ABSTRACT

The technique for the production of multifunctional scaffolds from bioactive ceramics and biodegradable polymers for use in tissue scaffolds remains challenging. Here, the goal was to fabricate 3-D nanocomposite nanofiber scaffolds of nanohydroxyapatite/poly(lactic acid) (nHA/PLA) prepared by air jet spinning (AJS) as a novel and facile composite fabrication process. The characteristics of the fabricated 3-D scaffolds were investigated using SEM, water contact angle, DSC, FTIR, XRD analyses and tensile tests. The surface morphology exhibited highly interconnected bonded fibers due to the high fabrication rates. It was also found that the nHA particles were effectively embedded in the fibers’ surface due to the difference in the kinetic energies between the nHA particles and polymer molecules. The as-received PLA film showed a low crystallinity value of about 19%, which was expected with the casting process. The crystallinities of the plain PLA and nHA/PLA membrane scaffolds were about 31.78% and 32.21%, respectively. This reveals that HA nanoparticles could engage in a beneficial interaction with the PLA chain molecules during the AJS process. The tensile strength of the membrane PLA mats, particularly the hybrid nanocomposite samples with low nHA contents, was considerably improved compared to that of the PLA casted film. Biological in vitro cell cultures of MC3T3-E1 osteoblast-like cells on the fabricated scaffolds were studied for up to seven days. The nanocomposite membrane mats of nHA/PLA, fabricated by AJS, had highly interconnected fibers. This facile technique has a high
production rate and is a new concept of potential interest for bone tissue engineering applications.

**Keywords**: Polymer-matrix composites (PMCs); Thermal properties; Tissue Engineering; Air jet spinning
2. Flash preparation of Polymeric Nanofibers Using Air Jet Spinning

Abdallah Abdel-hay, Nasser A.M. Barakat, Jae Kyoo Lim

Abstract

Although electrospinning is the most widely used technique in production of nanofibers due to its simplicity, but low production rate and dependence on high voltage power supply are the main dilemmas. Moreover, completely non conductive polymers solutions cannot be electrospun which adds further constrain. In this study, we introduce a novel, safe and low cost technique to produce the polymeric nanofibers in ultrafast production rate. The proposed technique is based on exploiting the high speed air jets in stretching the polymer solution into ultrathin fibers. The average production rate of the proposed technique is around 5 mL/min (300ml/h). The diameter of the obtained nanofibers can be controlled easily by adjusting the polymer concentration and the distance between the nozzle and collector. Interestingly, the nanofibers can be collected on solid and liquid collectors. Three polymers were examined in the introduced technique namely poly(lactic acid), poly(vinyl acetate) and polycaprolactone. The bioactivity of the Mg was strongly enhanced by utilizing the proposed technique to coat an Mg substrate by poly(lactic acid) nanofibers as a plenty of the bone-like precipitates was observed upon soaking of the coated samples for only one day in a simulated body fluid. The results indicated that the proposed technique can be invoked to produce large amount of nanofibers in an almost no time and very low production cost which overcomes the main drawbacks of the
electrospinning process and simultaneously opens a new avenue for synthesizing the nanofibers.

**Keywords:** Nanofibers fabrication; Flash production; Air-jet spinning system; Spinning of non conductive polymers
3. In Vitro Bioactivity of Titanium Implants Coated with Bicomponent Hybrid Biodegradable Polymers

Abdalla Abdal-hay, Hwang Myung-Goo, Jae Kyoo Lim

Abstract:

In order to improve the bioactivity and biocompatibility of titanium (Ti) implants, we designed a novel biodegradable hybrid (polycaprolactone/polylactic acid, PCL/PLA) membrane to coat Ti surfaces. The bicomponent PCL/PLA membrane was applied to a Ti substrate starting with the coating of Ti samples with a porous PLA film layer using a dip-coating technique. This was followed by deposition of electrospun PCL nanofibers onto the Ti substrate, resulting in a PCL/PLA bicomponent hybrid coating layer. The cytocompatibility, bioactivity and corrosion performance of PCL/PLA-coated Ti samples was compared to PLA-coated Ti samples and untreated Ti samples. When placed in Hanks’ solution, apatite formed on the treated Ti samples but not on untreated Ti samples. When assessing Ti cytocompatibility and MC3T3-E1 osteoblast adherence, proliferation, and survival, our results showed superior performance by polymer-treated Ti samples compared to untreated Ti samples, and maximal osteoblast cell viability was achieved with the bicomponent PCL/PLA hybrid coating layer. Furthermore, during the potentiodynamic polarization test in simulated body fluid (SBF), the polymer-coated Ti samples showed corrosion resistance. Therefore, the approach described herein may serve as a basis for the development of polymer-coated Ti surfaces that can be used in dental or orthopedic implants.

Keywords: Biomaterials; Interfaces; Polymers; coatings.
4. Fabrication of a Poly(vinyl alcohol)/Apatite-like Nanocomposite for Biomedical Applications Using a Hydrothermal Technique

Abdalla Abdel-hay¹,², Choong Moo Shim, Jae Kyoo Lim

ABSTRACT

In this communication, Poly(vinyl alcohol) (PVA)/apatite-like nanocomposite films were synthesized using a hydrothermal process at low temperatures for a facile and effective fabrication process. The morphological, structural, thermal, and mechanical properties of the nanocomposites were studied. The morphological analysis confirmed that this fabrication process produced amorphous apatite-like nanoparticles in the PVA solution that were homogenously distributed with controlled particle sizes of less than 20 nm in diameter. The incorporation of low quantities of apatite-like nanoparticles into the PVA matrix could significantly improve the mechanical strength of the resultant biocomposite film, which suggests an excellent load transfer between the apatite and the PVA matrix. This improvement in mechanical strength is due to the effective bonding of the filler nanoparticles in the PVA matrix during the hydrothermal reaction, which is supported by the FTIR and TGA/DSC data. This proposed process offers the possibility of using these synthesized nanocomposite materials in biomedical applications.

Keywords: Bionanocomposite; Bioceramic; Poly(vinyl alcohol)
Abstract

Natural fibers as reinforced polymer composites have recently been the center of attention among researchers. Surface modifications and variations in the fiber diameters are major factors that influence the fiber adhesion performance inside the matrix. Experiments have been performed to further the development of natural fiber reinforced polymers as a replacement for glass fibers. In the present research, date palm fibers (DPFs) with three different size ranges of diameters (800-600, 600-400, and 400-200 μm) and the influence of alkali treatment on their characteristics have been investigated. Morphology observations (SEM), EDS density mapping (quantitative elemental analysis), X-RD, and FTIR spectroscopy of treated and untreated fibers were carried out. In addition, the tensile properties of a single fiber and composites consisting of fibers/epoxy with discontinuous random oriented short fibers both with and without chemical modification were studied. The results showed that DPFs are amenable to chemical modification particularly in the fine fiber case. It was found that the ultimate tensile strength and percentage elongation of a single fiber after alkali treatment increased by 57% and 24.7 %, respectively. Because alkali treatment of the DPFs was able to provide a good adhesion within the matrix, the tensile strength, elastic
modulus and the fiber-matrix interaction of the composite were improved. Collectively, the addition of the proposed DPFs may open a new avenue for the exploitation of this natural cheap material to produce a green composite.
Abstract

The purpose of this research is to improve the biocompatibility of bone tissue engineering scaffolds using a one-step electrospinning process. Calcium phosphate (CaP) was coated on the surface of nylon 6 (N6) nanofibers to form an ultrathin layer, thereby increasing surface roughness and wettability of the N6 nanofiber membrane in order to further improve implant tissue integration. The morphology, composition, chemical interaction bonding and mechanical properties of CaP-coated N6 nanofibers were characterized. The wettability of the scaffold was measured in terms of the water contact angle, and the results indicated that N6 fibers coated with an ultrathin layer of CaP exhibited an excellent surface wettability (water contact angle = 0°). Mechanical testing revealed higher properties of coated CaP layers compared to a plain N6 mat. The biological response induced by the surface modifications of N6 fibers was evaluated by in vitro cell culture with MC3T3-E1 osteoblasts cells. It was found that CaP-coated N6 nanofibrous matrices definitely favored cell proliferation, with the efficiency dependent upon the coating technique. A combination of a nanoscale fibrous structure and a CaP coating could mimic the structure, composition and function of bone tissues.

Keywords: Nylon 6; Bone tissue engineering; Calcium phosphate; Coatings; Electrospun nanofibers
Abstract

The present study was aimed at designing a novel bimodal fiber diameter distributed electrospun hydroxyapatite/nylon-6 (HAp/N6) biocomposite nanofiber mat for bone tissue engineering. The manufacturing of pure N6 fibers and biocomposite fibers was explored by an electrospinning process. The synthesized HAp/N6 composite mats were characterized by XRD, TGA, FE-SEM, EDS, and TEM analyses and water contact angle measurements. The results revealed that fibers of distinct sizes (nano and true-nano scale) were obtained with the addition of a wide range (1-10 wt %) of HAp. Conversion of pristine hydrophobic N6 fibers (130.3°) to super-hydrophilic (0°) composite fibers by simple blending of different amounts of HAp with N6 solution prior to electrospinning could make N6 more biocompatible for hard tissue engineering. Biomineralization was carried out by immersing the composite into simulated body fluid for different lengths of time. Results showed that the nanocomposite had a better ability to form apatite layers on the surface of the fibers compared to the pristine fibers. Therefore, our results suggest that this newly developed HAp/N6 hybrid scaffold may have potential for bone tissue engineering.

Keywords: Hydroxyapatite; Nylon-6; Nanocomposite; Tissue engineering.
8. Biocorrosion behavior and cell viability of adhesive polymer coated magnesium based alloys for medical implants

Abdalla Abdal-hay, Montasser Dewidar, Jae Kyoo Lim

Abstract

The present study was ultimately aimed to design novel adhesive biodegradable polymer, poly(vinyl acetate) (PVAc), coatings onto Mg based alloys by the dip-coating technique in order to control the degradation rate and enhance the biocompatibility of magnesium alloys. The influence of various solvents on PVAc surface topography and their protection of Mg alloys were dramatically studied in vitro. Electrochemical polarization, degradation, and PVAc film cytocompatibility were also tested. Our results showed that the solvent had a significant effect on coating quality. PVAc/dichloromethane solution showed a porous structure and solution concentration could control the porous size. The coatings prepared using tetrahydrofuran and dimethylformamide solvents are exceptional in their ability to generate porous morphology even at low polymer concentration. In general, the corrosion performance appears to be different on different PVAc–solvent system. Immersion tests illustrated that the porous morphology on PVAc stabilized corrosion rates. A uniform corrosion attack in artificial simulation body fluid was also exhibited. The cytocompatibility of osteoblast cells (MC3T3) revealed high adherence, proliferation, and survival on the porous structure of PVAc coated Mg alloy, which was not observed for the uncoated samples. This novel PVAc coating is a promising candidate for biodegradable implant materials, which might widen the use of Mg based implants.
9. Hydroxyapatite-doped poly(lactic acid) porous film coating for enhanced bioactivity and corrosion behavior of AZ31 Mg alloy for orthopedic applications

• Abdalla Abdal-hay, Nasser A.M. Barakat, Jae Kyoo Lim

Abstract

The corrosion behavior of magnesium and its alloys in the electrolytic physiological environment is extremely poor; this imposes a limitation for their use in orthopedic applications. In the present study, the effect of spray coating AZ31 magnesium alloy with membrane films of pristine and hydroxyapatite-doped poly(lactic acid) on corrosion behavior and bioactivity is investigated. Polymer concentration was found to have a strong impact on the pore size of the coating layer. However, addition of HAp NPs distinctly stimulated the precipitation of an apatite-like compound upon soaking the samples in a simulated body fluid (SBF). Magnesium coated samples revealed three orders of magnitude less corrosion compared to the naked samples, which indicates a stable electrochemical corrosion resistance. During a 15 days in-vitro test, pH variation, weight loss, and bending strength were lower for the coated samples (with average values of 8.5%, 7.2% and 10%, respectively) than the control sample (10.5%, 15.5%, and 25%, respectively). Moreover, the coated samples showed good bending strength characteristics. Cytocompatibility studies on MC3T3 cells revealed a continuous increase in cell growth with the coated samples. Overall, the suggested strategy might open a new avenue to widen utilization of Mg alloys as implant materials for orthopedic applications.

Keywords: Mg alloys; Poly (lactic acid); Orthopedic applications; Biodegradable implants
10. Study on Water Absorption and Impact Properties of Vegetal Composites Material: Composite Structures

Abdalla Abdal-hay, Do Yeon Jung, Kang Il Le, G.Tag, Jae Kyoo Lim

Abstract.

The delamination and fibers pull out have been the main factors failure application of natural fibers in various engineering fields. To address these problems, particles reinforced composites are the promising candidate. The present paper investigates on vegetal particles (date palm seed particles/DPSp) and applies it as composites material reinforced unsaturated polyester (USP). The influence of alkali treatment on the surface morphology and structure of DPSp was investigated. They investigated by SEM and Energy Dispersive Spectroscopy (EDS) mapping. The water absorption results showed directly proportion with the particles loading as the relative increases were 0.645% and 7.345% for 10 wt% and 40 wt% of DPSp content, respectively. In addition, the water absorption ability of the composites showed low value comparing with many natural fibers. In addition, the fracture toughness of the composites was studied. Overall, addition of the proposed DPSp particles may be opens a new avenue to exploit the utilized natural cheap material to produce a green composite.

Keywords: Polymer-matrix composites, Surface treatment, Mechanical properties.
11. Titanium Dioxide Nanofibers and Microparticles Containing Nickel Nanoparticles

Faheem A. Sheikh, Javier Macossay, Muzafar A. Kanjwal, Abdalla Abdal-hay, Mudasir A. Tantry, and Hern Kim

Abstract

The present study reports on the introduction of various nanocatalysts containing nickel (Ni) nanoparticles (NPs) embedded within TiO₂ nanofibers and TiO₂ microparticles. Typically, a sol-gel consisting of titanium isopropoxide and Ni NPs was prepared to produce TiO₂ nanofibers by the electrospinning process. Similarly, TiO₂ microparticles containing Ni were prepared using a sol-gel syntheses process. The resultant structures were studied by SEM analyses, which confirmed well-obtained nanofibers and microparticles. Further, the XRD results demonstrated the crystalline feature of both TiO₂ and Ni in the obtained composites. Internal morphology of prepared nanofibers and microparticles containing Ni NPs was characterized by TEM, which demonstrated characteristic structures with good dispersion of Ni NPs. In addition, the prepared structures were studied as a model for hydrogen production applications. The catalytic activity of the prepared materials was studied by in situ hydrolysis of NaBH₄, which indicated that the nanofibers containing Ni NPs can lead to produce higher amounts of hydrogen when compared to other microparticles, also reported in this paper. Overall, these results confirm the potential use of these materials in hydrogen production systems.
12. Influence of electrospinning and dip-coating techniques on the degradation and cytocompatibility of Mg-based alloy

Abdalla Abdal-hay, Nasser A.M. Barakatc, Jae Kyoo Lim

Abstract

The high galvanic corrosion in the physiological environment of Mg-based implant materials is the main constraint hindering wide clinical application. However, a proper coating mechanism is potentially a strategy that may overcome this constraint. In this study, we investigated the influence of coating technique on the biological and corrosive properties of Mg alloy. Electrospinning and dip-coating techniques were employed to coat the Mg substrate with a biodegradable polymer. The results indicated that both techniques strongly enhance the corrosion resistance of the Mg substrate, especially for short immersion times. For longer immersion times, the electrospin nanofiber layer was stable enough to decrease the degradation in comparison to results obtained with porous film produced by dip-coating. The cytocompatibility of the coated and non-coated samples was assessed using in vitro studies with MC3T3-E1 osteoprogenitor cells. The nanofiber layer provided an excellent framework for cell adhesion, proliferation, and differentiation due to its three dimensional structure. Collectively, the results indicate that Mg-based implant materials with an electrospun layer and coated with biodegradable polymers may hold promise for increasing their use in orthopedic and cardiovascular applications.
Biocorrosion and osteoconductivity of PCL/nHAp composite porous film-based coating of magnesium alloy

Abdalla Abdal-hay, Touseef Amna, Jae Kyoo Lim

Abstract

The present study was aimed at designing a novel porous hydroxyapatite/poly(ε-caprolactone) (nHAp/PCL) hybrid nanocomposite matrix on a magnesium substrate with high and low porosity. The coated samples were prepared using a dip-coating technique in order to enhance the bioactivity and biocompatibility of the implant and to control the degradation rate of magnesium alloys. The mechanical and biocompatible properties of the coated and uncoated samples were investigated and an in vitro test for corrosion was conducted by electrochemical polarization and measurement of weight loss. The corrosion test results demonstrated that both the pristine PCL and nHAp/PCL composites showed good corrosion resistance in SBF. However, during the extended incubation time, the composite coatings exhibited more uniform and superior resistance to corrosion attack than pristine PCL, and were able to survive severe localized corrosion in physiological solution. Furthermore, the bioactivity of the composite film was determined by the rapid formation of uniform CaP nanoparticles on the sample surfaces during immersion in SBF. The mechanical integrity of the composite coatings displayed better performance (~34% higher) than the uncoated samples. Finally, our results suggest that the nHAp incorporated with novel PCL composite membranes on magnesium substrates may serve as an excellent 3-D platform for cell attachment, proliferation, migration, and growth in bone tissue.
This novel as-synthesized nHAp/PCL membrane on magnesium implants could be used as a potential material for orthopedic applications in the future.
14. **Inactivation of pathogenic *Klebsiella pneumoniae* by CuO/TiO$_2$ nanofibers: A multifunctional nanomaterial via one-step electrospinning**

**Ayman Yousef, Nasser A.M. Barakat, Touseef Amna, Salem S. Al-Deyab, M. Shamshi Hassan, Abdallah Abdel-hay, Hak Yong Kim**

**Abstract**

The fabrication and characterization of one-dimensional CuO/TiO$_2$ nanofibers with high photocatalytic and antibacterial activities are presented. The CuO/TiO$_2$ nanofibers were prepared by electrospinning of colloid composed of titanium isopropoxide, poly(vinylpyrrolidone) (PVP) and copper nanoparticles and calcination at 700 °C in air for 1 h. The antibacterial activity was tested using *Klebsiella pneumoniae* as model organism by calculation of the minimum inhibitory concentration (MIC). The obtained CuO/TiO$_2$ nanofibers showed prominent photocatalytic activity under visible light to degrade reactive black5 and reactive orange16 dyes in aqueous solutions and effectively catalyze *K. pneumoniae* inactivation. The decomposition process of the cell wall and cell membrane was directly observed by TEM analysis after the exposure of the *K. pneumoniae* to the nanofibers. Interestingly, the introduced photocatalyst can be reused with the same photocatalytic activity. Overall, the combination of CuO and TiO$_2$ can be synergistic and resulted in CuO/TiO$_2$ composite nanofibers having superior photocatalytic and antimicrobial potential to impede *K. pneumoniae* growth which causes bacterium to die ultimately.
Study on Applied load and Irradiation of Ultraviolet (UV) Aging of Polycarbonate Characteristics: Consequences on Mechanical Properties

Do Yeon Jung, Abdallah. Abdel-hay, Jae Kyoo Lim

Abstract

In present study, the effect of irradiation along with applied load and three periods on the ultraviolet (UV) aging of the polycarbonate polymer was investigated by observing the changes in the mechanical properties. Dog bone specimens of polycarbonate were subjected to different test conditions namely in the amount of applied load and the variation in exposure time to the UV lamp. The results showed that, with a constant applied load of 1kN, increase in the exposure time in UV radiation caused the mechanical properties to relatively increase at first and decrease gradually afterwards. However, for constant exposure time, the tensile strength showed much decrease with increase in applied load. For the test conducted at higher applied load of 3kN, the mechanical properties of material relatively improves in a drastic manner for the higher exposure time. It was inferred that the increase in applied load has a crucial role in the UV aging of polycarbonate for the given exposure time. Moreover, the morphology of the fracture area under different test conditions was observed by Scanning Electron Microscope (SEM).

Keywords: Injection molding, Engineering polymers, Scanning Electron Microscope (SEM), Fracture toughness.
16. Characterization and Possibility of Coconut Filter Fibers as Reinforcement for Polymers

N.P.G. Suardana, Ali Abdalla, Ho Cheol Yoon, Jian Guo Cui, Do Yeon Jung, Jao Kyoo Lim

Untreated and treated with alkali (NaOH), acrylic acid (AA), diammonium phosphate (DAP), and maleic anhydride (MA) of coconut filter (CF) fibers were characterized using scanning electron microscopy (SEM), Fourier transform infrared (FTIR) spectroscopy, X-ray diffraction (XRD), thermogravimetric analysis (TGA), and single fiber test. The composites were fabricated from CF and film resins (Polylactic-acid (PLA) and Polypropylene (PP)) using a hot press machine. Generally, our results indicated that chemical treatments improved the mechanical properties of CF fiber composites, except for the DAP-treated fiber/PP composite. The AA treatment of fiber produced the best adhesion at the fiber-matrix interface. Consequently, the tensile and flexural strengths of AA-treated fiber-reinforced polymer were the highest. CF fiber-reinforced PLA composites had better mechanical properties than CF fiber-reinforced PP composites. Our results show that CF fiber is feasible as reinforcement for polymer composites.