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Novel bioactive acrylic-based bone cement reinforced with bioglass nanofillers: chemical and mechanical assessment

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ABSTRACT: This study assesses the utilization of 20 weight % bioglass nano-sized fillers to bioactivate and reinforce the conventional acrylic-based bone cement. The chemical assessment of the novel prepared bone cement was carried out by investigation of calcium and phosphorus ions released by inductively coupled plasma. Moreover, surface examination and chemical elemental analysis were performed by using an environmental scanning electron microscope (SEM) and energy dispersive x-ray spectroscopy (EDX). The mechanical assessment was done by using universal testing machine to determine the compressive strength. The results revealed that the intervention group showed a higher calcium and phosphorus ions concentration and compressive strength than the control group (P-value $\leq$ 0.05). The intervention group showed also apatite layer precipitation on the surface of the specimens which not noticed in the control group.

KEYWORDS:PMMA, acrylic, bone cement, bioglass, bioactivity, nanofillers.

I. INTRODUCTION

Bone cements are frequently used in both orthopedic and dental surgery to repair bone defects. In orthopedic bone cements is a dominant to be used as fixation material in joint arthroplasty, however in dentistry bone cements are commonly used for augmentation of sinus floor, retrograde filling materials and bone defect filling. Currently, two different types of bone cements are available which are acrylic-based or calcium phosphate-based bone cements[1].The main chemical composition of the commercial acrylic bone cement systems is nearly comparable to the traditional formulation of acrylic resin, except for some modifications as the addition of copolymers of polymethyl methacrylate (PMMA) and various comonomers in the liquid to enhance handling properties and minimize polymerization shrinkage[2]

Acrylic-based bone cements have been considered as the gold standard bone cements in anchoring artificial prosthetic appliance to the bone structure. Its widely applications and success have been obtained from their high mechanical properties, easily handling. Moreover, cement reaches its full strength rapidly, thus providing immediate support. However, its main disadvantage is attained from the lack of osseointegration and direct contact with bone and subsequent loosening and failure of the cemented implant or filled space due to debonding at the bone-cement interface[3].

Calcium phosphate-based bone cement has been used as bone repairing material owing to its biocompatibility and osseoinduction ability with bone structure. Though, their inherent brittleness, low compressive strength, higher solubility and long setting time, limits their applications as bone cement[4,5]. Therefore, it could be beneficial to combine the high mechanical feature of acrylic-based bone cement with the bioactive behavior of calcium phosphate-based cement.

Bioglass (SiO_2, Ca, Na_2O, H, and P) are amorphous silicate-based materials which are compatible with the human body, bond to bone and can stimulate new bone growth while dissolving over time. They therefore have the potential to restore diseased or damaged bone to its original state and function[6]. Bioglass (BG) has been used in many medical applications such as coating of implants and as a tissue engineering scaffolds[7,8]. Bioglass have an established excellent capability for apatite formation[7].

The bioactivity behavior of materials comprise the capability of a bioactive material to release calcium and phosphorus ions into the surrounding environment, promoting repair and remineralization of the hard structures[7,9]. Hench et al. was the first to provide the concept of bioactivity to demonstrate the bond developed between the bioactive glass and surrounding hard tissue along the interface. They concluded that the developed strong chemical bonds created by the influence of increased surface reactivity of bioglass[10].

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Nano-sized fillers provide improved properties than conventional micro-sized fillers[11]. Moreover, there is a trend to induce bioactive features to the traditional materials that lack bioactive behavior to promote healing and regeneration to tissues[12]. Thus, the addition of a bioactive ceramic fillers such as bioglass may not only permit creation of strong chemical bond along the cement-bone interface, but also may provide reinforcement and support to the surrounding tissues. Therefore, the aim of the current study is to create a novel bioactive bone cement via improvement of the traditional acrylic-based bone cement by incorporation of nano-sized bioglass fillers, in an attempt to induce bioactivity and enhance their properties to evolving evidence to increase their application in bone tissue engineering

II. METHODOLOGY

A total of 40 specimens (10 specimens per group) (n=10) were tested for the calcium and phosphorus ion release and compressive strength. The control group was prepared from the commercially available acrylic-based bone cement (Cemex ® Isoplastic, Tecres, Italy). While, the Intervention group was prepared from addition 20 weight % of nano-sized BG fillers (45S5 bioglass with an average size 30 nm) prepared by sol gel approach (The lab of Inorganic Chemistry at Suez Canal University, Egypt) into the powder of the traditional acrylic-based bone cement.

The novel bone cement was prepared by incorporation 20 weight % nano-sized bioglass fillers into the powder of the traditional acrylic-based bone cement then the monomer liquid was added to form the coherent plastic mass according to the manufacture instruction. The bone cements were packed into their specific molds in the dough stage. After setting of the mixed bone cements, all specimens were removed from their mold and inspected for any defects, then polished with silicon carbide papers 2000 grit, then visually re-inspected for any defected specimens.

The potential bioactivity was assessed by investigation of the calcium and phosphorus ions release. Ten disc-shaped specimens were prepared for each group from stainless-steel mold (10 mm in diameter, 1 mm in height). After specimen's removal from the mold, it immersed in distilled water (50 cm^3), then stored into an incubator (CBM 2431/V, Italy) for 14 days at 37°C. After that, 10 ml of the immersion solution were withdrawn by a syringe after and filtered using 0.25 or 0.45 µm Millipore filters. The immersion solutions were then assessed to determine the concentrations of calcium and phosphorus ions in mg/L using inductively coupled plasma optical emission spectroscopy (ICP-OES) (Ultima 2 ICP, Horiba, USA).

Surface examination of the prepared specimens and chemical elemental analysis were performed using an environmental scanning electron microscope (SEM) (JSM-5200, JEOL, Tokyo, Japan) and energy dispersive x-ray spectroscopy (EDX) (Oxford Inca Energy 350, Oxford Instruments, Abingdon, UK) , magnification 500X, at a distance of 10 mm, resolution of 3 nm and accelerating voltage 30 kV. After collecting the EDX spectra, automatic identification of elements and element quantification in both weight % and atomic % was carried out. SEM images and corresponding EDX spectra were specifically observed for apatite white patches formation and identification and quantification of elements. Ca/P atomic ratio of the layers formed on specimens was calculated from the EDX results according to the following equation[13–15]:

$$\text{C/P atomic \%} = \frac{\text{Calcium mean atomic \%}}{\text{Phosphorus mean atomic \%}}$$

The compressive strength test was carried out in ambient laboratory conditions (air, at 22 ± 1 °C). Ten cylindrical specimens for each group (6 mm in diameter,12 mm in height) were prepared in special constructed cylindrical Teflon model according to standard specification for acrylic bone cement specified in the ASTM F-451-99 standard[16]. A separating medium was applied to facilitate removal of the specimens from the mold. After setting, specimens were removed from the mold. The ends of the cylindrical specimens were ground and visually inspected for any defect. The compressive strength was determined using universal testing machine **(**Shimadzu Autograph AG-X plus 5 kN, Kyoto, Japan) with a cross head speed (20mm/min) with applied load corresponding to (5KN), the maximum failure load was determined; accordingly the compressive strength was calculated.

Independent sample T-test analysis statistical test was used. The significance level was set at $P \leq 0.05$. All statistical analysis was done with IBM® SPSS® Statistics Version 20 for Windows (SPSS Inc., IBM Corporation; USA).

III. RESULTS

Calcium and phosphorus ion release concentration values after immersion of specimens in distilled water for 14 days are listed in (Table 1). Independent sample T-test results revealed that the intervention group showed a higher statistically significant mean calcium and phosphorus ion concentration value than the control group.

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The EDX elemental analysis of calcium and phosphorus atomic % values of the specimens after immersion of specimens in distilled water for 14 days are listed in (Table 2). A representative SEM micrograph for the control and intervention groups are shown in (Figures 1 and 2).

Independent sample T-test results revealed that the intervention group showed a higher statistically significant mean calcium and phosphorus ion values than the control group.

The qualitative descriptive results obtained from SEM images revealed that the control group showed spherical and irregular shaped beads region. moreover, the micrographs of the intervention group showed dense white patches at the corner of beads forming a coating layer.

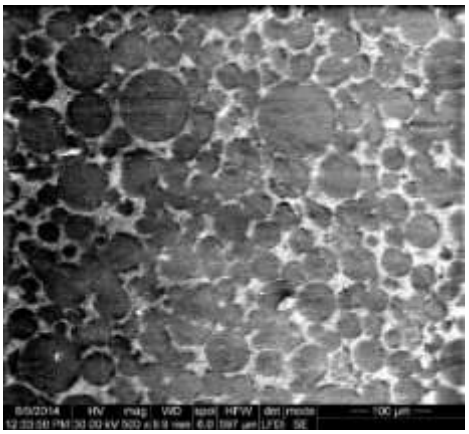

Figure 1. A representative SEM micrograph of the intervention group after 14 days storage showing spherical shaped beads region.

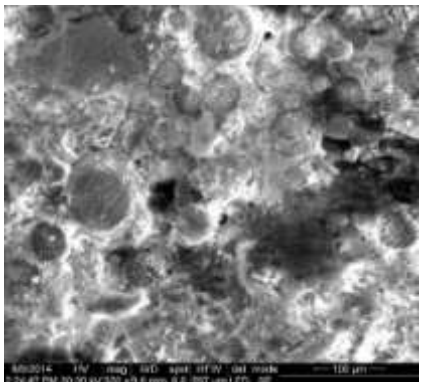

Figure 2. A representative SEM micrograph of the intervention group after 14 days storage showing white patches formation.

EDX chemical elemental analysis results revealed the absence of both calcium and phosphorus elements in control group. In addition, the dark area dark area was associated with an increased amount of carbon element; whereas the light area was associated with an increased amount of barium element. In the other hand, in the intervention group it was noted that the dark area was associated with an increased amount of carbon element and the light area was associated with an increased amount of calcium element.

The mean and standard deviation values of the compressive strength of the specimens are represented in (Table 3). Independent sample T-test results revealed that the intervention group showed the higher statistically significant mean compressive strength value (98.1 MPa) compared the intervention groups (80.7 MPa).

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Table 1. The mean, standard deviation values and results of Independent sample T-test for comparison between calcium and phosphorus ion release concentration values after 14 days storage.

Concentration (Mean, SD)	Intervention group	Control group	P-value
Calcium	42.5 (0.2) [a]	1.6 (0.3) [b]	<0.001*
Phosphorus	31.1 (0. 4) [a]	1.2 (0.5) [b]	<0.001*

*: Significant at P ≤ 0.05, Different superscripts are statistically significantly different.

Table 2. Descriptive statistics values and results of Independent sample T-test for comparison between calcium and phosphorus atomic % valuescalculated from EDX analysis after 14 days storage.

Atomic % (Mean, SD)	Intervention group	Control group	P-value
Calcium	1.92 (0.11) [a]	0.00 (0.00) [b]	0.002*
Phosphorus	1.31 (0.07) [a]	0.00 (0.00) [b]	0.002*

*: Significant at P ≤ 0.05, Different superscripts are statistically significantly different.

Table 3. The mean, standard deviation values and results of Independent sample T-test for comparison between compressive strength**.**

Compressivestrength (Mean, SD)	Intervention group	Control group	P-value
	98.1 (1.9) [a]	80.7 (2.0) [b]	<0.001*

*: Significant at P ≤ 0.05, Different superscripts are statistically significantly different.

IV. DISCUSSION

Over time, acrylic-based bone cement has gained a great acceptance to be utilized as a bone cement. It has been widely used to affix orthopedic implants to host bone and is considered as the gold standard for implant fixation. It acts as a glue; adapting the surface irregularities of the surrounding bone tissue to the surface of the implanted prosthesis[17,18]. Despite its strong mechanical attachment between the implant and bone, the primary problem associated with acrylic-based bone cement is its poor bioactivity. Hence, acrylic-based bone cement bone cement may lead to loosening of the prosthetic appliance with a subsequent prosthetic failure[14,19]. Hence, the aim of the current study was to render acrylic-based bone cement bioactive via the addition of nano-sized bioglass bioactive fillers, to form a composite system.

A biomaterial to bond chemically to bone, it must possess a certain degree of bioactivity. This is measured by the ability of a material to form a calcium-phosphate layer. In-vitro ion release tests are carried out to analyze the bioactivity of the filler. First, in-vitro ionic dissolution of calcium and phosphorus ions is performed. This is followed by evaluation of the ability of the material to form an apatite layer. The degree of apatite formation on the surface of a material in-vitro can be correlated to the degree of in-vivo bone bioactivity of the material[20,21].

ICP analysis is the most used test to assess the bioactivity of a material through quantifying the changes in ion concentration in the solution. The highest ionic release of calcium and phosphorus by intervention group (contained bioglass filler) may be contributed to the release of calcium and phosphorus ions from bioactive glass compared to that of control group which did not contain any fillers[22].

SEM and EDX analysis that were carried out in order to assess the qualitative descriptive changes in surface morphology of the specimens and detection of any apatite precipitate. The

spherical and irregular shaped beads region in the control group may be represent the prepolymerized PMMA beads region which surrounded by in-situ PMMA. While, the white patches in the intervention group may be represent the calcium phosphate dense precipitate on the corner of PMMA beads which forming an apatite coating layer[22].

Nano-sized bioceramic particles serving as a reinforcing agent that could be enhance the mechanical and biological properties of the implants[23]. The improved compressive feature of the intervention group may be due to the reinforcement effect of the strong nano-sized bioglass ceramic fillers that the control group which composed only from a weak polymer[23,24].

V. CONCLUSION

Bioactivation of conventional acrylic-based bone cement by addition of nano-sized bioglass filler give rise to calcium and phosphorus ions release, apatite layer deposition which may be valuable for chemical bonding to bone structure with enhancement compressive strength of the set bone cement.

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