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## Original Research Article

# The tension-shear fracture behavior of polymeric bone cement modified with hydroxyapatite nano-particles



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## ABSTRACT

Polymethylmethacrylate (PMMA)-based bone cement is a well-known polymer in the medicine, especially orthopedic. However it has some drawbacks like lack of enough biocompatibility and poor mechanical properties. These problems can be addressed by incorporation of nano-materials. Hydroxyapatite has been proved to enhance biocompatibility of acrylic bone cements. This bioceramic can affect the mechanical properties of polymeric cements as well. In this study, a number of fracture tests were carried out to investigate the influence of nano-hydroxyapatite (HA) on the fracture behavior of acrylic bone cement under combined tension-shear (mixed mode) loading conditions. Semi-circular specimens were prepared by incorporating different amounts of HA powder into the cement matrix. It was found that adding up to 10 wt% HA into the cement causes an increase in the fracture toughness of PMMA/HA nano-composite in all modes. However, pure cement exhibited the greatest fracture resistance among all samples. Moreover, the comparison between the experimental and theoretical results showed that the generalized maximum tangential stress criterion could estimate the experimental data satisfactorily.

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## 1. Introduction

Having numerous applications in arthroplasty and orthopedics, PMMA-based bone cements are used for medicating bone imperfections [1] or filling the cavity between the bone and orthopedic implants. However, low compatibility with the host tissue, toxicity of the monomer, and high heat generation during the reaction are considered as important drawbacks

that may restrict the comprehensive use of the acrylic bone cements [2]. In this regard, incorporation of additive biomaterials into the PMMA-based cements has been recognized as an effective method to overcome these disadvantages. Amongst various types of additives, hydroxyapatite (HA), which constitutes the main mineral part of bones, is widely employed in orthopedics [3,4]. It has been demonstrated that introducing HA nano-particles, as bone-compatible fillers, into PMMA polymer matrix can enhance the biocompatibility of the

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cement [5,6]. One reason for this fact is that adding HA to the cement leads to a porous composite which enhances the interaction of the cement with the osteoblast cells [7]. Furthermore, since homogeneously distributed solid materials in the polymeric matrix absorb some portion of the heat generated during the polymerization reaction, addition of HA can improve the thermal properties of the bone cement [8].

Mechanical properties of bone cement in its main application, i.e. transferring body loads from the implant to the bone, are of utmost importance. Improvements in some mechanical properties of HA-reinforced bone cement such as ultimate compressive strength, compressive elastic modulus [8] and hardness [9,10] were previously reported. Another important mechanical property is fracture toughness which describes the resistance of a material to crack growth under quasi-static loading. In this regard, incorporation of HA particles into the acrylic bone cement was proved to have both positive [11,12] and negative [13] effects on tensile (mode I) fracture toughness. Nevertheless, since in vivo stress state to which bones are subjected is a combination of tensile and shear (mixed mode) loading condition, considering merely tensile loading to study fracture resistance of PMMA/HA nano-composite seems to be inadequate [14]. The aim of this paper is to evaluate the effects of HA nano-particles on the mixed mode fracture toughness of acrylic bone cements. For this purpose, samples of PMMA cement and its composites with different amounts of HA were prepared and subjected to mixed modes I and II loading.

## 2. Materials and methods

### 2.1. Nano-composite preparation

Commercial acrylic bone cement EUROFIX GUN® (Low viscosity, Synimed Company, France) was purchased to prepare the nano-composite cement samples. Acrylic bone cements normally are constituted of liquid and powder phases. The composition of these phases is given in Table 1. Nano-hydroxyapatite (particle size <200 nm, Sigma-Aldrich Chemie-GmbH, Germany) was chosen as the biocompatible nano-filler. Weighted amounts of HA and cement powder phase were mixed using a planetary ball mill (Retsch, Germany) in absolute ethyl alcohol, as a mixing medium, to reach HA weight fractions of 5, 10, and 15% in the composite samples. The ball/powder weight ratio was 10:1 and the powders were milled for 1 h. The mixtures were then dried in the environment for 5 days and were milled under above described conditions for another 1 h. The blend of resultant HA and PMMA powders were hand mixed with the liquid phase by

using powder/monomer ratio of 2:1 according to the manufacturer's instructions. Afterwards, the cement dough was poured into the semi-circular mold cavities of radius 40 mm and pressurized under 60 kPa (the pressure which is exerted clinically) [15] at the temperature of 23 °C. Finally, the samples were separated from the molds and left in ambient conditions for three weeks to reach equilibrium before conducting fracture toughness test.

### 2.2. Fracture toughness test

From a mechanical standpoint, fracture mechanics is a field in which the extension of pre-existing cracks under different loading conditions, such as impact [16] and quasi-static loading [17,18] is dealt with. In this regard, linear elastic fracture mechanics (LEFM) is used to investigate the mechanical behavior of brittle materials in which the size of plastic zone in front of the crack tip is negligible. Since both PMMA and hydroxyapatite bioceramic are classified as brittle materials, the assumption of LEFM for investigating the fracture behavior of these materials as well as their composites seems rational. Three basic modes are implicated in the state of stress in front of the crack tip. Mode I fracture (opening mode) occurs when the crack faces open as a result of symmetric geometry and loading conditions with respect to the crack plane. In-plane sliding of crack faces (perpendicular to the crack front) is described as mode II fracture or shear mode. By changing loading conditions from pure mode I toward pure mode II, the shear stresses along the crack line increase and the tensile stresses decrease. In complex loading conditions like the loads applied to biomaterials in orthopedic applications, cracks experience a combination of opening and shear modes often called mixed mode I-II loading.

The semi-circular bend (SCB) specimen is among favorite specimens for performing mixed mode fracture tests on brittle polymers like PMMA. Fig. 1 shows the test setup and the specimen with an edge crack of angle  $\alpha$ . To prepare the specimens for the fracture test, cracks with the length of  $a = 12$  mm were introduced to all specimens. For this purpose, first a notch with a length of slightly less than 12 mm was generated by the use of a fret blade with the thickness of approximately 0.3 mm. Next, to reach a total crack length of 12 mm, a sharp pre-crack was created by pressing a razor blade. The fracture toughness of cracked samples was determined using three point bend test. In order to achieve this aim, the specimens were placed on two fixed supports, and compressive load was applied from the top at a speed of 1 mm/min. The distance between two bottom supports  $2S$  was considered to be 28 mm.

When  $\alpha$  is zero, the specimen is symmetric relative to the crack line. Therefore, under three-point-bending, the crack experiences only opening (or pure mode I) loading. By increasing the crack angle  $\alpha$  crack faces slide relative to each other and shear stresses become larger in front of the crack tip. At a specific angle  $\alpha_{II}$  (which depends on the geometry parameters  $S$ ,  $a$  and  $R$ ), the crack undergoes pure mode II loading and only shear stresses appear in front of the crack tip along the crack line.

Depending on the ratios of  $S/R$  and  $a/R$ , the angle  $\alpha$  can be such chosen that different combinations of modes I and II are

**Table 1 – Composition of bone cement phases.**

Powder phase	Liquid phase		
Polymethyl methacrylate	52.56 g	Methylmethacrylate	29.64
Barium sulfate	1.44 g	N,N-Dimethyl p-toluidine	18–20 ppm
Benzoyl peroxide	6.00 g	Hydroquinone	0.36 ml

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