattered electron detectors, the mixing of the signals offer both topographic as well as compositional information.

The secondary electrons are ejected electrons from the shells of the specimen atoms by inelastic scattering interaction with the beam electrons. Since they originate from the surface of the sample, the information obtained is mainly on surface topography.

The backscattered electrons are reflected high energy electrons from the electron beam which interacted with the specimen atoms. Heavier elements will reflect a higher number of electrons than light ones appearing brighter in the image. Using the backscattered electron detector composition information can be gathered.

Merging via software these signals mixed information can be obtained, both on topography as well as composition.

The SEM micrograph shown in figure 3 offers such information.

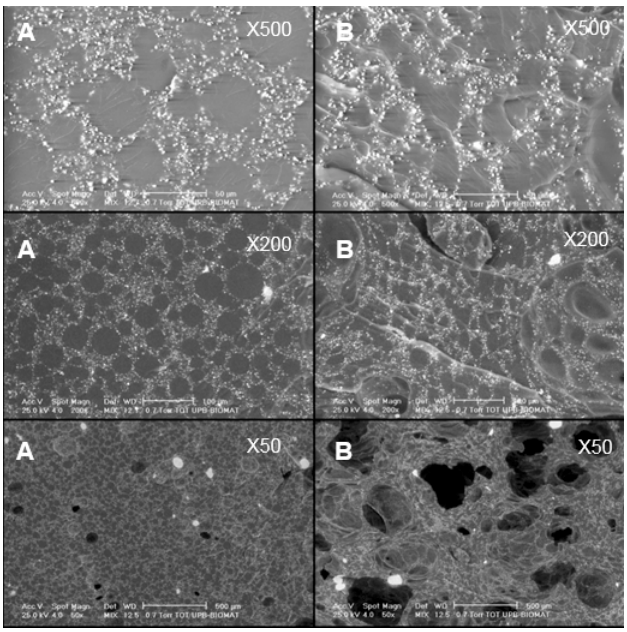

Fig. 3. SEM micrographs of the experimental PMMA samples

At lower magnification, 50X, pores and embedded particles are present within both samples.

Classified by dimension, pores can be macropores, with a diameter greater than 1mm and micropores when their dimension varies between 0.1-1.0mm [28].

The presence of pores is caused by air entrapment during component mixing, transfer and volatile monomer evaporation during curing. In the investigated surface, based upon their linear dimensions, micropores are present [29].

In this case, the pores account for 1.39% of the investigated surface for sample A, while for sample B the pores cover 13.59% of the surface, almost 10 times larger. Increasing mixing time creates the premises for better homogenization of the bone cement.

The pore presence has a double effect: they can act either as stress raisers and crack initiation sites on one hand, and, on the other, they can interact with cracks stopping their propagation.

The general consensus remains the pore number reduction, which was obtained by mechanical mixing.

Increasing magnifications, 200X and 500X, two immiscible components are observed: the PMMA polymer surrounded by a network where antibiotics are embedded in the matrix.

N	Sample	Liquid	CA [°]	IFT(1)	IFT(d)	IFT(p)
1	A	Water	66.4	72.10	19.90	52.20
2	A	Diiodomethane	56.5	50.00	47.40	2.60
3	A	Ethylene glycol	56.1	47.70	30.90	16.80
4	B	Water	66.6	72.10	19.90	52.20
5	B	Diiodomethane	57.0	50.00	47.40	2.60
6	B	Ethylene glycol	54.7	47.70	30.90	16.80
IFT(1) –liquid tension, IFT(d) –dispersive component, IFT(p) - polar component, N - current number						

Table 1
EXPERIMENTAL RESULTS FOR THE MEASURED CONTACT ANGLE

Contact angle measurement

The surface wetting characteristics, either hydrophilic or hydrophobic, can be estimated by contact angle (*CA*) measurements. The contact angle is measured where a liquid - vapour interface meets a solid surface and, upon its value, the surface can be classified as either hydrophilic, when the contact angle is less than 90° or hydrophobic at values exceeding 90°.

For contact angle measurement the *DSA100 KRUESS/ Germany* system was employed using the sessile drop method: a constant volume of water, diiodomethane and ethylene glycol (each liquid represents a different measurement) were placed on the surface and the angle where the liquid - vapour and solid interfaces meet was determined using the Young - Laplace curve fitting method.

Table 1 shows the experimental results.

The experimental results show a contact angle, at both samples, with values lower than 90°, thus PMMA is a hydrophilic polymer. The different mixing times did not affect the surface characteristics of the PMMA, the contact angle values did not modify in a significant manner.

Slight angle variations are caused by surface roughness change, the manual mixing method associated with a lower mixing time produced a slightly less rough surface observed by a low contact angle value decrease.

Flexural tests

The mechanical properties were investigated using a flexural test since the loading mode was considered most similar with the in vivo one.

Flexural tests for PMMA can be performed in accordance to ISO 5833 which uses a 4 point bending configuration and ASTM D790 with a 3 point configuration. In both standards there are specific requirements for sample preparation, but these were not followed, intentionally, in this current study. The aim was to determine the flexural strength of the polymer when it is processed for implantation.

The samples size and testing fixture required a three point bending configuration thus the test was performed using ASTM D790 as guide.

The support span was set at 40 mm and as indenter a steel cylinder with 20mm diameter was used; the test was displacement controlled at a constant crosshead speed of 5mm/min until sample failure occurred.

Three samples from each specimen were tested, the averaged stress -strain curves in flexure are shown in figure 4.

The PMMA behaves in a brittle manner, it fails with no yielding or any sign of plastic deformation.

The flexural strength of sample A is 42.86MPa with a strain at failure of 2.42% while for sample B the flexural strength is 13.49MPa and a strain at failure of 2.22%. The force at failure for sample A was 72N while for sample B 41N.

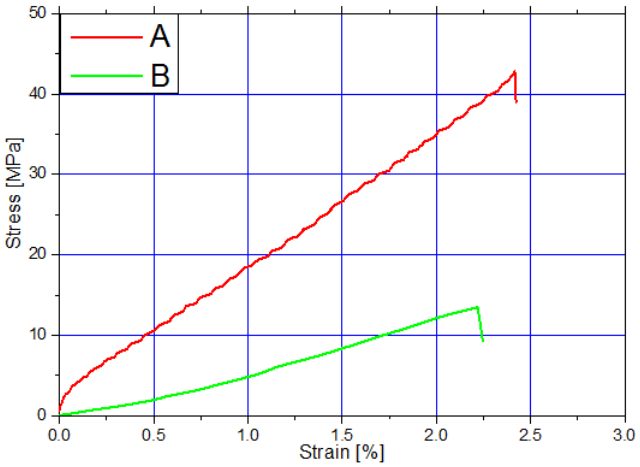

Fig. 4. Averaged stress - strain in flexure for sample A and B

The tangent modulus of elasticity determined as described in ASTM D790 was 1610MPa for sample A and 716MPa for sample B.

The stiffness of sample A was found to be 44.7N/mm and for sample B 37.3N/mm.

Using mechanical mixing and increasing mixing duration has a clear advantage over the hand mixing associated with a lower mixing time. The obtained cement from mechanical mixing was more homogenous, with a low pore content, which translated in superior mechanical characteristics.

According to ISO 5833/2002, Implants for surgery – Acrylic resin cements, the minimum flexural strength is 50MPa, and the flexural modulus should exceed 1800MPa when tested in a four point bending configuration. The loading configuration of a three point bending test will lead to higher flexural strength than in a four point configuration, so it is safe to assume that the bone cement prepared in operating room conditions would not comply with standard requirements because of addition of antibiotics and radiopacifier.

The flexural strength results obtained are strongly influenced by cement formulation, mixing method, curing and curing conditions. A current trend in increasing mechanical characteristics of PMMA bone cement is the addition of various reinforcing phases, most frequent hydroxyapatite particles [30].

Conclusions

In the current stage of the study it was established that, even when the mixing time is increased as reported to producer specifications, using the manual mixing method the bone cement produced this way is prone to be less homogenous and with larger pore content than one obtained by mechanical mixing. Pores remain the major problem and reducing the content requires mixing methods with vacuum. The wetting characteristics of the bone

cement are not altered by mixing time or method, rather surface roughness and, in consequence, a slight increase in contact angle values are to be expected as roughness increases.

The process parameters for the bone cement production are difficult to control, especially in the operation room and the mechanical characteristics are influenced by the cement formulation and process parameters.

The success of the cranial reconstruction depends on the biomaterial characteristics: it should be accepted by the body, here the surface characteristics are of utmost importance; also it should perform its intended role, brain protection and aesthetics, by resisting the loading scenarios which could appear, case where the mechanical characteristics play the major role.

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