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Tensile Properties and Morphological Studies on HA/PLA Biocomposites for Tissue Engineering Scaffolds

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Abstract : ***This paper investigates the tensile properties and the morphology of the fractured surface of the biomimetic composite scaffold material which is used to fabricate the scaffold that grows the hard tissue like substances. Morphology of the fractured surface was analyzed under scanning electron microscopy (SEM).***

Keywords : Tissue Engineering, Scaffold, Mechanical Properties, Morpholpogy

I. Introduction

A composite material is a material with at least two phases, a continuous phase and a dispersed phase. The continuous phase is the matrix which holds the filling with a specific weight or volume and transfer loads to the dispersed phase [1]. On the other diversified area of composites, biocomposite is one of the promising materials which promotes the field of biomedical research, by mimicking the properties found in the natural materials such as tissues or bones [1]. Mineralized tissues such as bones, tooth and shells have attracted considerable interest as natural anisotropic composite structures with adequate mechanical properties. Ideally, a replacement material should mimic the living tissue from a mechanical, chemical, biological and functional point of view [1]. Hydroxyapatite (HA) has been applied widely in medical field as a bone repair material because of its excellent bioactive and biocompatibility properties [2]. In most cases, biocompatible, degradable polymers are utilized to induce surrounding tissue ingrowth or to serve as temporary scaffolds for transplanted cells to attach, grow and maintain differentiated functions [3]. Therefore, composition of HA and PLA composites not only improves the mechanical properties of the composites but also endows its excellent bioactivities. It can form bone bonding with nature tissue through osteo-conduction mechanism [2]. Polymer matrix composites have the advantage of being very versatile, allowing for the tailoring of its final properties. Composites can be designed and produced with specific requirements, using a wide range of polymeric matrixes, reinforcements and processing routes [1-2]. Bonfield *et al.*(1981) proposed the use of composites of HDPE with hydroxyapatite (HA), introducing the so-called bone-analogue concept. Also, it is a well-known fact that hydroxyapatite granules have been used clinically as substitutes for autografts in filling bone defects [4]. One of the present challenges in polymer scaffold processing is the fabrication of three-dimensional (3D) architectures with an adequate mechanical performance to be used in the tissue engineering of hard tissues [3]. Some of the short comings of these materials include stiffness and rigidity of the composite, porosity and density are not similar to bone material, brittleness of the bulk bioceramic, bioactivity and bioabsorbability, biodegradability and co-efficient of friction [4]. The materials (ie. metals, ceramics [5] and polymers) that were used in the medical field were originally developed for general engineering applications rather than for tissue replacement. However, there are also short comings of these materials for their intended medical applications such as bone graft.

Various processing methods have and are being developed to fabricate these biocomposite scaffolds, such as impregnatation and sintering ceramic scaffolds processing, solvent casting, particulate leaching, gas forming, non-woven fibre, fibre knitting, phase separation/ emulsion freeze drying for manufacturing scaffold [6]. Some of the emerging methods such as indirect fabrication of scaffold, by Solid free form (SFF) [6], and high pressure injection moulding method where a wide range of biomaterials or a combination of materials (composite such as polymer/ceramic) is cast in the mould [7-8]. The major problem of the scaffolds produced by the methods developed so far is their mechanical weakness, which does not allow for their use in hard-tissue regeneration where high-strength scaffolds are required [10]. Therefore, the search for better ways of producing porous scaffolds, so that physical and chemical properties can be simultaneously optimized, is currently an important issue in hard tissue engineering research [10].

In the present work, HA was incorporated in PLA with different proportions by melt extrusion and followed by injection moulding process. The HA/PLA composites were tested for their tensile properties and scanning electron microscopy was used to observe the effect of the HA in the PLA biocomposites.

II. Material and Methodology

Materials

Poly-lactic acid (PLA resin, Specific gravity: 1.24, grade 3051D from NatureWorks®, China) was dried in a vacuum oven at 60 ^{0}C for 24 h to remove residual moisture before use. Synthetic Hydroxyapetite (HA, Fluka 21223) powder from Sigma-Aldrich with grade Fluka 21223 (Particle size: 10-20 microns, M_w: 502.24 g/mol) was used in this study.

Methods

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This work included the development and preparation of biodegradable, bioactive PLA composite with 10wt%, 20wt% and 30wt% of HA as shown in Table 1. The PLA and the HA were melt-mixed together in a twin-screw extruder (HAAKE rheometer) at 180^0C and extrusion pressure of 30-40MPa.

Table 1 Formulations of HA/PLA composites

Sample ID	HA (wt%)	PLA (wt%)
Neat PLA	0	100.0
HA/PLA10	10.0	90.0
HA/PLA20	20.0	80.0
HA/PLA30	30.0	70.0

The maximum torque set during the mixing process was 100Nm for composites with 10-30wt% HA. The extruded mixtures were then molded in an injection molding machine (Battenfeld HM 600), to produce tensile specimens according to ASTM D 638 as shown in Figure 1.
The barrel temperature was set from 175^0C – 185^0C.Composite samples prepared by injection molding process were characterized in terms of their mechanical properties such as tensile strength, tensile modulus and % elongation and their fractured surface morphology. From Figure 1, we can observe the images of the fractured samples of Neat PLA, 10wt% HA/PLA, 20wt% HA/PLA and 30wt% HA/PLA from left to right.

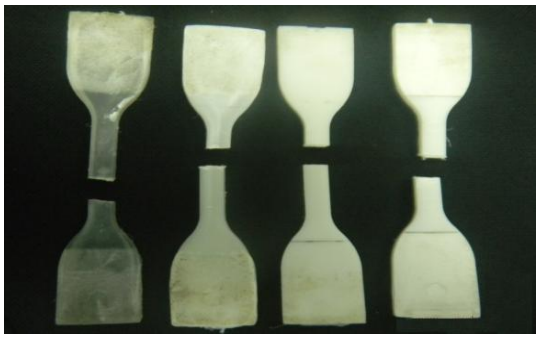

Figure 1 Fractured tensile specimens of HA/PLA composites (ASTM D 638)

The developed composite test specimens were tested in an Instron 8201 universal tensile testing machine using a load cell of 50 kN for its mechanical properties such as tensile strength, modulus and % elongation at break. The tests were carried out in a controlled environment (23.5C and 55% RH) and at a cross-head speed of 1 mm/min. A minimum of three specimens were tested for each composition.
Scanning electron microscopy (Hitachi, S-3400N, Japan) was used to analyze the fractured surface morphology of the HA particle distribution and bonding between the matrix (PLA) and the filler (HA) particles.

III. Results and Tables

Mechanical properties
The results of HA/PLA composites are shown in Figure 2-4. The composites were made into tensile test specimens by injection molding process. The results indicated that the tensile strength of HA/PLA samples consistently decreased with increasing of HA content. For the PLA composite containing 10wt% HA and 30wt% HA, the tensile strength reduced to 52.5 MPa and 45.5MPa respectively.

The tensile modulus of the HA/PLA composites increases with the increasing filler loading, which indicated that the stiffness of the composites increases with increasing filler loading. The relationship between the HA loading and the tensile modulus for the PLA composites is illustrated in Figure 3.

The presence of the HA substantially increase the tensile modulus relative to the pristine PLA. For the PLA composite containing 30 wt% HA, the modulus even reaches to 4.7GPa as compared to 3.8GPa for pure PLA. From Figure 4, it was observed that the percentage elongation at break (E_b) of the HA/PLA composites decreased as the filler loading increased. The E_b for neat PLA was found to be 1.52% and decreased to 0.97% as the HA loading increased to 30wt%.

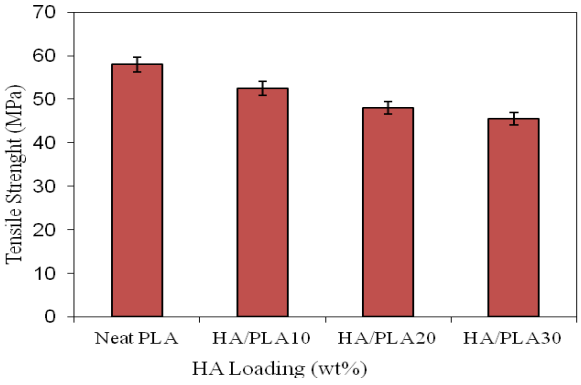

Figure 2 Tensile strength of HA/PLA composites

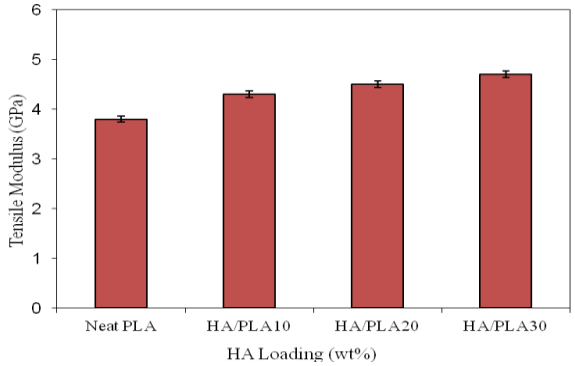

Figure 3 Tensile modulus of HA/PLA composites

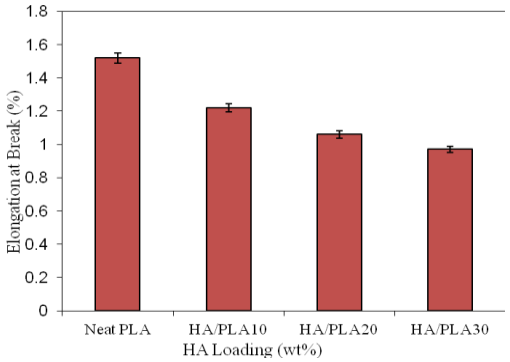

Figure 4 % elongation at break (E_b) of HA/PLA composites

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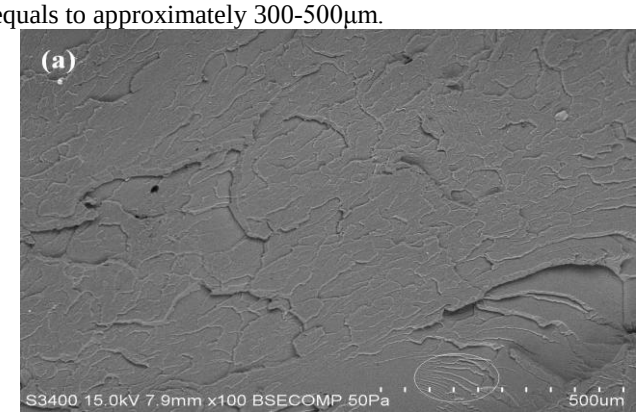
Morphological Properties

Scanning electron microscope (SEM) was used to analyses the morphology of tensile fracture surface of HA/PLA composites. Figure 5 (a) – (b) show the SEM micrographs of tensile fractured surface of Neat PLA at 100X mag and 500X mag respectively. The SEM micrograph shows the brittle fracture of the Neat PLA Sample. There is no fillers seen in the images confirming the Neat PLA sample.

Figure 6(a) – (b) show the SEM micrographs of tensile fractured surface of 10wt%HA/PLA composite at 100X mag and 500X mag respectively. Based on the observation, it shows that the HA particles dispersed evenly except some part of the composites were agglomerated particles can be found and some parts were left with voids due to the filler detachment. The average size of the agglomerated particles equals to approximately 100-150µm.

It was observed that HA was detached from the PLA matrix and voids occurred in the HA/PLA composites. The average size of the agglomerated particles equals to approximately 200-300µm. Figure 8(a) – (b) show the SEM micrographs of tensile fractured surface of 30wt%HA/PLA composite at 100X mag. and 500X mag. respectively. It was observed that the agglomeration of the HA particles at higher filler loading. Surface treatment of the filler and by introducing a compatibilizer could prevent the agglomeration of the filler particles. The average size of the agglomerated particles equals to approximately 300-500µm.



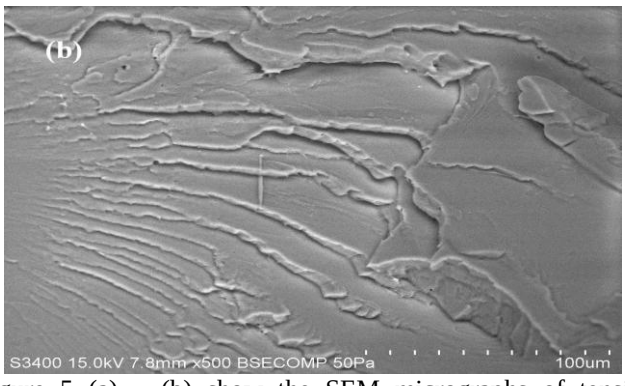
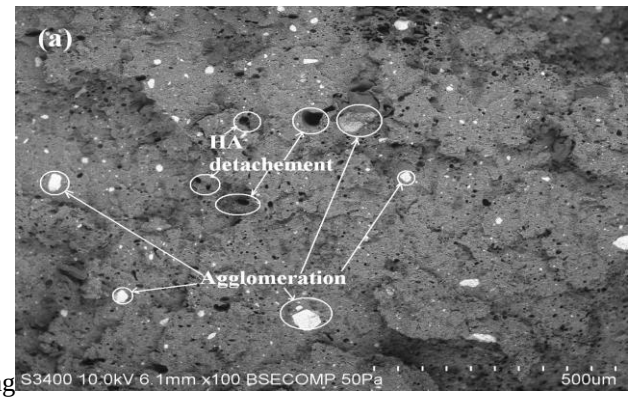


Figure 5 (a) – (b) show the SEM micrographs of tensile fractured surface of 20wt%HA/PLA composite at 100X Figure 5. Typical microstructure obtained from the fractured surface of Neat PLA sample(a) 100X mag. (b) 500X



mag

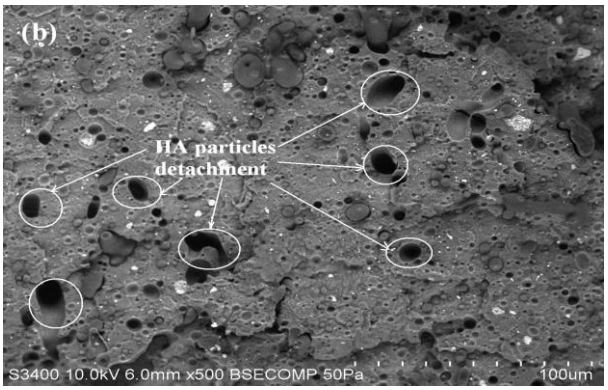
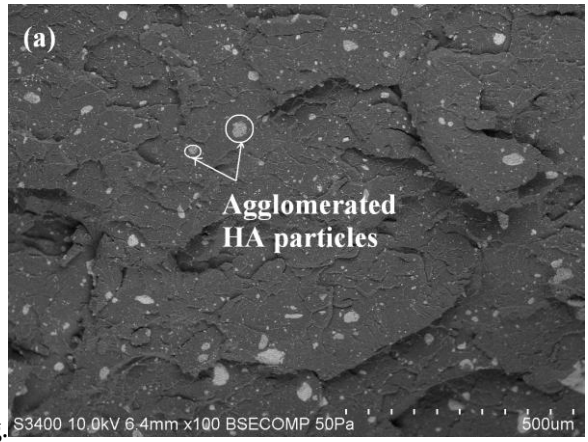


Figure 6. Typical microstructure obtained from the fractured surface of HA/PLA10 (a) 100X mag. (b) 500X



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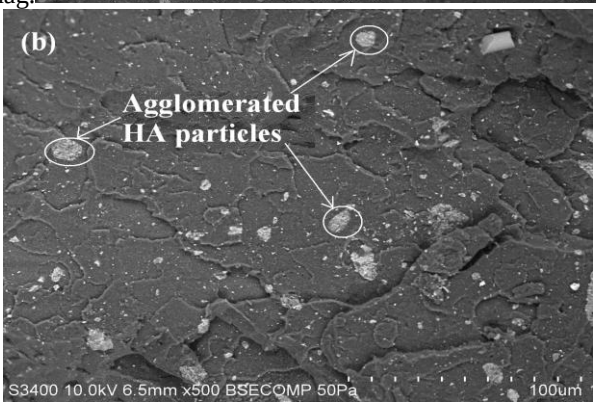

Figure 7. Typical microstructure obtained from the fractured surface of HA/PLA 20 (a) 100X mag. (b) 500X mag.

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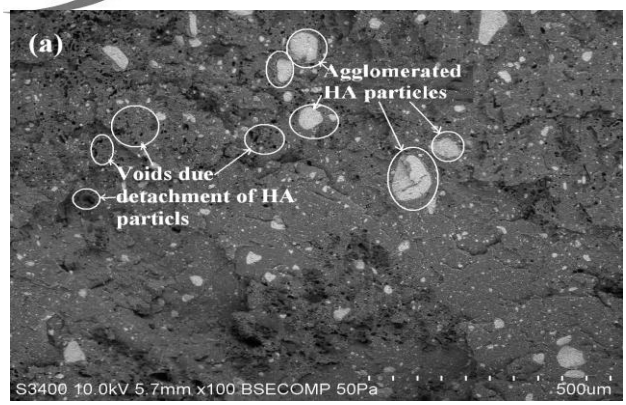

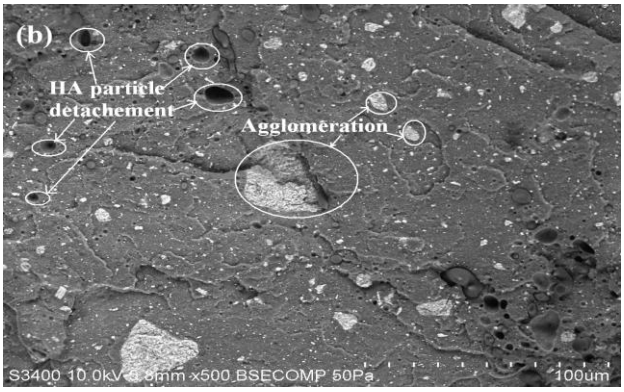

Figure 8. Typical microstructure obtained from the fractured surface of HA/PLA30 (a) 100X mag. (b) 500X mag.

IV. Conclusion

Biocomposite from hydroxapatite and Poly(lactic) acid (HA/PLA) test specimen were prepared using twin screw extruder and injection moulding process which is one of the processing method for the production of the biodegradable polymer composites. Tensile properties of PLA, HA/PLA composites such as tensile strength, tensile modulus and % elongation at break were determined. It was found that 10wt% HA/PLA has better tensile strength compared to 20wt% HA/PLA and 30wt% HA/PLA composites. Consequently, tensile modulus of the HA/PLA composites increased with the increase in HA content. The percentage elongation at break was decreased with the increase in the HA loading. The fractured surface of the samples were analyzed using scanning electron microscope. Optimum composition of HA/PLA will be selected for scaffold preparation and their bioactive and biodegradable properties will be studied later.

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