HYDROXY APATITE AND MONTMORILLONITE FILLED HIGH DENSITY POLYETHYLENE HYBRID COMPOSITES FOR BIOMEDICAL APPLICATIONS

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HYDROXYAPATITE AND MONTMORILLONITE FILLED HIGH DENSITY POLYETHYLENE HYBRID COMPOSITES FOR BIOMEDICAL APPLICATIONS

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To my beloved mother, wife and three sweet daughters
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ABSTRACT

In this study, new composite formulation for biomedical applications was investigated. The effects of hydroxyapatite (HA) and montmorillonite (MMT) on the mechanical, morphological, thermal and biological properties of high density polyethylene (HDPE) composites which compatibilized with high density polyethylene grafted maleic anhydride (HDPE-g-MAH) were studied. These formulations were compounded using a single screw nano-mixer extruder followed by injection moulding. The effect of HA loadings up to 50 phr were studied and the compositions of MMT and HDPE-g-MAH were kept constant at 5 phr. The performance of the single screw nanomixer extruder was compared with a twin screw extruder. The mechanical properties were studied through tensile, flexural and izod impact testing. X-ray diffraction (XRD) was used to investigate the dispersibility of MMT layers. The thermal properties were analyzed using differential scanning calorimetry (DSC). The morphology of the composites were characterized by scanning electron microscopy (SEM) and transmission electron microscopy (TEM). The study of biological properties was carried out through bioactivity test using simulated body fluid (SBF) immersion. The morphology and calcium-phosphate (Ca-P) precipitation in SBF was characterized by SEM, accompanied by energy dispersive analysis x-ray (EDX) and XRD. The result showed that, the addition of HA significantly increased the strength and stiffness of composites but the elongation at break and impact strength were decreased. The HDPE-HA composites containing 50 phr of HA had the highest elastic modulus, tensile and flexural strength. However, with addition of MMT and HDPE-g-MAH, the composites containing 30 phr HA exhibited high tensile and flexural strength. The melting temperature ($T_m$) and crystallisation temperature ($T_c$) of the composite were not affected by the addition of HA particles, and the crystallinity of the HDPE matrix was increased with increasing of HA content. Incorporation of HA increased the thermal stability of the composites significantly. Based on the mechanical properties of the composite, the performance of single screw extruder nanomixer was more effective in enhancing the HA dispersion compared to twin screw extruder. The bulk formation of apatite layer covering at the composites surface indicated the excellent bioactivity properties of HA and depiction of bioactive composites. Results showed that the composite with 30 phr of HA had optimal mechanical and biological properties.
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LIST OF ABBREVIATIONS

ASTM - American Standards Test Method
DSC - Differential scanning calorimetry
MMT - Montmorillonite
MPa - Mega pascal
GPa - Giga pascal
HDPE - High density polyethylene
HDPE-g-MAH - Maleic anhydride grafted high density polyethylene
XRD - X-ray diffraction
EDX - Energy dispersive X-ray
SBF - Simulated body fluid
BET - Brunauer-Emmet-Teller
TEM - Transmission electron microscopy
LIST OF SYMBOLS

\begin{itemize}
\item cm^2 - centimeter square
\item cm^3 - centimeter cubic
\item d - Spacing between diffractional lattice plane (interspacing)
\item g - gram
\item H_f - Heat of Fusion
\item \Delta H_{100} - Heat of Fusion of theoretically 100\% crystalline polymer
\item mg - milligram
\item T_g - Glass transition temperature
\item T_m - Melting temperature
\item T_c - Crystallization temperature
\item w_i - weight fraction
\item wt\% - weight percent
\item X_c - Crystallinity content
\item ^\circ C/min - Degree celsius per minute
\item \theta - Diffraction angle
\item \lambda - Wave length
\item Cu - Copper
\item K - Pottasium
\item \alpha - Alpha
\end{itemize}
CHAPTER 1

INTRODUCTION

1.1 Background of Research

Nanocomposites, in the sense of hybrid materials with novel properties beyond the realm of unfilled polymers and conventional composites, bear high promise for enabling new uses and applications of polymer materials. In the simplest approach, they can expand the window of applications of a given polymer, and in the best case they can enable the use of polymer–matrix composites in applications where metal or ceramic materials are currently used.

Nowadays, the commercial interest of polymer nanocomposites applications are widely used in motor vehicle parts and packaging. With further research and development, the applications of nanocomposites have been explored intensively in medical area especially in medical device, drug-delivery systems, diagnostic and bio-analytical systems, surgical implants, and innovative packaging. Polymers which represent the largest class of biomaterials continue to replace metals, glass and other conventional materials in the medical field. Polymers of biomaterial class have been studied to cater usage in biomedical devices that include orthopaedic, dental, soft tissue, and cardiovascular implants.

Biomaterials artificial material used to make implants, to replace crocked biological structure and restore form and function. Thus, this biomaterials help to improve the quality of life and longevity human beings. Field of biomaterials have shown rapid growth to keep demands from an aging population. These materials are
used in different parts of the human body as a replacement for artificial valves in the heart, replacement implants in shoulders, hips, elbows, ears and orodental structures stents in blood vessels, knees (Ramakrishna et al., 2001). The number of implants used for knee and hip replacements are significantly high. There is substantial increase in the demand for the new type of long lasting implants.

Based on the data collected on total joint replacements surgery, the number of total hip replacements is estimated to rise by 174% (572,000 replacements) by the end of 2030. Meanwhile total knee arthroplasties is projected to grow up to 3.48 million replacements (Kurtz et al., 2007). The number of replacements in Asian countries is significantly lower than the revision of primary total hip replacement in some countries of Central Europe which was 2.2 per 1000 population. However, the fastest growth is expected in emerging markets of Asia (Kiefer, 2007). The incidence rates of hip fractures in Korea were 173 per 100,000 in women and 91 per 100,000 in men. Statistic obtained for other Asian countries including Taiwan (505 per 100,000 in women and 225 per 100,000 in men), Hong Kong (459 and 180 per 100,000), Singapore (442 and 164 per 100,000), and Thailand (269 and 114 per 100,000) (Ko et al., 2011). The incidence of hip fracture in the city of Kuala Lumpur and the surrounding districts has increased from 1981 to 0.7 per 1,000 population in 1989 with the mean age was 73 (50-103) years (Lee et al., 1993). the development of specific Asian implant designs started for cemented in the year 1980s, followed with cementless implants in the 1990s with a greater focus on specific sizing needs for smaller patients anatomy and different bone morphology according to ethnology and regions (Kiefer, 2007).

The reason for joint replacements is caused by diseases such as osteoporosis (weakening of the bones), osteoarthritis (inflammation in the bone joints) and trauma. The effect of degenerative diseases leads to degradation of the mechanical properties of the bone due to excessive loading or absence of normal biological self-healing process. Thus the solution for these problems is artificial biomaterials since the three critical issues in today’s biomedical implants and devices are the design, material selection and biocompatibility.
The properties of material used for orthopaedic implants especially for load bearing applications need to be excellent biocompatibility and superior corrosion resistance in body environment. Recently, the materials that have been used for these applications are high density polyethylene (HDPE), ultra high molecular weight (UHMWPE), polypropylene (PP), polyetheretherketone (PEEK), polymethyl methacrylate (PMMA) and polysulfone (PSU). In the development of biomaterials for biomedical applications, as far as mechanical properties are concerned, the main target is to strike balance of stiffness, strength, toughness and biocompatibility.

At present, two approaches have been identified as potential route to achieving this goal. This involves inclusion of fillers or nanofillers into thermoplastic matrix or compound to form thermoplastic biocomposites or nanocomposites. The second approaches is compounding of thermoplastic biomaterials with compatibilizer. Hydroxyapatite (HA) reinforced HDPE composite has been under study since 1980s, when Bonfield et al., (1981) lead pioneering work to develop of composite as an alternative for bone replacement. Much attention has been paid towards the development of material for bone tissue engineering with HA as bioactive filler in polymer composites (Fang et al., 2006, Albano et al., 2006). Applying of HA filler particles to form composites has been shown to enhance bone-bonding rates and mechanical properties. The emerging interest of using HA is due to its chemical and structural similarity with natural bone mineral. Liang et al., (2003) has been reported on the use of montmorillonite (MMT) filled HDPE in enhancing the mechanical and thermal properties of the composites for the purpose of biomedical application. MMT offers special properties due to its small size and huge specific surface area. Thus, the excellence performance of MMT leads to the development of nanocomposites materials system in combining the advantages of polymers, HA and MMT.

However, the interfacial problem between HA and the polymer matrix is one of the major factors in determining the properties of the composites. A number of researchers have conducted improvement of interfacial strength between the HA and polymer matrix using coupling agent and grafting methods. Wang et al., (2001) and Deb et al.,(1996) used silane coupling agent and acrylic acid grafting to improve
interfacial bonding between HDPE and HA as it was revealed that only mechanical interlocking occurred between the two phases.

Therefore, this work introduces HA as a particulate bioactive phase with combination of MMT in development of biomedical composite. In order to improve the properties of the composite, the interfacial bond strength between HA and HDPE was improved by using compatibilizer. Thus, this research focuses on the preparation of MMT-reinforced HA/HDPE composites and its effects on mechanical, thermal and biocompatibility properties.

1.2 Problem Statement

In the last few decades, the use of HA reinforced HDPE composite as bone analogue materials has been successfully used clinically in minor load bearing applications such as orbital floor implants and middle ear bone replacement. The main advantage of polyethylene as polymer matrix is due to the biocompatible properties and the ability to allow the incorporation of a large amount of bioceramic particles in the system. However, the use of HDPE as polymer matrix in load bearing applications is limited due to the low stiffness and strength of HDPE. In addition, the mechanical properties of the composites still greatly lower than cortical bone due to the micron-sized reinforcement. Thus, the incorporation of MMT provides an alternative choice to improved the HDPE/HA composite properties which can be melt-processed using current plastics technologies.

In order to improve the strength and stiffness of HDPE, various methods have been explored. Filler surface coated with coupling agents and polymer graft treatment were used to improve the interface adhesion which produced marginal enhancement (Wang and Bonfield, 2001), but the bioactivity of the filler might lose after treatment. Hydrostatic extrusion or shear controlled orientation in injection moulding (SCORIM) was also tried to align polymer chains, the stiffness was significantly enhanced but the yield strength was not improved much (Reis et al., 2001; Sousa et al., 2002).
Therefore, in the present work, the HA was incorporated into HDPE in combination of MMT as reinforcement filler processed through single screw extrusion nanomixer was developed. It is expected that, by using an established manufacturing route, the HA and MMT particles would be finely disperse and distribute in the HDPE matrix. The effect of HA concentration ranging from 10 to 50 phr on the mechanical, thermal and morphology properties were explored. Further, the addition of compatibilizer to improve the interface adhesion between polymer matrix and filler without the expense of bioactivity properties was also investigated.

1.3 Objectives of Research

The present work aims to develop new biocomposites materials namely HDPE/HA/MMT composite. In this research HDPE/HA composites with the presence of MMT and compatibilizer HDPE grafted maleic anhydride (HDPE-g-MAH) were produced using single screw extruder and twin screw extruder nanomixer followed by injection moulding method. The target application of these new materials is for biomedical application such as load bearing (acetabular cup). In this work, five alternative approaches have been investigated to achieve a good combination of properties and processability of bioactive ceramic reinforced polymer composites. The aims are:

i) To investigate the effect of HA on mechanical properties and determine the optimum percentage of HA in the composites.

ii) To investigate the effect of MMT and the HDPE-g-MAH incorporation into the HDPE/HA composites on the mechanical properties.

iii) To study in vitro test on bioactive properties for biocompatibility testing using simulated body fluid (SBF).

iv) To examine the performance of the composite produced using single screw extruder nanomixer as compared to twin screw extruder from– particle distribution and mechanical properties point of view.
1.4 Scopes of Research

In order to achieve the objectives of the research, the following activities have to be carried out:

1. Sample preparation
   Sample preparation was conducted via melt intercalation method. It involves:
   a) Single screw extrusion nanomixer process of compounding HDPE, HA, MMT and HDPE-g-MAH.
   b) Injection moulding to prepare test specimen according to standard.
   c) Twin screw extruder process to compare with single screw extruder nanomixer.

2. Mechanical properties studies
   a) Tensile test
   b) Flexural test
   c) Impact test

3. Sample characterization and morphological study. To characterize the composites, the following apparatus were used:
   a) Differential Scanning Calorimeter (DSC)
   b) Brunauer-Emmett-Teller (BET)
   c) Scanning Electron Microscope (SEM)
   d) Transmission Electron Microscopy (TEM)
   e) X-ray Diffraction (XRD)
   f) Biological Testing

4. Data analysis
1.5 Significance of the Study

From this research, a new HDPE/HA composites formulation with nanofiller MMT for biomedical implant applications was developed which has the potential to be commercialized. The incorporation of MMT and compatibilizer have significantly improved the mechanical and bioactivity properties of HDPE/HA composite.
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