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Table of contents

Volume 602

2019

Previous issue Next issue

Conference on Innovation in Technology and Engineering Science 8–9 November 2018, Padang, Indonesia

Accepted papers received: 31 July 2019
Published online: 06 September 2019

Open all abstracts

Preface

Papers

Sustainable Innovation in Electrical Engineering

+ Open abstract View article PDF

OPEN ACCESS 012009

Harmonic analysis in electrical system at Andalas University Hospital

N Afni, R Nazir, E P Waldi and A Pawawoi

+ Open abstract View article PDF

OPEN ACCESS 012010

The tensile properties of alumina and silica bionanocomposite material for high voltage insulation

Aulia, E P Waldi, M H Setiawan, A Winarto, Darwison, Novizon, Y Nugraha, Abdurrahman, M A Hafizi and Z Nawawi

+ Open abstract View article PDF

OPEN ACCESS 012011

Multichannel audio steganography based on MPEG surround using direct sequence spread spectrum

M Tomas, Baharuddin and I Elfitri

+ Open abstract View article PDF

OPEN ACCESS 012012

Voltage profile evaluation based on power flow analysis using Newton Raphson method: Central and South Sumatera Subsystem

M A Haq, Syafii, H D Laksono and G Hidayat

+ Open abstract View article PDF

OPEN ACCESS 012013

Performance analysis of error control coding and diversity in image transmission on wireless channels

Baharuddin, M Muharam, H Andre and R Angraini

+ Open abstract View article PDF

OPEN ACCESS 012014

Performance evaluation of image transmission using diversity selection combining technique

Baharuddin and R Angraini

+ Open abstract View article PDF

OPEN ACCESS 012015

Development of HFCT for partial discharge sensors

E P Waldi, AY Frenzi, R Fernandez, Darmawan, Darwison, H D Laksono, Aulia, Novizon, A Hazmi, H Abral, S Arief, Z Nawawi, M H Ahmad and N Hozumi

+ Open abstract View article PDF

OPEN ACCESS 012016

Study on static electrification of the PFAE-mineral oil mixture

A Rajab, H Gumilang, M Tsuchie, M Kozako, M Hikita and T Suzuki

+ Open abstract View article PDF

OPEN ACCESS 012017

PWM speed control of dc permanent magnet motor using a PIC18F4550 microcontroller

M W Fatma and M I Hamid

+ Open abstract View article PDF

OPEN ACCESS 012018

Partial discharge characteristics of nanosilica biopolymer under AC voltage

Aulia, E P Waldi, Darwison, M Anggaravidya, Novizon, M H Setiawan, Y Nugraha, Abdurrahman, M A Hafizi and I Jambak

+ Open abstract View article PDF

OPEN ACCESS 012019

Analysis of the unbalanced harmonic propagation in a three-phase power system using a parallel program

S Yunus, U G S Dinata, R Nazir and Aulia

+ Open abstract View article PDF

OPEN ACCESS 012020

Morphological characteristics of preliminary breakdown pulses of hybrid intra cloud–negative cloud-to-ground lightning at low lattitude

P Emeraldi, M I Hamid and A Hazmi

+ Open abstract View article PDF

OPEN ACCESS 012021

Increasing the quality and power capacity of HERIC PV-Inverter through multilevel topology implementation

M I Hamid and D Ardiansyah

+ Open abstract View article PDF

OPEN ACCESS 012022

Dipole planar bowtie printed antenna for ism application

H Andre, R Fernandez and Baharuddin

+ Open abstract View article PDF

OPEN ACCESS 012023

Improving the quality and quantity of cinnamon drying process using art cave in Lambung Bukit West Sumatra

A U Baiqi, P P Utami, D Anugrah, A A Fauzan, W S Ningsih and M I Rusydi

+ Open abstract View article PDF

OPEN ACCESS 012024

Design of fuzzy logic controller for temperature control of small-scale food storage

M Latif, M Muharam, Darmawan, Darwison and R R Costa

+ Open abstract View article PDF

OPEN ACCESS 012025

Shape object selection using the chi-square method

R Kurnia, F Kurnia and Fitrilina

+ Open abstract View article PDF

OPEN ACCESS 012026

Characteristics of acoustic signals from lightning using a microphone array observation system

A Hazmi, P Emeraldi and M I Hamid

+ Open abstract View article PDF

OPEN ACCESS 012027

A wireless monitoring system for comparison photovoltaic and photovoltaic thermal characteristics

Krismadinata, R Lapisa and Asnil

+ Open abstract View article PDF

OPEN ACCESS 012028

Robot mobile control based on three EMG signals using an artificial neural network

M I Rusydi, I Aryeni, Joefrinaldo, Z Romadhon and A Rusydi

+ Open abstract View article PDF

OPEN ACCESS 012029

Recognition of horizontal gaze motion based on electrooculography using tsugeno fuzzy logic

Muhammad Ilhamdi Rusydi, Mardiah Bahri, Rizky Syahreza Ryaldi, Fauzan Akbar, Kojiro Matsuhita and Minoru Sasaki

+ Open abstract View article PDF

OPEN ACCESS 012030

Towards hand gesture-based control of virtual keyboards for effective communication

Muhammad Ilhamdi Rusydi, Oktrison, Willy Azhar, Samuel W Oluwarotimi and Febdian Rusydi

+ Open abstract View article PDF

OPEN ACCESS 012031

Development of rogowski coil sensor for partial discharge detection

E P Waldi, A Y Frenzi, R Fernandez, Darmawan, Darwison, H D Laksono, Aulia, A Hazmi, A Andre, H Abral, S Arief, Z Nawawi, M H Ahmad and N Hozumi

+ Open abstract View article PDF

OPEN ACCESS 012032

Static VAR compensator for improving voltage profiles and transmission losses: Case study in Batam

S Yunus, Y I Rahmi, R Nazir, Aulia and U G S Dinata

+ Open abstract View article PDF

OPEN ACCESS 012033

Modification of arms patch of double layer printed antenna for partial discharge detection

U Khayam and Y M Hamdani

+ Open abstract View article PDF

OPEN ACCESS 012034

Partial discharge signal denoising by using hard threshold and soft threshold methods and wavelet transformation

A Zaeni, T Kasnalestari and U Khayam

+ Open abstract View article PDF

OPEN ACCESS 012035

Preliminary results on the development of monoester type insulating oil from coconut oil

A Rajab, F E Putra, J S Ramadhani, M S I Silitonga, R Kurniawan, K Qibran, M Latif and M I Hamid

+ Open abstract View article PDF

Industrial and Manufacturing Systems

OPEN ACCESS 012036

Formulation of optimization model of raw material composition to achieve clinker quality standards (Case study PT Semen Padang Plant IV)

S Rijal, A S Indrapriyatna and A H B Adi

+ Open abstract View article PDF

OPEN ACCESS 012037

A system for improving suppliers evaluation: the case of procurement in educational institution (Case study: Andalas University)

M Farid, R A Hadiguna and I Kamil

+ Open abstract View article PDF

OPEN ACCESS 012038

An evaluation on Dr. M. Djamil Hospital Padang parking lot capacity

Alfadhlani, W S F Yasrin and F Afrinaldi

+ Open abstract View article PDF

OPEN ACCESS 012039

Analysis of the application of quality management systems in the rubber industry based on ISO 9001:2015

N Fajrah, N T Putri and E Amrina

+ Open abstract View article PDF

OPEN ACCESS 012040

Setup time efficiencies of quick die change system in metal stamping process

R K Arief and Q Nurlaila

+ Open abstract View article PDF

OPEN ACCESS 012041

A framework to improve equipment effectiveness of manufacturing process - a case study of pressing station of crude palm oil production, Indonesia

A Susilawati, A Tasri and D Arief

+ Open abstract View article PDF

+ Open abstract View article PDF

OPEN ACCESS 012049

Optimization of significant factors of cement compressive strength at PT Semen Padang

P Fithri, D Meilani, N T Putri and F H Chotimah

+ Open abstract View article PDF

OPEN ACCESS 012050

Waste assessment using a lean approach in receiving process of container terminal: a case of Teluk Bayur Port

E Amrina, I Kamil and D Rahmad

+ Open abstract View article PDF

OPEN ACCESS 012051

The evaluation of bullwhip effect on distribution system of a supply chain using centralized demand information method

I Kholidasari, JR A Bidiawati and M E Sari

+ Open abstract View article PDF

Innovation in Environmental Engineering

OPEN ACCESS 012052

The use of protein binder from shaving waste for leather finishing: Judging from the physical, chemical, and morphological properties of lizard skin leather

S Sutyasmi, I F Pahlawan and G Griyanitasari

+ Open abstract View article PDF

OPEN ACCESS 012053

Food packaging development of bioplastic from basic waste of cassava peel (*manihot uttilisima*) and shrimp shell

Dasumiati, N Saridewi and M Malik

+ Open abstract View article PDF

OPEN ACCESS 012054

Effect of tannery wastewater exposure on chromium detected in the gill of *oreochromis niloticus*

T Edwin, T Ihsan and H T Tamsin

+ Open abstract View article PDF

OPEN ACCESS 012055

Minimization of household hazardous solid waste (HHSW) with 4R concepts (reduce, reuse, recycle and recovery) in Padang City, Indonesia

Y Ruslinda, S Raharjo, Y Dewilda, Hidayatullah and R Aziz

+ Open abstract View article PDF

OPEN ACCESS 012056

Greenhouse knockdown in Merauke

M Alahudin, R D Latuheru and N L S Suryaningsih

+ Open abstract View article PDF

OPEN ACCESS 012057

Distribution of organic contamination based on depth stratification in Maninjau Lake, Indonesia

P S Komala, A Nur and I Nazhifa

+ Open abstract View article PDF

OPEN ACCESS 012058

Effect of pipe diameter changes on the properties of fluid in closed channels using Osborne Reynold Apparatus

A Nur, R Afrianita and R D T F Ramli

+ Open abstract View article PDF

OPEN ACCESS 012059

Study of recycling potential of solid waste of tourist area in Pariaman City

R Aziz and Mira

+ Open abstract View article PDF

OPEN ACCESS 012060

The effect of additional vegetables and fruits waste on the quality of compost of cassava chip industry solid waste on takakura composter

Yommi Dewilda, Rizki Aziz and Restu Ayu Handayani

+ Open abstract View article PDF

OPEN ACCESS 012061

Effects of different pre-treatment methods on anaerobic mixed microflora for hydrogen production and COD reduction from domestic effluent

B Primasari, M Z A Tamin and M A H Mustafa

+ Open abstract View article PDF

OPEN ACCESS 012062

Spatial distribution of coliform bacteria in Batang Arau River, Padang, West Sumatera, Indonesia

D. Helard, S Indah and M Wilandari

+ Open abstract View article PDF

Mechanical and Thermal Systems

OPEN ACCESS 012063

Analysis of cracks in the welded zone of stainless steel pipe used in high-pressure decomposer equipment

Husaini, M Najib and I Hasanuddin

+ Open abstract View article PDF

OPEN ACCESS 012064

The determination of workspace and the performance evaluation of PRoM-120 with 3 and 4 kinematic constants

Adriyan and Sufiyanto

+ Open abstract View article PDF

OPEN ACCESS 012065

Thermal characteristics and phase transformation of iron ores containing varied crystalline water with coal mixtures

M M F Sinuhaji, S Harjanto and A Hapid

+ Open abstract View article PDF

OPEN ACCESS 012066

The influence of some solution candidate on the performance of boundary element inverse analysis in detecting rebar corrosion

S Fonna, Gunawarman, S Huzni and A K Ariffin

+ Open abstract View article PDF

OPEN ACCESS 012067

Sound absorption characteristics of the natural fibrous material from coconut coir, oil palm fruit bunches, and pineapple leaf

M Rusli, M Irsyad, H Dahlan, Gusriwandi and M Bur

+ Open abstract View article PDF

OPEN ACCESS 012068

Effect of garlic oil as lubricant additive into coconut and palm oils on the physical and tribological properties

D Gasni, D Chandra, A A Putra and R Fajri

+ Open abstract View article PDF

OPEN ACCESS 012069

Natural frequencies of twisted cantilever beam

J Malta, Jefri, M Bur and E Satria

+ Open abstract View article PDF

OPEN ACCESS 012070

Corrosion Resistance of β type titanium (TNTZ) in 3%NaCl solution

J Affi, Gunawarman, Y Yetri, H Fajri, D Juliadmi, N F Nuswantoro, Nurbaiti, S Fonna, D H Tjong and M Manjas

+ Open abstract View article PDF

OPEN ACCESS 012071

Hydroxyapatite Coatings on Titanium Alloy TNTZ using Electrophoretic Deposition

Gunawarman, N F Nuswantoro, D Juliadmi, H Fajri, A Budiman, D H Tjong and M Manjas

+ Open abstract View article PDF

OPEN ACCESS 012072

Synthesis and characterization of calcium precursor for hydroxyapatite synthesis from blood clam shell (*Anadara antiquata*) using planetary ball mill process

Gunawarman, J Affi, Y Yetri, Ilhamdi, D Juliadmi, N F Nuswantoro, H Fajri, A Ahli, R Gundini and Hadi Nur

+ Open abstract View article PDF

OPEN ACCESS 012073

Production of pig iron nugget from low-grade iron ore and pyrolyzed oil-palm-empty-fruit-bunch composites

A Setiawan, R P Suratha, S Harjanto and E Kusrini

+ Open abstract View article PDF

OPEN ACCESS 012074

Experimental evaluation of tuned liquid column damper and tuned mass damper in a space structure model

L Son, M Bur and A D Andria

+ Open abstract View article PDF

OPEN ACCESS 012075

Atmospheric corrosion map of structural steel in industrial area: a preliminary investigation

S Huzni, Affandi, I Tanjung and S Fonna

+ Open abstract View article PDF

OPEN ACCESS 012076

Corrosion potential of reinforced steel in reinforced concrete in Kabupaten Bireun: Analysis of groundwater content used as a concrete mixture

R D I Kurnia, Suhaimi, S Huzni and S Fonna

+ Open abstract View article PDF

OPEN ACCESS 012077

Design of solid desiccant air conditioning system

D A Saputra, N A Saputra, L Susanti, P Fithri and D I Putra

+ Open abstract View article PDF

OPEN ACCESS 012078

Numerical analysis of U-shaped hysterisis steel damper with energy absorber for seismic areas

E Satria, L Son, M Bur, M D Akbar and S Haris

+ Open abstract View article PDF

OPEN ACCESS 012079

Optimization of matrix compositions of Al_2O_3, SiO_2, Caolin, and CaO on the mechanical properties of a geopolymer composite with short carbon fiber

J Akmal, M Badaruddin, M K Ismoyo and S D Yuwono

+ Open abstract View article PDF

OPEN ACCESS 012080

Characterization on particle size distribution of reduced lateritic nickel ore using biomass carbon reduction

F Abidin, S Harjanto, A Kawigraha and N V Permatasari

+ Open abstract View article PDF

OPEN ACCESS 012081

The effect of solar water heater performance by variation of the plate shaped

D Harun, M I Maulana and Akhyar

+ Open abstract View article PDF

OPEN ACCESS 012082

The experimental performance of the semi-cylindrical type of solar concentrator collector on the addition of heat storage material

D Harun, Zulfadhli and Akhyar

+ Open abstract View article PDF

OPEN ACCESS 012083

Analysis cutting forces and surface roughness of fibre reinforced polymer for end mill processes

F Ridwan, R Havendri, O Susanti, Gusriwandi and Yulhizhar

+ Open abstract View article PDF

OPEN ACCESS 012084

Surface characterization of the ceramic coating process on aluminum matrix composite reinforced particulate

H Sukma, D Rahmalina, B Sulaksono and E A Pane

+ Open abstract View article PDF

OPEN ACCESS 012085

The potential of rising husk fiber/native sago starch reinforced biocomposite to automotive component

Nusyirwan, H Abral, M Hakim and R Vadia

+ Open abstract View article PDF

OPEN ACCESS 012086

The effect of particle compositions on the activation energy of the pa6/bagasse composite

S Thalib, S Huzni, S Fonna, C H Azhari and S Zakaria

+ Open abstract View article PDF

OPEN ACCESS 012087

Hardness and impact energy absorbed produced by Q&T steel and DQ&T teel

Yurianto, Pratikto, S Rudy, S Wahyono, Y Eflita, S Agus and U Yusuf

+ Open abstract View article PDF

OPEN ACCESS 012088

Mechanical properties of mild steel by adding *Theobroma Cacao* Peels Extract (TCPE) inhibitor

Y Yetri, Gunawarman, R Hidayati and A Zamri

+ Open abstract View article PDF

OPEN ACCESS 012089

The needs to investigate the effect of road surface vibrations to the fatigue life of a coil spring

M Ali, Husaini, T E Putra and N Ali

+ Open abstract View article PDF

OPEN ACCESS 012090

Corrosion behavior of Ti6Al4V ELI coated by bioceramic HA in artificial saliva at fluctuating temperatures

R Muharni, Gunawarman and Y Yetri

+ Open abstract View article PDF

OPEN ACCESS 012091

In vitro of Mg-1.6 Gd alloys after hot extruded for biomaterial application

O Susanti, E W Bachtiar, S Harjanto and Gunawarman

+ Open abstract View article PDF

OPEN ACCESS 012092

Effect of coating time and protective current on thickness of paint layer of Steel ST-37 by continuous painting

Z Mansjur, Arrijani and M F Suharto

+ Open abstract View article PDF

OPEN ACCESS 012093

Effect of Pouring Temperatures on Porosity and Mechanical Properties of Gravity Die Casting Magnesium Alloy

I P Nanda, M H Jahare, M H Idris, S B Kumar, M H Hassim and A Arafat

+ Open abstract View article PDF

OPEN ACCESS 012094

Mechanical and degradation properties of zinc adopted magnesium alloys for biomedical application

I P Nanda, M H Hassim, M H Idris, M H Jahare, S S Abdulmalik and A Arafat

+ Open abstract View article PDF

Sustainable Civil Engineering Solutions

OPEN ACCESS 012095

The Effects of the distance between ground-sill and double cylinder-piers against the scour patterns

M Mera and M Thaahaa

+ Open abstract View article PDF

OPEN ACCESS 012096

Analytical Network Process (ANP) for priority setting of strategic roads handling at Tebo Regency

Yosritzal, J Permana, B Istijono, B Hidayat, T Ophiyandri and H Gunawan

+ Open abstract View article PDF

OPEN ACCESS 012097

Simulation of the effect of floodway on Batang Kandis River flood control

Junaidi, S Marona and Dalrino

+ Open abstract View article PDF

OPEN ACCESS 012098

Identification and analysis of application of Construction Management System (CMS) in the implementation of construction management

B Hidayat, A Suraji and R Frankly

+ Open abstract View article PDF

OPEN ACCESS 012099

Intersection performance evaluation and designing intersection at concourse between arterial road and ramp of Medan-Kualanamu-Tebing Tinggi Highway

Amrizal and A H S Harahap

+ Open abstract View article PDF

OPEN ACCESS 012100

Sensitivity analysis of stormpav composite pavement

E E Putri, F J H Rewani, M A Mannan, W H W Ibrahim, M R Kabit, L S Tirau, R A Chan and R Buking

+ Open abstract View article PDF

OPEN ACCESS 012101

Infrastructure maintenance system for community development projects to improve the quality of infrastructure services in West Sumatra Province

G Vitri and H Herman

+ Open abstract View article PDF

OPEN ACCESS 012102

The Study Of Riverbed Change And Bed-load Transport In The Middle Segment Of The Batang Kuranji River

Junaidi, E D E Putra, A Junaidi, Sunaryo and Nurhamidah

+ Open abstract View article PDF

OPEN ACCESS 012103

Determining the priority of new road development according to the West Sumatera provincial government perception

R D Susanti, Purnawan and Yossyafra

+ Open abstract View article PDF

OPEN ACCESS 012104

Shear behavior of fly ash reinforced concrete beam without shear reinforcement

A E Nasution, R Kurniawan and R Thamrin

+ Open abstract View article PDF

OPEN ACCESS 012105

Determining the priority criteria and ranking of provincial bridge maintenance in West Sumatra using a combination of the Fuzzy Analytical Hierarchy Process and VIKOR-Modification methods

Yossyafra, N Angelia, Yosritzal, Meyadtri and D I Mazni

+ Open abstract View article PDF

OPEN ACCESS 012106

Marshall immersion test of warm mix asphalt polymer using Bayat natural zeolite

A T Handayani, S N Peni and H Pandita

+ Open abstract View article PDF

OPEN ACCESS 012107

Analysis of water balance on Lake Maninjau, West Sumatera

Sunaryo, Y D Nola, B Istijono and Junaidi

+ Open abstract View article PDF

OPEN ACCESS 012108

Seismic retrofitting analysis using concrete jacketing and shear wall on dental hospital building of Andalas University

Fauzan, F A Ismail and Z A Jauhari

Mechanical and degradation properties of zinc adopted magnesium alloys for biomedical application

by Is Prima Nanda

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Mechanical and degradation properties of zinc adopted magnesium alloys for biomedical application

Mechanical and degradation properties of zinc adopted magnesium alloys for biomedical application

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Abstract. The demand for short-term degradable implant in bone fixation applications is growing steadily due to the aging population worldwide. Degradable implants have the advantage that the second surgery for implant removal is not required. Magnesium is one of the best candidates because it is biodegradable, physiologically compatible and even stimulates bone reconstruction. However, the high degradation rate of pure magnesium in human body fluids may prevent its wider application. In this study, Zinc (Zn) was added in magnesium (Mg) to improve its properties. The effects of five different weight percentage of Zinc (2%, 4%, 6%, 8%, 10%) were investigated. The microstructure and mechanical properties evolution of the alloys were characterized and evaluated using optical microscopy, Scanning Electron Microscope (SEM), tensile test and Vickers hardness test, while degradation behavior was examined using electrochemical corrosion test. The binary Mg-Zn cast alloy with 6 wt. % zinc content (labeled as Mg-6Zn) shows optimum mechanical strength with slowest degradation rate.

Keywords - Mg-Zn alloys; biomedical application; mechanical properties; biodegradation

1. Introduction

Biomaterials used as implants in the body have been emerging over the past half of century, and currently a great number of implants available throughout all medical application such as orthopedic surgery, general surgery, maxillofacial surgery, cardiology, gynecology, and urology. Implantation of artificial bone fixation requires it to stay in the body permanently, while certain implication only requires implant support for a short period. When a permanent implant is used for a temporary application, additional surgeries are required to remove these devices after the healing process. Thus, the removal process increases the patient grim and cost of health care.

On the other hand, biodegradable implants offer advantages over metal analogs in term of zero implant removal, zero revision surgery, and minimum radiological imaging, as they dissolve in a period [1]. It provides resultant high strength while allowing osseointegration and replacement by the host tissue. The biodegradable implant also eliminates the complications associated with the long-term presence of implants in the body.

The material for biodegradable implants is essential to have few characteristics such as biocompatible, bioabsorbable, secure initial fixation strength and promote osseointegration [2]. Magnesium is a suitable candidate for this requirement. Pure Magnesium can be metabolized by the body system and is the fourth highest element present in the human body. Normal adult consumed about 300 to 400 mg of magnesium daily, and Mg2+ excessive is not toxic and ejaculated through the urinary system [3]**.** Magnesium also has a value of elastic modulus, compressive yield strength and density close to the natural bone [4]**.** In vitro cell test on pure magnesium showed positive cell proliferation and viability with no sign of growth inhibition [5]**.** However, the critical issue for pure Mg is due to the relatively very susceptible to degradation, inadequate strength, the release of hydrogen gas and a relatively high degradation rate in the human body [6]. Rapid degradation rates consequently disturb bone ingrowth performance, thus making healing problematic. For a better effect of these materials in the human body, their degradation behavior needs to be altered, and the interactions between the degradation product and the biological environment need to be explored.

Alloying with other biocompatible materials is of special interest in the biomedical application. Zn is one of important elements in the body system and has a positive record in biomedical applications. It was reported that zinc could improve the strength of magnesium alloy through increase age hardening response, produce intermetallic compounds, refine grain size and improve castability [7]. The addition of Zn can improve both tensile strength and ductility of the alloys as well as reduces the degradation rate by slowing the rate of anodic dissolution [8].

The degradation resistance of alloying-element in Mg alloys is not widely reported and therefore not well established. The toughness of pure magnesium is greater than that of ceramics. However it degrades rapidly in the human body system, losing strength before tissue healing. To maintain the mechanical integrity, and biocompatibility, sufficient alloying content is essential. Thus, the interaction between microstructure, phase transformation and degradation properties of the alloying composition must be extensively investigated. This study, therefore, contributes to clarifying the correlation between alloying and degradation, which plays an important role in an efficient alloy design for accomplishing desired benefits.

2. Material and method

Mg-Zn alloys were prepared by melting pure 99.99 wt.% Mg (Figure 1(a)) and pure 99.99 wt% Zn (Figure 1(b)) in an induction furnace (Figure 2(a)). A graphite crucible was used, and the metal was melted under a flowing argon atmosphere to prevent ignition and oxidation of the molten metal. Pure Zn was added into crucible after magnesium metal was melted at about 650°C. The temperature was maintained at 750°C for about 15 minutes while vigorously stirred 7-9 times. The molten Mg-Zn alloy was then poured into a preheated steel mold (Figure 2 (b)).

Figure 1. Pure magnesium and pure zinc

Figure 2. Induction furnace and steel mold

The surface morphology, microstructure, and phases distribution for produced Mg-Zn alloys were observed using an Olympus BX-60 optical microscope and Philips XL-40 scanning electron microscope equipped with energy dispersive spectroscopy (EDS. Before the analysis, the samples were polished up to mirror finish by using a polishing cloth and alumina suspension.

Vickers microhardness test was performed on alloys surface to reflect the ability to resist plastic deformation. The tests were performed using Matsuzawa DVK-2 microhardness testing machine conforming to the ASTM-E98-82 standard. Before the test, the samples were ground up to 1200 grit SiC paper finish and then cleaned with acetone and hot air. Data concluded in this study were an average value of 5 measurements at a different location.

The tensile strength values of the alloys were tested with a tensile speed of 1 mm/min using an Instron universal testing machine. The tests were conducted according to ASTM-A370. The specimens were prepared from the as-cast Mg-Zn alloy with a gauge length of 25mm and thickness of 10mm. The elongation was determined using extensometer. A minimum of 4 separate tests was performed for each alloy.

To explain the mechanistic aspects that determine the ultimate degradation rates realized, the principal corrosion test method used in this study is potentiodynamic polarisation. The corrosion test was carried out in the solution of simulated body fluid at the temperature of 37 ± 1 ºC using an advanced electrochemical system of potentiostat (Parstat-2263), with three electrode cells of saturated calomel electrode (SCE) as a reference electrode, graphite electrode as counter electrodes, and the sample as the working electrode. The surface area of the sample exposed to the electrolyte was 1 cm^2. The polarization in the anodic direction proceeded at a scan rate of 0.9 mV/s. The potentiodynamic curves of pure Mg and Mg-Zn alloys were analyzed by Tafel extrapolation method, and the values of corrosion potential (E_{corr}) and corrosion current density (i_{corr}) have been obtained. Degradation rate has been calculated using the values of E_{corr} and i_{corr}. Three replicates were conducted for each group of samples.

3. Result and discussion

3.1. Effect of Zn alloying on the microstructure

The chemical compound of the specimens was examined by Energy Dispersive Spectrometer (EDS) connected to Scanning Electron Microscopy (SEM), has appeared in Table 4.1.

Table 1. The chemical composition of the Mg-Zn alloys in wt. %

Sample	Code	Zn	Mg
1	Mg-2Zn	1.62	98.38
2	Mg-4Zn	4.03	95.97
3	Mg-6Zn	6.33	93.67
4	Mg-8Zn	8.46	91.54
5	Mg-10Zn	9.65	90.35

Figure 3 shows the optical microstructures of pure Mg and as cast Mg–Zn alloys. All materials show nearly equiaxed grain structure. In Figure 3b to f, it can be observed that after Zn addition, eutectics form mainly along the grain boundaries of Mg were present, and the width of eutectics along grain boundary becomes clearer as the content of zinc increases. According to the Mg-Zn phase diagram [9], the eutectics were predominantly composed of Mg_2Zn_{11} and $MgZn_2$. The development of $MgZn_2$ over the equilibrium phase Mg_2Zn_{11} was expected to non-balance solidifying conditions experienced by the alloy during fast cooling in a ferrous mold under atmospheric environment [10]. Moreover, the grain boundaries are portrayed by an intermittent conveyance of small precipitates.

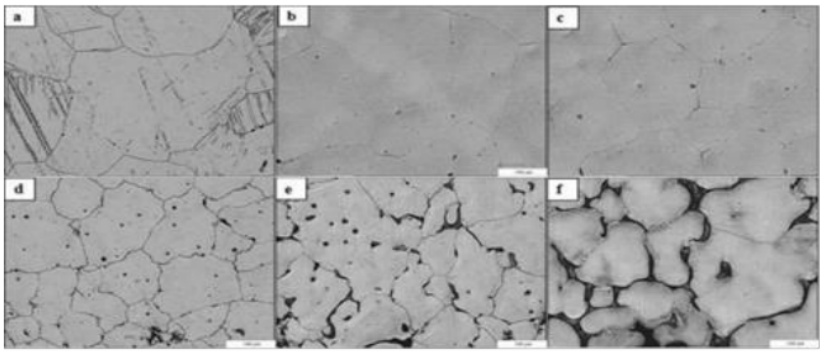

Figure 3. Optical microstructure pure Mg and Mg-Zn alloys (a) pure Mg, (b) Mg-2Zn, (c) Mg-4Zn, (d) Mg-6Zn, (e) Mg-8Zn, (f) Mg-10Zn

3.2. Effect of Zn alloying on mechanical properties

Vickers hardness tester measures the resistance of a material to indentation. During testing, the indenter is pushed into the sample surface normally. The ASTM-E98-82 standard was followed for testing the hardness of the benchmark components. The hardness of specimens was measured five times in different places to reduce the measurement error. The hardness based on wt.% alloy composition of all samples tested in this study appears in Figure 4. It is observed that the hardness of Mg-Zn alloys is improved essentially because of alloying augmentations (for 2, 4 and 6 wt.% alloying), and then slightly drop at Mg-8Zn and Mg-10Zn alloys. Most outcomes represent a general pattern that increasing total alloying loading in Mg prompts higher hardness, in a moderately monotonic manner. Slight drop in hardness value for 8 and 10 wt.% alloying may be ascribed by the development of the secondary phases on these alloys Mg-8Zn, and Mg-10Zn. Since the maximum solubility of zinc in magnesium is 6.2 wt.% and as previously reported that the excess Zinc will interact with Mg and form abundant of Mg and Zn containing phases in the matrix and grain boundary [11]. These phases isolate the matrix and increase the number of crack sources. Consequently, disturb the mechanical properties improvement.

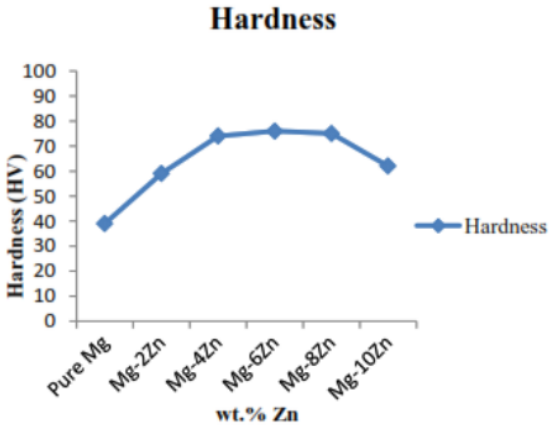

Figure 4. Vickers microhardness value of Mg-Zn alloys based on Zn wt.%

The tensile strength of the alloy based on Zn wt.% is shown in figure 4. It is also observed that the tensile strength value increases until 6 wt.% of Zn while it drops when reaching 8 and 10 wt.% of Zn. Also, Table 2 summarized the value of tensile strength, yield strength and elastic modulus of the Mg-Zn alloys. While the pattern of tensile strength and yield strength follow a similar trend as the hardness graph, the elastic modulus value shows an irregular pattern. These phenomena of the may be attributed to the formation of secondary phase by the excessive presence of Zn. Zn reacts with magnesium and progress toward becoming wellsprings in the matrix and the grain boundary, hence, the elasticity of the alloy decrease [11].

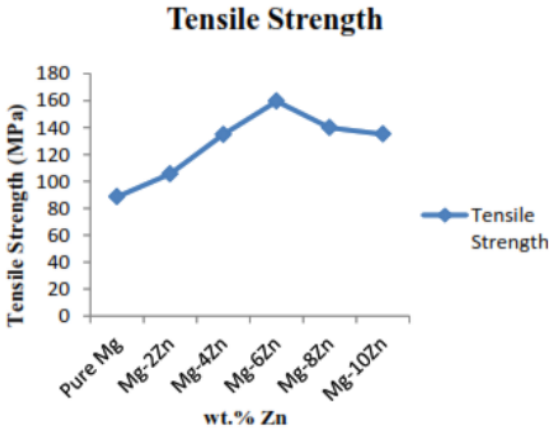

Figure 5. Tensile strength value of Mg-Zn alloys based on Zn wt.%

Table 2. The Tensile strength, Yield, and Elastic Modulus value of Mg-Zn alloys

Sample	Tensile Strength (MPa)	Yield strength (MPa)	Modulus (GPa)
Pure Mg	88.5	27.5	37.5
Mg-2Zn	105.57	54.19	35.94
Mg-4Zn	134.82	65.61	29.93
Mg-6Zn	159.57	128.8	48.66
Mg-8Zn	139.84	89.98	41.55
Mg-10Zn	135.12	83.43	33.29

Alloying Mg with Zn will lead to grain refinement and development of the second phase along the grain boundary and matrix [12]. According to Xiaobo Zhang et al., non-basal dislocation slip, as well as basal dislocation slip, were actuated in fine grain Mg alloys as a result of the grain boundary compatibility impact and their HCP crystal structure [13]. It implies that for the single-crystal Mg, at ambient air environment the disfigurement mechanism of the material is dominated by the basal dislocation slips. Nonetheless, due to neighboring grains restraint, the circumstance in the polycrystalline Mg alloys is unique. The occurrence of basal dislocation slip in polycrystal can lead to strain incompatibility at grain boundaries. For Mg alloys, the grain boundaries are sufficiently solid thus extra stress emerges to keep up strain compatibility at grain boundaries. This compatibility stress promotes to the initiation of non-basal dislocations slip. In this manner refinement of grains because of Zn integration can constrain the occurrence of dislocations and confine the movement of the non-basal dislocations slip mechanisms which improve the mechanical performance of the alloy [14].

Moreover, based on the Mg-Zn binary phase diagram, in the stable state, the highest possible solubility of Zn in Mg is up to 1.6 wt.% at room temperature. Subsequently, the Zn element dissolved into α-Mg to some amount [15] and developed intermetallic MgZn phases. Subsequently, with expanding Zn content from 2wt.% to 6wt.% in Mg-Zn alloy, similar with grain strengthening, solid solution strengthening and second phase strengthening enhance the mechanical behavior of Mg-Zn alloys. On the other hand, the grains are more refined, and a total of intermetallic phases in Mg-8Zn and Mg-10Zn alloys is greater than lower Zn alloys. Expanding the total of intermetallic phases and precipitating of these Mg-Zn phases along the grain boundaries can viably fortify the alloy grain boundaries [13] which increase the strength of-of the Mg-Zn alloys compare to pure Mg. Table 2 likewise demonstrates that by adding more than six wt.% Zn tensile strength of the Mg-Zn alloy decreased. It was specified by the previous researcher that the precipitated second phase could impressively enhance the hardness while reducing the ductility of the alloys [16]. In another word, the second phase may expand the dislocation density.

3.3. Effect of Zn alloying on degradation performance

Potentiodynamic polarization curves provide useful information on the degradation behavior and degradation rate. The potentiodynamic polarization curves measured in simulated body fluid solution for Mg-Zn alloys samples are displayed in Figure 6. The corrosion potential (E_{corr}) and the corrosion current densities (i_{corr}) derived from the polarization curves by using Tafel extrapolation method are mentioned in Table 3. Electrochemical tests showed that alloying is a promising way to improve the degradation properties of Mg. As expected, the polarization curves for all Mg-Zn alloys indicate lower current density and more positive potential, while pure Mg exhibits the most negative corrosion potential (−2.027 V). The corrosion potential of the alloy increases as the content of Zn increases. This indicates the improvement in degradation resistance of the Mg by alloying. The enhanced degradation properties are attributed due to the formation of the MgZn phase in the alloy matrix, which directly proportional to the increase of Zn content [12]. Generally, this MgZn phase protects by acting as a barrier against electrons and ions diffusion, thus reducing the electrochemical reactions of alloy and

electrolyte. For this reason, Mg-6Zn samples offer better degradation resistance properties compared to Mg-2Zn and Mg-4Zn specimens.

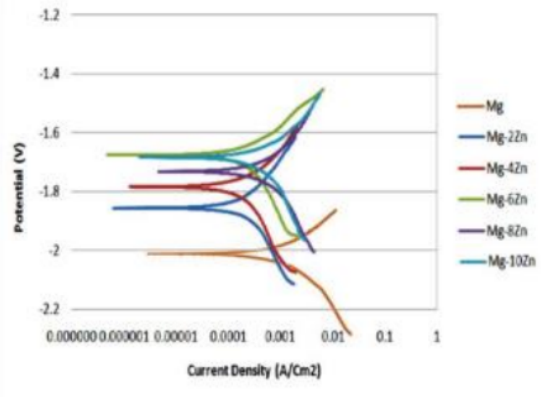

Figure 6. Electrochemical polarization curves of Mg-Zn alloys under investigation

However, the E_{corr} decrease after Zn content exceeds 6 wt.%. It was reported that the increase in Zn content over 6 wt.% would promote other intermetallic phase formation in the alloys [12]. These phases created a ceaseless network structure, prompting to the development of more anode-cathode sites. Hence, more galvanic degradation would occur, leading in a high degradation rate. Thus, it can be concluded that the degradation performance of Mg-8Zn and Mg-10Zn alloys are induced by the galvanic couple reaction, which accelerated the dissolution of the α-Mg matrix, and directly contributed to increment of degradation rate.

Table 3. Potentiodynamic polarization curveparameters derived by the Tafel extrapolation

Specimen	E_{corr} (V)	i_{corr} (μA)	Corrosion rate (mmpy)
Pure Mg	–2.027	309	7.06
Mg–2Zn	–1.860	210	4.8
Mg-4Zn	–1.781	187.5	4.28
Mg–6Zn	–1.675	122	2.78
Mg–8Zn	–1.761	178.4	4.07
Mg–10Zn	–1.740	135	3.08

4. Conclusion

According to the objective of the experiment, analyzes that have been performed, and interpretation of the results, it can be concluded that:

i. Mechanical properties of the Mg improved as the Zn content increased from 2 to 6wt%. Further increase of Zn content until 10 wt.% resulted in a considerable drop in mechanical performance.
ii. Zn addition up to 6wt.% provided 3 times better degradation resistance when compared with pure Mg due to the formation of the MgZn phase. The present of $Mg_{51}Zn_{20}$ phase in Zn content more than 6wt.% leads to rapid degradation rate.

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