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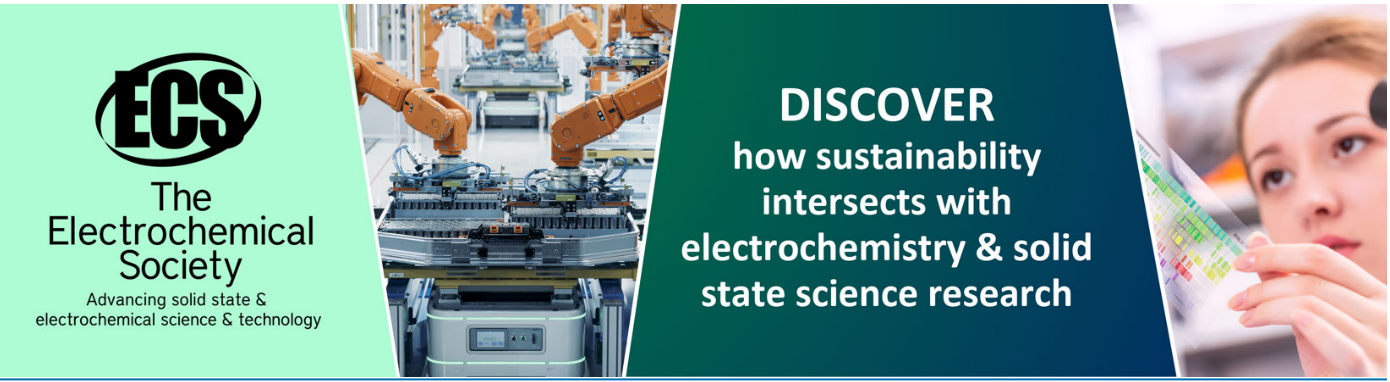

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Analysis of the mechanical properties of magnesium equal-channel angular pressing of a plate on mandibular fracture through bending and ductility tests in physiological fluids of Dulbecco's Modified Eagle Medium

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Abstract. Magnesium is used as a degradable body implant material. Utilization of magnesium as a degradable implant is hampered because of the high degree of degradation of the physiological environment. Given the nature of magnesium biodegradation, one must know how to prevent or suppress the speed of the rate of biodegradation, so that it can be adjusted to the rate of bone healing process. Several studies have been performed to overcome these limitations, such as the equal-channel angular pressing (ECAP) procedure, which is used for grain refinement; this procedure can decrease the degradation rate and increase the mechanical properties of magnesium. We analyzed the mechanical properties of ECAP magnesium after immersion in DMEM solution using 10 ECAP magnesium samples each for the bending and ductility tests. The obtained values were used to analyze the mechanical properties on their respective tests. Time of immersion affected the bending and ductility values of ECAP and decreased both bending and ductility value of ECAP on immersion in DMEM physiologic liquid.

1. Introduction

Treatment of maxillofacial fractures requires a fixation tool in the form of implants applied to bone during a certain period. During such a treatment, implants applied to fractured bone fragments are immobilized until bone unification is achieved [1]. The bone implant material should have sufficient mechanical strength for a duration until bone healing occurs and must also have resorption speed in accordance with the rate of bone healing, biocompatibility, and good biosafety, as required for use in human tissue [2,3].

Because of the limitations of the metal material, resorbable implant material has been developed to avoid further surgery to remove the implant after bone healing occurs. Magnesium is a metal material that can be potentially used as a hard tissue implant. However, it is necessary to control the velocity of biodegradation so it can be adjusted with the appropriate duration to ensure adequate bone healing [4,5]. In the presence of biodegradation characteristics of magnesium, it is necessary to prevent or suppress the rate of biodegradation so that it can be adjusted with the rate of the bone healing process.

Karayan et al reported the equal-channel angular pressing (ECAP) process for magnesium, which was processed through ECAP with six presses at a temperature of 300°C to reduce the grain size from 700 µm to 10 µm and obtain good surface morphology. In addition, the study also found an increase in corrosion of pure magnesium in Ringer solution [6]. Orlov et al reported that ZK60 magnesium alloy ECAP became stronger from 264 MPa to 351 MPa with the improvement in mechanical properties [7].

Based on previous findings, they studied the magnetic mechanical properties of ECAP, including the in vitro bending and ductility tests, on immersion of Dulbecco's Modified Eagle Medium (DMEM) solution. DMEM solution contains organic and inorganic ions resembling the body's plasma fluid. Therefore, magnesium ECAP is expected to be used as an implantable material, particularly in fracture cases, in the field of oral and maxillofacial surgery.

2. Methods

This experimental laboratory research on ECAP magnesium processing was performed at Jakarta State Polytechnic, and the bending and ductility tests were conducted at the B2TKS Laboratory, BPPT Puspiptek Serpong Area, from June to August 2014. The samples included 20 ECAP magnesium specimens formed according to ASTM E8–04 standard test methods. ECAP magnesium processing was performed using 6x pressing speed of 0.1 mm/sec at 300°C. We utilized the 3-point bending method using a homogeneous magnesium metal material which had the same chemical and physical structure; titanium was used as a control.

For the clear test, ECAP magnesium samples were examined according to the ASTM D 790 bending test standards for metallic materials Standard Test Methods for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials. The ECAP magnesium was formed according to the die form and was immersed in a 50 cc DMEM solution at 37°C. The samples were subsequently washed with chromate acid solution based on the ASTM G 31-72 Standard practice for laboratory immersion corrosion testing of metals by soaking the specimens in a DMEM solution (pH7, 50 cc). Next, using the 3-point bending machine, the bending test was performed on the sample. This research used two samples for each immersion time; therefore, the results of the bending test were recorded as the mean of both samples.

For ductility testing, ECAP magnesium samples were tested based on the ASTM E190 standard test methods for bend testing of material for ductility. The ECAP magnesium was formed according to the die form and was immersed in a 50 cc DMEM solution at 37°C. The samples were then washed with chromate acid solution based on the ASTM G 31-72 Standard practice for laboratory immersion corrosion testing of metals by soaking the specimens in DMEM solution (pH7, 50 cc). The samples were then tested for ductility using the universal testing machine UPM 1000. This study used two samples for each immersion time; therefore, the results of the ductility test were recorded as the mean value of both samples.

The data obtained in this study were recorded and collected as primary data. Next, the average and standard deviation were determined by performing descriptive data analysis. The initial data distribution was tested using the Shapiro–Wilk test, and the data were considered normal if the probability value was $P>0.05$. The data obtained were not homogeneous ($p < 0.05$) and hence could not be continued using the ANOVA test. The data were analyzed using the Kruskal–Wallis nonparametric test. Computation was performed using the SPSS statistics program, and $p < 0.05$ was considered statistically significant.

3. Results

This study was conducted to determine the difference between the ECAP ductility and bending value of ECAP magnesium immersed in DMEM. Ductility and bending tests were performed on each sample using the universal machine testing UPM 1000 prior to and following immersion in DMEM for 3, 6, 12, and 24 days.

Table 1. Bending Value of ECAP Magnesium Before and After Immersion

	Duration of Immersion in DMEM					P-value*
	0 day	3 days	6 days	12 days	24 days	
Bending Value (MPa)	83.25	95.6	102.4	129.4	81	0.199

*Shapiro–Wilk normality test, $p > 0.05$

The mean bending value of each ECAP magnesium sample at various immersion times is shown in Table 1. The data were distributed based on the Shapiro–Wilk normality test ($p > 0.05$). Because the obtained data were normally distributed and were not homogeneous ($p < 0.05$), they were analyzed using the Kruskal–Wallis nonparametric test. The p-value of the ECAP magnesium bending value was 0.199 ($p > 0.05$), which was not a significant for bending values at each treatment.

Table 2. Ductility Value of ECAP Magnesium Before and After Immersion

	Duration of Immersion in DMEM					p*
	0 day	3 days	6 days	12 days	24 days	
Ductility Value (MPa)	133	137	156	148	111	0.166

*Shapiro–Wilk normality test, $p > 0.05$

The mean value of each ECAP magnesium sample at various immersion times is presented in Table 2. The data were normally distributed based on the Shapiro–Wilk normality test ($p > 0.05$). Because the obtained data were normally distributed and were not homogeneous ($p < 0.05$), they were analyzed using the nonparametric Kruskal–Wallis test. The statistical value of ECAP magnesium tensile was 0.263 ($p > 0.05$), which was not significant for tensile values at each treatment.

Table 3. Elongation Value of ECAP Magnesium Before and After Immersion

	Duration of Immersion in DMEM					p*
	0 day	3 days	6 days	12 days	24 days	
Elongation (%)	3.7	7.05	5.8	6.4	5.55	0.166

*Shapiro–Wilk normality test, $p >0.05$

The mean elongation value of each ECAP magnesium sample at various immersion times is presented in Table 3. The data were normally distributed based on the Shapiro–Wilk normality test ($p > 0.05$). Because the obtained data were normally distributed and were not homogeneous ($p < 0.05$), they were analyzed using the Kruskal–Wallis nonparametric test. The statistic value obtained for ECAP magnesium elongation was 0.166 ($p > 0.05$), which was not significant for elongation values at each treatment.

4. Discussion

The present study was conducted to assess the magnetic strength of ECAP magnesium using the bending and ductility tests as potential biomaterials for bone implant for use in the field of oral and maxillofacial surgery. Orlov et al (2010) stated that magnesium alloy ZK60 ECAP became stronger with improvement in mechanical properties from 264 MPa to 351 MPa [7]. Several studies have reported an increase in the mechanical strength of ECAP magnesium on tensile strength. In addition, the authors reported that the magnesium alloy ZK60 ECAP was stronger with the increase in tensile strength from 264 MPa to 351 MPa [7]. According to this research, ECAP magnesium tensile values increased from 15 Kgf/mm2 (147.09 MPa) to 19 Kgf/mm2 (186.33 MPa).

Fang et al reported an increase in the tensile strength of ECAP Al-Cu alloy from 83 MPa to 239 MPa [6]. In addition, Bin Chen et al obtained an increase in the magnesium alloy AZ91 to 417 MPa from its initial strength [8], which supports the statement that ECAP could increase the mechanical strength of a metal material. The results of this study also found that the tensile values of magnesium ECAP were better than those of polymers and were close to the tensile strength of titanium. A study by Vroman and Tighzert obtained mean tensile values of 32.22 MPa for polyglicolyde polymer material (PGA), 45–70 N/MPa for poly(L-lactide) (PLLA), and 23 MPa polycaprolactone (PCL) [9]. Buijs et al (2007) obtained a mean PGA-PLLA plate and screw value ranging from 57.05 MPa to 156.81 MPa, while the average plate and screw value of 1.5 and 2.0 mm titanium was 251.21 MPa and 369.84 MPa, respectively [10].

In the present study, the highest ECAP magnesium bending value of ECAP magnesium was obtained at 12 days (129.375 MPa) and the bending value decreased upon immersion at 24 days (81 MPa). Meanwhile, the highest ECAP magnesium tensile value of ECAP magnesium was obtained at 12 days of immersion (156 MPa) and the value decreased at 24 days (111 MPa). In the present study, the highest elongation value at the time of immersion was observed on day 3 (7.05%) and was relatively stable until day 12. The ductility of ECAP magnesium began to decline on day 24 (5.55%).

5. Conclusions

The present study demonstrates the influence of immersion time on ECAP magnesium bending value and that of decreased ECAP magnesium bending value on immersion in DMEM. The time of immersion of ECAP magnesium in DMEM affected the ductility value and decreased the ECAP magnesium ductility. Further research on the mechanical properties of ECAP magnesium, including the torsion and fatigue tests, is warranted.

6. References

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