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Comparison of the diametral tensile strength of bone cement based on carbonate apatite between micron and nano particles calcium carbonate as a precursor

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ABSTRACT

INTRODUCTION: Bone cement is inorganic material which can be used for bone substitute materials. One of the bone cement which has been attracted much attention in orthopedic and dental fields is apatite cement. As a result of apatite cement could transform to carbonate apatite (CO₃Ap) after implanted in the body, high bone replacement ability could be developed. The current study about diametral tensile strength (DTS) value of bone cement using calcium carbonate (CaCO₃) as one of the component of CO₃Ap cement in limited to submicron particles. There have been no data using CaCO₃ reduced into nano particles. Meanwhile, the studies about reduction of CaCO₃ particles into nano particles have been found by the precipitation and ultrafine grinding. Objective: The aim of this study is to investigate the DTS value of bone cement based on CO₃Ap between micron and nano particles using CaCO₃ as a precursor. Materials and methods: The powder phase of micron particles of CaCO₃ or nano particles of CaCO₃ which was milled by beads mill combined with dicalcium phosphate anhydrous (CaHPO₄) was mixed with 1 mol/L of disodium hydrogen phosphate (Na₂HPO₄) solution in 0.5 of liquid to powder ratio. The paste was packed into a split Teflon mold, covered with glass slide and kept at 37°C and 100% relative humidity for 24 hours. Results: The result showed that the diametral tensile strength of the set CO₃Ap cement using micron particles of CaCO₃ was 3,7787 MPa and nano particles of CaCO₃ was 2,4013 MPa. The data was analyzed statistically with t independent test (α=0.05) which showed that the DTS between micron and nano particles of CaCO₃ was statistically significant. Conclusion: In conclusion, the bone cement based on CO₃Ap using micron particles of CaCO₃ has higher DTS value than bone cement based on CO₃Ap with nano particles of CaCO₃.

Keywords: Diametral tensile strength, bone cement, carbonate apatite, calcium carbonate
INTRODUCTION

Bone is one of important parts in our body that support our bodies and protect vital organs such as heart, lungs, etc. Bone has limitation for self repair. When large defect bone occurs, so the repair processed need an alternative materials as a bone graft [1]. Autograft is one of the bone grafting materials which is still considered as the “golden standard” compared to other grafting materials since it shows high remodelling process performance. This material is harvested from healthy parts of the bones of the same patient. However, the availability of the healthy bone in our body is limited and this may involve massive blood loss, sepsis, and also pain [1, 2]. Therefore, the use of synthetic graft can be used as an alternative material for bone substitute [3].

Bone cement is a synthetic bone graft material (alloplastic) which can be used for bone substitute materials [4]. One of the bone cement which has been attracted much attention in orthopedic and dental fields is apatite cement (AC) [5]. The AC could transform to carbonate apatite (CO\textsubscript{3}Ap) when implanted in the body and high bone replacement ability could be developed. Another advantages possessed by AC lies in its ability to show good osteoconductivity and excellent tissue response [6].

The candidates of starting material for fabrication of CO\textsubscript{3}Ap cement are calcium carbonate (CaCO\textsubscript{3}) and dicalcium phosphate anhydrous (CaHPO\textsubscript{4}) [6]. Cahyanto et al. reported that CO\textsubscript{3}Ap cement consisted of one of the polymorph of CaCO\textsubscript{3} which is vaterite that has the smallest particle size with the highest diametral tensile strength (DTS) [7]. However, the current study about DTS value of bone cement using CaCO\textsubscript{3} as one of the component of CO\textsubscript{3}Ap cement was limited. Furthermore, there was no data using CaCO\textsubscript{3} reduced into nanoparticles as a precursor for CO\textsubscript{3}Ap cement. Meanwhile, the study about reduction of CaCO\textsubscript{3} particles into nanoparticles has been found by the precipitation and ultrafine grinding [8]. The aim of this study was to investigate the DTS value of bone cement based on CO\textsubscript{3}Ap between micron and nanoparticles using CaCO\textsubscript{3} as a precursor.

METHODS

This research was conducted by CO\textsubscript{3}Ap based bone cement using micron and nanoparticles of CaCO\textsubscript{3}. The nanoparticles of CaCO\textsubscript{3} were derived on the basis of previous report [8]. In brief, 1.15 mL of Tween 80 as the surfactant was mixed into 568.1 mL aquadest and 5.75 g of CaCO\textsubscript{3} using magnetic stirrer. The grinding was conducted by beads mill for 3 hours at the slurry concentration of 5%, bead size of 300 μm, and the rotor speed 3000 rpm. The obtained particles were collected by dried at 40°C for 24 hours. Finally, each micron particles of CaCO\textsubscript{3} and nanoparticles of CaCO\textsubscript{3} were mixed with CaHPO\textsubscript{4} homogeneously on weight powder ratio 40:60 to obtain CO\textsubscript{3}Ap powder.

Powder phase of CaCO\textsubscript{3} combined CaHPO\textsubscript{4} were mixed with liquid phase of 1 mol/L Na\textsubscript{3}HPO\textsubscript{4} using a spatula at a L/P ratio 0.5 until it became a paste. The paste was packed into Teflon mold (6 mm in diameter x 3 mm in height). Both ends of the mold was covered
with a glass slides and clamped by a metal clip. The molds were placed inside a plastic container containing distilled water to maintain 100% relative humidity. Finally, the plastic container was placed into an incubator and kept at 37˚C for 24 hours. Upon completion of the treatment times, the samples were removed from the mold and immersed in the 99% ethanol for 3 minutes then dried in the oven 80˚C for 3 hours.

The mechanical strength was examined in terms of diametral tensile strength (DTS). The samples were crushed using universal testing machine (Lloyd with Nexygen Plus material test and analysis software, Florida, USA) at 1 mm/min crosshead speed. DTS values were taken from an average five samples. The data has been collected, analysed using t independent test to determine significances.

RESULTS

Fig. 1. DTS values of CO\textsubscript{3}Ap cement consisted of micron particles of CaCO\textsubscript{3} or nanoparticles of CaCO\textsubscript{3} and CaHPO\textsubscript{4} mixed with 1 mol/L Na\textsubscript{2}HPO\textsubscript{4} solution treated for 24 hours. At least five samples were measured for DTS. (p<0.05)

Fig. 2. The mean differences of DTS values of CO\textsubscript{3}Ap cement consisted of micron particles of CaCO\textsubscript{3} or nanoparticles of CaCO\textsubscript{3} and CaHPO\textsubscript{4} mixed with 1 mol/L Na\textsubscript{2}HPO\textsubscript{4} solution treated for 24 hours. At least five samples were measured for DTS. (*p<0.05)
Figure 2 summarizes the DTS measurement of micron particles that have higher DTS value compared to nanoparticles sample. T-independent test showed statistically significant (p<0.05). Figure 1 shows the DTS value of set CO$_3$Ap cement consisted of micron particles of CaCO$_3$ or nanoparticles of CaCO$_3$ and CaHPO$_4$ mixed with 1 mol/L Na$_2$HPO$_4$ solution after treatment at 37˚C and 100% relative humidity for 24 hours. DTS value of set CO$_3$Ap cement was 3.78 ± 0.37 MPa for micron particles and 2.40 ± 0.26 MPa for nanoparticles.

DISCUSSION

The CO$_3$Ap cement with similar chemical composition may have different mechanical strengths. It may be due to additional factors such as impurities were present. Other factors which involve associated with specimen preparation and measurement procedures, such as liquid to powder (L/P) ratio. Particle size of CaCO$_3$ may be a major factor to be regulated for higher mechanical strength of CO$_3$Ap cement [7-9]. As a result, since the smaller particle size is known has the highest surface area, therefore bonding between particles will be more firm and reactive.

The low DTS value of CO$_3$Ap based bone cement using nanoparticles of CaCO$_3$ may be due to the poor handling of L/P ratio. The CO$_3$Ap cement which consisted of nanoparticles CaCO$_3$ generated higher viscosity than CO$_3$Ap cement which consisted of micron particles CaCO$_3$. As stated previously, the smaller particle size is known to make the surface area becomes higher; therefore, it might be due to smaller particles which need more liquid to bind the entire surface area. The L/P ratio becomes necessary to have good handling mechanical property [10].

Another factor affecting the low DTS value of CO$_3$Ap cement using CaCO$_3$ particles which had been grinded into nanoparticles was the sanitation of beads mill. The result of CaCO$_3$ particles grinding, which uses beads mill, might be contained impurities derived from previous grinding process. This was shown in the result of grinding powder discoloured to grey. The impurities present might prevent crystal growth and result in slight decrease in mechanical strength [10].

The results of this study showed that the CO$_3$Ap based bone cement using micron particles and nanoparticles of CaCO$_3$ had DTS value relatively low. Therefore, this cement might be used for non-load-bearing applications.

CONCLUSION

The CO$_3$Ap based bone cement using micron particles of CaCO$_3$ had higher DTS value than CO$_3$Ap based bone cement using nanoparticles of CaCO$_3$.

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