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Prof. Osman Adiguzel
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Fabrication and Microstructure Evaluation of Fibrous Composite for Acetabular Labrum Implant

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Abstract. This paper will report the fabrication process and microstructure analysis of fibrous composite incorporating ultra-high molecular weight polyethylene (UHMWPE) fabric, electrospun polycaprolactone (PCL), and bioglass particles. Briefly, electrospinning was performed to form PCL fibre lamination in the surface of UHMWPE fabric. This UHMWPE/PCL material was then bioglass-coated. Sequentially, microstructure of the UHMWPE fabric, UHMWPE/PCL, and UHMWPE/PCL/bioglass was imaged and analysed. The composite showed aligned ultrafine PCL fibres and distribution of bioglass particles in the layer of electrospun PCL. The results of this study provide groundwork for more advanced investigation, as well as development of implant prototype.

Introduction

Acetabular labrum has important roles in biomechanical function of hip joint. This fibrocartilage rim encircles the hip joint between femur and acetabular cup, improving hip stability [1], [2]. It also provides sealing to protect fluid inside the hip joint [3]. Thus, tears in the labrum may impact negatively on activities related to hip joint and even progress to osteoarthritis [4]. In the case of labral injuries, it is considered important to preserve the function of labrum. In several cases where the damaged labrum is irreparable, such as degeneration or tissue deficiency, labral reconstruction is often required [5]. This process involves the use of tissue graft, including ligamentum teres, iliotibial band, or gracilis autograft [5]-[9]. However, application of graft may possess some limitations, for example limited availability and need of additional surgery [5], [10]. To overcome these limitations, synthetic graft can be an alternative approach.

Broad range of synthetic implants have been developed for treatment on fibrocartilage injury, including meniscus, intervertebral disc, and anterior cruciate ligament (ACL). Many of them employed inert polymer that offers mechanical and chemical stability, for example polytetrafluoroethylene (PTFE), polyethylene terephthalate (PET), and ultra-high molecular weight polyethylene (UHMWPE) [11]-[26]. However, these materials are inert and may be unable to aid tissue remodeling and bond with surrounding tissue.

Another approach in developing fibrocartilage implant is the application of biodegradable materials as temporary scaffold, which degrades overtime and gradually replaced by ingrown tissue. Synthetic materials, particularly biodegradable polyesters, are arguably the most investigated materials for tissue engineering scaffold and have been already approved by FDA [27]-[29]. Among those polyesters, polycaprolactone (PCL) gains popular use in soft tissue applications. It has been extensively used as drug-delivery devices, sutures, and wound dressings, as well as developed as tissue engineering scaffolds for cartilage, ligament, blood vessel, skin, and nerve [30].

The ability of an implant to integrate with surrounding tissue is important for successful implantation. Implant and tissue bonding could maintain implant stability and avoid dislocation. Therefore, addition of bioactive substances is required to promote this bonding. Bioglass is a highly bioactive material that can bond with both bone and soft tissue, and even stimulate gene activation [31]. Due to its brittle nature, bioglass is commonly applied as coating to stimulate implant attachment to host tissue [31]-[33]. Related to soft tissue application, bioglass showed promising results in promoting implant integration, vascularization, and chondrogenesis [34]-[39].

As fibrocartilage, acetabular labrum possesses fibrous architecture, in which the fibre structure follows applied stress [40]. Therefore, fibre-based material is proposed for the labrum implant. For macrostructure, UHMWPE fabric is applied to provide mechanical strength and structural stability. It demonstrated promising fatigue and abrasion resistance in the studies on intervertebral disc and ACL replacement [22], [24], [25], [41]. Furthermore, this fabric has been used in clinical applications [20], [42]. The implant also has degradable layer of PCL fibres providing temporary environment suitable for cell attachment and tissue formation. The application of PCL in fibrocartilage tissue engineering is reported to have reached preclinical stage, particularly for meniscus and intervertebral disc [43], [44]. To produce the fibrous PCL, electrospinning technique will be employed. A specific collector design was developed to obtain aligned fibre structure [45]. This alignment is important, as it directs cells alignment and extracellular matrix formation [46], [47]. It can also mimic the native tissue, as the main layer of labrum also consists of highly oriented collagen fibres [40]. Additionally, the implant is coated using bioactive glass to promote bonding with bone and soft tissues, since it will interact with both tissues.

This paper presents fabrication process of UHMWPE/PCL/bioglass fibrous composite. As a preliminary study, an investigation on the microstructure was carried out. Microstructure is important since it can dictate cell responses, in terms of morphology and extracellular matrix deposition. The morphology of electrospun fibres deposited onto UHMWPE fabric will be examined, along with the effect of bioglass addition on fibre diameter and morphology.

Method

Electrospinning. Polycaprolactone solution was prepared by dissolving the polymer pellets (Mw 80.000) in acetone (Barnes, Australia) overnight to create 10% w/v concentration. The electrospinning process is depicted in Fig. 1. The collector was a rotating aluminium tube with gap feature and insulator covering [45]. The process was set at flow rate of 4.5 ml/h, 12.5 cm working distance, and mandrel rotation of 1500 RPM. 10 ml syringe with 20G needle was used to dispense the solution onto the grounded rotating collector. The needle tip was connected to a van der Graaf generator to charge the solution. Flow rate was adjusted using syringe pump (Injectomat Tiva Agilia).

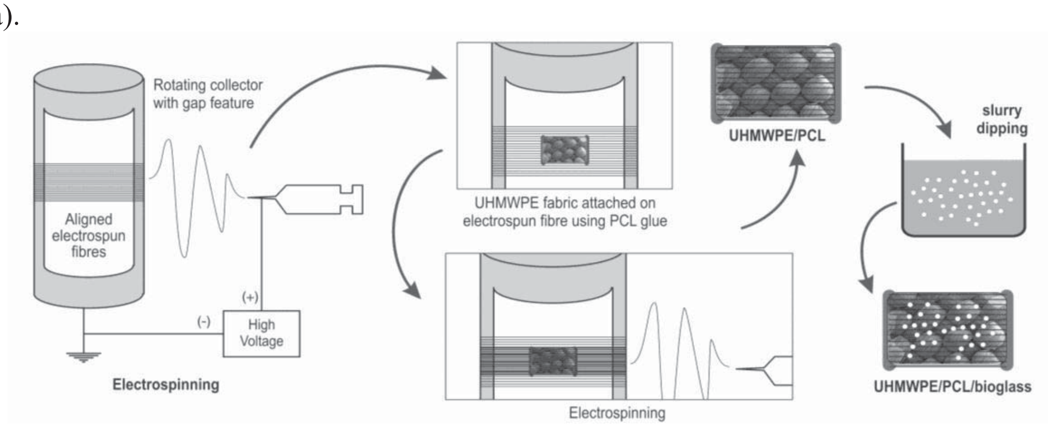

Fig. 1. Fabrication process of UHMWPE/PCL/bioglass

Fabrication of UHMWPE/PCL. Electrospinning was run in two steps (Fig. 1). The first step provided bottom layer of aligned electrospun PCL fibres. Patches of UHMWPE fabric were then placed over the PCL fibres deposited in the gap area of the collector. The fabrics were attached to the fibres by applying PCL/acetone solution as glue at the fabric edges. Once the PCL glue dried, the second electrospinning step was started to form the upper layer of PCL fibres. This process resulted in UHMWPE fabric patches laminated with electrospun fibres, termed UHMWPE/PCL.

Bioglass coating. The obtained UHMWPE/PCL was then bioglass-coated using slurry dipping method [48], [49]. Melt-derived 45S5 Bioglass particles were sourced from earlier study in our research group. The slurry was made in 5% concentration, by suspending the bioglass particles in DI water. The suspension was then stirred for 30 minutes using magnetic stirrer prior to dipping.

The UHMWPE/PCL patch was then gently immersed in the stirring slurry for 5 minutes using tweezers. The obtained UHMWPE/PCL/bioglass samples were dried in room temperature.

Microstructure Analysis. Microstructure of the composite materials was imaged using scanning electron microscope (Zeiss Ultra plus). Prior to imaging, the samples were gold-coated using plasma sputter. Samples from both groups (UHMWPE/PCL and UHMWPE/PCL/bioglass) were evaluated by taking two samples from two replications for each group. 90 random measurements of fibre diameter were performed for each sample using imageJ software. To quantitatively compare the fibre thickness between groups, statistical analysis was carried out using Minitab 17 software.

Results and Discussion

Fibrous composite of UHMWPE/PCL has been fabricated using electrospinning technique, followed by bioglass addition through slurry dipping method. The morphology of UHMWPE fabric, bioglass powder, UHMWPE/PCL, and UHMWPE/PCL/BG materials is depicted in Fig. 2. The UHMWPE fabric (Fig. 2a) had thick fibres of 13.35 ± 3.95 μm, tightly arranged in specific orientation. Histogram shows that the thickness of UHMWPE fibres distributed mostly around 10-12 μm (Fig. 2d). The electrospun fibre on UHMWPE/PCL (Fig. 2b) material possessed softer fibres with diameter of 1.543 ± 0.79 μm with fibre distribution leaned toward smaller diameter of less than 1 μm (Fig. 2e). The electrospun fibres showed specific directionality as intended, as well as spaces between fibres. The addition of bioglass slightly changed the morphology of electrospun fibre (Fig. 2c), which became thicker with diameter of 1.771 ± 0.88 μm. Despite still followed certain directionality, the filaments appeared to be slightly bent and overlap each other. It was possibly due to spinning process during slurry dipping, which might break the smaller fibres, thus increasing the average thickness. Bioglass appeared to deposit evenly, taking place in the surface, in between, and underneath the PCL fibrils. To examine whether the difference in fibre diameter due to immersion treatment is significant, nonparametric statistic of Mann-Whitney was carried out to compare both group. The result suggested that the difference in PCL fibre diameter between UHMWPE/PCL and UHMWPE/PCL/bioglass was significant ($p<0.05$).

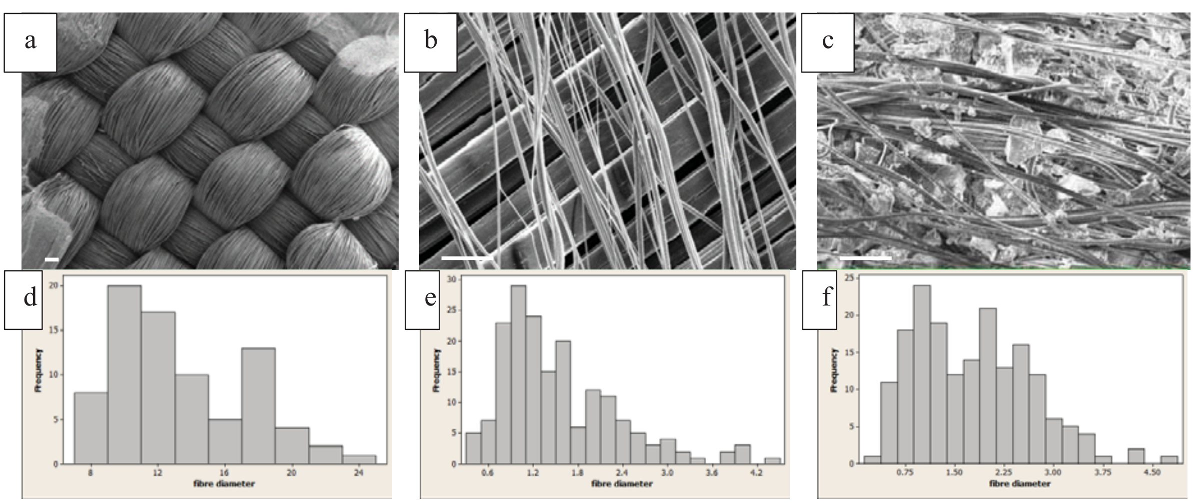

Figure 2. SEM images of (a) UHMWPE fabric (scalebar 200 μm), (b) UHMWPE/PCL (scalebar 20 μm), (c) UHMWPE/PCL/bioglass (scalebar 200 μm); fibre diameter distribution of (d) UHMWPE fabric, (e) PCL fibres on UHMWPE/PCL, (f) PCL fibres on UHMWPE/PCL/bioglass.

UHMWPE fabric offers strength and durability required for fibrocartilage implant [20]-[25]. However, its inert behaviour may only be able to stimulate minimum tissue response. Besides, the dense fibrous structure of the fabric may be less favourable for cell attachment, as cells tend to prefer fibrous architecture with higher surface area for binding site [50]. The layer of ultrafine fibre was introduced to accommodate this need of larger surface area. Aligned structure was also achieved and could be expected to support tissue regeneration, as cells appeared to perform better alignment, propagation, and formation of extracellular matrix in highly aligned architecture [47],

[51]. Furthermore, bioglass coating has been attained with relatively uniform and even distribution of particles. The particles were also able to infiltrate the layer of electrospun fibre and deposited between the UHMWPE fabric and fibrous PCL layer. Bioglass is well known for its ability to bond with both hard and soft tissue [31]-[33], providing the implant with potential ability to integrate with surrounding tissue. However, the bioglass addition altered the structure of electrospun fibre, noticeably in the increase of average fibre diameter. Whether this will influence cells behaviour remains need investigation, although previous studies showed that fibres with thickness of 1.5–2.5 μm could facilitate cells attachment and proliferation [47], [52], [53]. Optimum amount of bioglass addition could further enhance positive cells responses, even facilitated neovascularization [34], [35]. Referring to the previous studies, microstructure obtained in this study could be expected to deliver suitable environment for cell growth. This result also provided a basis for further development of fibrocartilage implant, particularly for acetabular labrum. To assess the potential performance of these composite materials, in-vitro study has been being prepared, along with mechanical tests to examine the strength and durability.

Conclusion

Fibrous composite of UHMWPE/PCL/bioglass can be fabricated by applying electrospinning process and slurry dipping. The composite features aligned ultrafine PCL fibres and even distribution of bioglass particles.

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