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Mechanical, thermal and morphological characterisation of 3D porous *Pennisetum purpureum*/PLA biocomposites scaffold

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ABSTRACT

The mechanical, thermal, and morphological properties of a 3D porous *Pennisetum purpureum* (PP)/polylactic acid (PLA) based scaffold were investigated. In this study, a scaffold containing *P. purpureum* and PLA was produced using the solvent casting and particulate leaching method. *P. purpureum* fibre, also locally known as Napier grass, is composed of 46% cellulose, 34% hemicellulose, and 20% lignin. PLA composites with various *P. purpureum* contents (10%, 20%, and 30%) were prepared and subsequently characterised. The morphologies, structures and thermal behaviours of the prepared composite scaffolds were characterised using field-emission scanning electron microscopy (FESEM), Fourier transform infrared spectroscopy (FTIR), and thermogravimetric analysis (TGA). The morphology was studied using FESEM; the scaffold possessed 70–200 μm-sized pores with a high level of interconnectivity. The moisture content and mechanical properties of the developed porous scaffolds were further characterised. The *P. purpureum*/PLA scaffold had a greater porosity factor (99%) and compression modulus (5.25 MPa) than those of the pure PLA scaffold (1.73 MPa). From the results, it can be concluded that the properties of the highly porous *P. purpureum*/PLA scaffold developed in this study can be controlled and optimised. This can be used to facilitate the construction of implantable tissue-engineered cartilage.

1. Introduction

Recently, hyaline cartilage has been studied extensively. It functions as a surface covering for all the diarthrodial joints in the human body, protecting them from abrasion and wear [1]. Hyaline cartilage is also frequently known as articular cartilage. Articular cartilage is a heterogeneous and mechanically anisotropic tissue. Since cartilage does not contain nerves, it has very limited capacity to repair itself because of its low cellular content and lack of blood vessels [2]. In, recent years, the development of new artificial cartilage, used to repair and restore defective cartilage, has been of considerable interest [3].

Biomedical composites can be used as a scaffold for tissue repair and regeneration; the growth of cells prior to implantation; and the deposition of engineered tissue. Their development offer significant market potential and attract continuous financial investment [4]. Concepts such as long-term sustainability, ecological awareness, and the emerging "green" economy have motivated the identification of green materials that are compatible with living human cells. Unlike synthetic polymers, natural fibre composites (NFCs) have increased in popularity among researchers because of their availability, carbon neutrality, low density, renewability, and biodegradability. In addition, they can be used without damaging the environment [5]. Moreover, the incorporation of fibre reinforcement within the polymer matrix of a composite is proven to increase its mechanical strength. It is also known to improve other crucial parameters, such as the degradation rate and structural properties of the composite, as well as the resultant cell proliferation, and viability [6]. In addition, these natural fibre reinforced polymer composites have an extended lifespan, which could be used to improves the quality of life of human. They could be used for various potential applications, and are considered suitable for orthopaedic application and tissue engineering [7–12].

In tissue engineering, the most commonly used polysaccharides, such as starch, collagen, chitosan, alginate, and gelatin, have been widely studied with regard to various biomedical applications [13]. Several studies related to the application of 3D chitosan scaffold in xenotransplantation have been published by Steck [14] and Ehrlich [15]; where the human chondrocytes were seeded into chitosan scaffold before transplanted into mice for tissue regeneration. Owing to its biocompatibility, Chevallay and Herbage suggested that collagen could potentially be used as a material for scaffold synthesis; it can also be prepared in

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different forms with varying physical properties [16]. Martino et al. reviewed the current application of chitosan in tissue engineering. They concluded that chitosan-based composite biomaterials are suitable for various tissue engineering applications because of their suitable degradation behaviour, and microstructures with desirable pore sizes [17]. Gong et al. studied the biological performance of polylactic acid (PLA)/gelatin scaffold materials. They determined that the gelatin particles increased the hydrophilicity and biocompatibility of the scaffolds [18]. Despite all these promising findings, it is difficult to utilise collagen, chitosan, and gelatin, as reinforcement material during the synthesis of scaffold. They are not ideal materials for tissue engineering because they promote the immune activation of cells, and inflammatory reactions occur due to their degradation behaviour [19]. However, these polysaccharides are still favoured as reinforcement materials in tissue engineering studies. The use of plant-based fibres as reinforcement in scaffolds is of novel interest with regard to tissue engineering applications. The development of new plant fibre-based composites for various applications in biomedical fields is of interest for future research.

Among all the synthetic polymers, PLA has been used widely in tissue engineering applications because of its superior biodegradability and biocompatibility [20–23]. In particular, PLA has been extensively studied to develop scaffold, which has been used to prepare bone tissues, drug delivery systems, stents and artificial organs, for both research and commercial use [24]. Chen et al. studied the morphology of a porous PLA scaffold prepared by thermally induced phase separation. The scaffolds were prepared via solid—liquid phase separation and liquid—liquid phase separation, where they exhibited anisotropic tubular morphologies and isotropic morphologies, respectively [25]. Cheung et al. studied the influence of silkworm silk/PLA on the mechanical and thermal behaviour of biocomposites. According to the authors, the tensile properties of the silk fibre/PLA composites were superior to those of pure PLA. The addition of the silk fibre further enhanced the thermal and physical properties of the composites, forming an ideal scaffold. The findings provide the impetus for this material to be potentially used as a material for tissue engineering [26].

A key challenge faced by the field of tissue engineering is the design of cartilage scaffold with favourable microstructure and mechanical properties that promote cell growth and attachment, and new tissue regeneration [27]. The use of *P. purpureum* as a reinforcement filler in a PLA matrix offer many advantages; it results in the production of the biocomposites with favourable mechanical properties and controllable biodegradability. Ridzuan et al. determined that *P. purpureum* fibres could potentially be used as reinforcement materials in polymer composites. They determined that the application of alkaline treatment increases the surface roughness and minimises the hemicelluloses quantities of the fibre [28]. Hameem et al. studied the tensile and flexural properties of *P. purpureum* and concluded that when polyester composites were reinforced with *P. purpureum*, their mechanical properties improved [29]. Moreover, Ridzuan et al. studied the effect of elevated temperatures on the mechanical behaviour of *P. purpureum*/glass-reinforced composites. The tensile and flexural strength of the composites decreased as the temperature approached the glass transition temperature (T_g) at >60 °C; this was owed to the softening of the composite's matrix [30]. Similarly, Ridzuan et al. reported that moisture absorption influences the mechanical properties of hybrid *P. purpureum*/glass–epoxy composites [31]. They observed that as the glass fibre content of the samples is increased, the degree of moisture absorption decreased and the tensile and flexural strength of the composites increased. Recently, based on several studies, researchers suggested that *P. purpureum* fibres could effectively be used as reinforcement in polymer composites for tissue engineering applications [28–31,33].

To benefit from the outstanding properties of *P. purpureum* and its derivatives, we have designed highly porous *P. purpureum*-PLA scaffolds, which are prepared using the solvent casting and particulate leaching technique. This preparation method was selected because it can be used to control the porosity and pore sizes of the scaffold. Furthermore, compared with other methods, this technique requires less energy and time, and it is also inexpensive and consumer friendly. The physical, morphological and mechanical properties of the resultant *P. purpureum*-PLA biocomposite scaffolds with various compositions are subsequently discussed.

2. Materials and methods

2.1. Materials

P. purpureum powder was prepared from *P. purpureum fibre* also well known as Napier fibre which was purchased from a local plantation in Bukit Kayu Hitam, Kedah, in northern peninsular Malaysia. PLA of label 4032D was acquired from NatureWorks LLC. Chloroform and dichloromethane were purchased from Fisher Chemical Co. and were used as organic solvents. Analytical-grade sodium chloride (NaCl) was purchased from Sigma-Aldrich, Germany and used as a porogen.

2.2. Extraction and preparation of Pennisetum purpureum powder

The *P. purpureum* grass leaves were removed from the internodes of each stem. Subsequently, the stems were crushed and then immersed in a tank filled with water. The stems were immersed in the water-filled tank for approximately two to three weeks for the retting process to occur. Prior to the extraction process, the soaked *P. purpureum* grasses were thoroughly washed with running water. The fibres were extracted manually from the stems, and the extracted raw fibres were then sun-dried to remove any excess moisture present within the fibres. To prepare the *P. purpureum* into powder form, the dried fibres were cut into small pieces, and then ground and sieved. The powder was ground once again and sieved using a 63 μm sieve to obtain fine *P. purpureum* powder.

2.3. Preparation of porous PLA scaffold reinforced with Pennisetum purpureum as a filler

The PLA and *P. purpureum* composite scaffolds with a porous microstructure were prepared by a salt-leaching and solvent casting method. The PLA pellets were dissolved in chloroform and dichloromethane (volume ratio of 1:1) to obtain a viscous solution (60.0 g/L). To produce a composite scaffold consisting of PLA and *P. purpureum*, the desired amount of fibre powder (composition listed in Table 1) was dispersed in a solution of PLA using a magnetic stirrer; the temperature was maintained at 70 °C for 2 h. Subsequently, NaCl was added to the solution, and the solution was stirred again for 20–30 min to obtain a homogenous composite. The PLA composite and NaCl were blended to a ratio of 1:9. The salt—PLA composite solution was then cast into a rectangular Teflon mould (width, length, and thickness of 13 mm, 25 mm, and 13 mm, respectively), to produce samples suitable for mechanical testing. The dried PLA composites were placed in distilled water for 48 h to leach out the salts; every 4 h, the distilled water was refreshed to complete elimination of the salt. The leached scaffolds were stored overnight in a vacuum drier at room temperature. The resultant scaffolds were then stored in a desiccator prior to characterisations.

Table 1
Composition, porosity and average pore size of the *P. purpureum*/PLA scaffold.

Samples	PLA (g)	*Pennisetum purpureum* (g)	Sample code	Porosity (%)	Average pore size (μm)
S1	2.4	0.00	PLA_C	99.6	105 ± 36
S2	2.4	0.24	$PLA\text{-}PP_{10}$	99.4	170 ± 45
S3	2.4	0.48	$PLA\text{-}PP_{20}$	99.1	103 ± 21
S4	2.4	0.72	$PLA\text{-}PP_{30}$	99.2	158 ± 23